INTERNATIONAL STANDARD

ISO 20795-2

Second edition
2013-03-01

Dentistry — Base polymers —

Part 2: Orthodontic base polymers

Médecine bucco-dentaire — Polymères de base —

Partie 2: Polymères pour base orthodontique

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

This second edition cancels and replaces the first edition (ISO 20795-2:2010), which has been technically revised.

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

Polymeric materials based on methacrylates have been widely used in the construction of both active and passive removable orthodontic appliances for many years. These removable appliances are mainly used in the orthodontic treatment of children. The method of preparing the polymeric part of the orthodontic appliance has several potential problems. Depending on the polymerization process and polymer/monomer mixing ratio, the polymer part of the removable orthodontic appliance may be weaker than if conventional flasking and heat systems of polymerization were used. There may be a greater risk that an appliance will have more residual substances such as monomers than a conventional heat-cured denture base polymer. In addition, a high monomer content of the polymer/monomer mix may cause increased contraction on polymerization.

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 2: Orthodontic base polymers

1 Scope

This part of ISO 20795 is applicable to orthodontic base polymers and copolymers used in the construction of both active and passive orthodontic appliances and specifies their requirements. It also specifies 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.

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 1942, *Dentistry — Vocabulary*

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

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

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

ISO 20795-1:2008, *Dentistry — Base polymers — Part 1: Denture base polymers*

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
build up technique
spray on technique
gradual addition of increments of powder and liquid on the master cast until the desired shape is attained

3.3
immediate container
container that is in direct contact with the ⟨orthodontic⟩ base materials

3.4
light activated polymers
products having polymerization initiated by the application of energy from an external radiation source, such as visible light

3.5
liquid
monomeric liquid to be mixed with polymeric particles to form a mouldable dough or fluid resin mixture used for forming ⟨orthodontic⟩ bases

3.6
orthodontic base
polymer part of the ⟨orthodontic⟩ appliance

3.7
outer packaging
labelled container or wrapping within which other containers are packed

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

3.9
processing
procedure of preparing a solid ⟨orthodontic⟩ base polymer plate and/or specimen by polymerization or injection

3.10
thermoplastic material
hard ⟨orthodontic⟩ polymeric material that can be softened by application of heat to make it mouldable, and then returned to the hardened state upon cooling

4 Classification

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

— Type 1: autopolymerizable materials;

— Type 2: light-activated materials;

— Type 3: thermoplastic 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).

5.2 Polymerized material

5.2.1 Biocompatibility

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.

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, orthodontic base polymer test specimens prepared in accordance with 8.5.2 and 8.6.3 shall have a smooth, hard, and glossy surface (see 8.1.1).

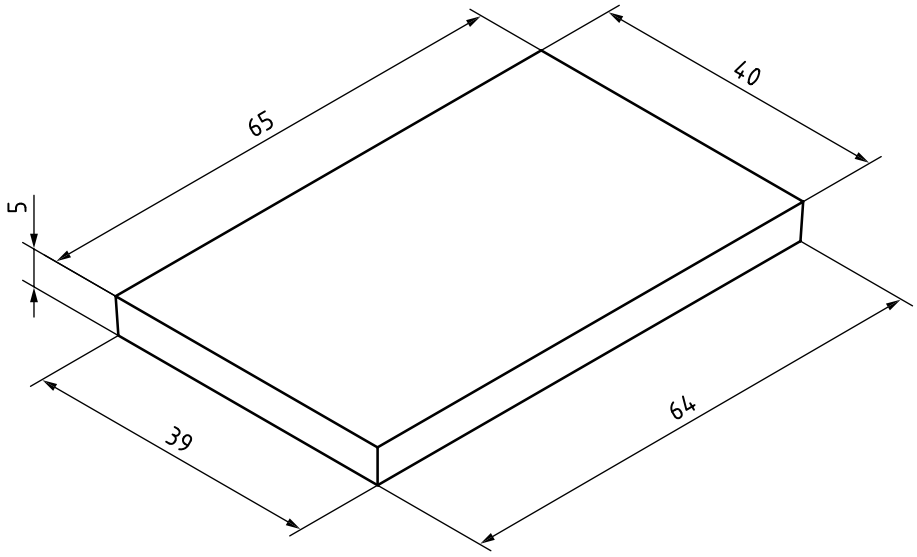
5.2.2.2 The test specimens for residual methyl methacrylate monomer (see 8.5) and the specimens for water sorption and solubility testing (see 8.7) shall retain their form without visible distortion after processing (see 8.1.1).

5.2.2.3 When polished in accordance with 8.3.1.4, 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 orthodontic base polymers shall produce a test specimen plate (see 8.3.1.4) with defined edges and dimensions as given in Figure 1.

Dimensions in millimetres



NOTE Dimensional tolerance shall be ± 1 mm.

Figure 1 — Model of the specimen plate (see 8.3.1.2.1)

5.2.4 Colour

The colour of a test specimen strip prepared in accordance with 8.3.2.3 shall be as stated by the manufacturer when tested and inspected in accordance with 8.1.1 and 8.2.

Coloured orthodontic base polymers shall be evenly pigmented and/or coloured.

Transparent orthodontic base polymers shall be transparent or clear.

5.2.5 Freedom from porosity

When prepared in accordance with 8.3.2.3, the test specimen strips shall not show pores that can be observed by visual inspection (see 8.1.1).

5.2.6 Ultimate flexural strength

When determined in accordance with 8.3.2.5, the ultimate flexural strength shall be not less than 50 MPa (see Table 1).

5.2.7 Flexural modulus

When determined in accordance with 8.3.2.5, the flexural modulus of the processed orthodontic base polymer shall be at least 1 500 MPa (see Table 1).

5.2.8 Maximum stress intensity factor

When determined in accordance with 8.4, the maximum stress intensity factor shall be at least 1,1 $MPa \cdot m^{1/2}$ (see Table 1).

5.2.9 Total fracture work

When determined in accordance with 8.4, the total fracture work shall be at least 250 J/m^2 (see Table 1).

5.2.10 Residual methyl methacrylate monomer

When orthodontic base polymers are prepared and tested in accordance with 8.5, the following shall apply (see Table 1).

The maximum mass fraction of residual methyl methacrylate is 5 % for all three types of orthodontic base polymers.

The residual methyl methacrylate content claimed by the manufacturer [see 9.3 b)] shall not exceed the stated value by more than 0,2 % mass fraction when tested in accordance with 8.5.

5.2.11 Plasticizers

If the orthodontic base polymer contains extractable phthalate plasticizer(s), identify and quantify the plasticizer(s) as percent mass fraction determined in accordance with 8.6. The content shall not exceed the stated value by more than 10 % (see Table 1).

5.2.12 Water sorption

When the processed polymer is tested in accordance with 8.7, the increase in mass per volume (water sorption) shall not exceed 32 $\mu g/mm^3$ (see Table 1).

5.2.13 Water solubility

When the processed polymer is tested in accordance with 8.7, the loss in mass per volume (water solubility) shall not exceed 5 $\mu g/mm^3$ (see Table 1).

Table 1 — Summary of requirements described in 5.2.6 to 5.2.13

Requirements	Flexural properties		Fracture toughness		Residual methyl methacrylate monomer	Phthalate plasticizers	Water sorption	Water solubility
	Ultimate flexural strength	Flexural modulus	Maximum stress intensity factor	Total fracture work				
	σ	E	K_{max}	W_f			w_{sp}	w_{sl}
	MPa min.	MPa min.	MPa $m^{1/2}$ min.	J/m^2 min.	Percent mass fraction max.	Percent mass fraction max.	$\mu g/mm^3$ max.	$\mu g/mm^3$ max.
All types	50	1 500	1,1	250	5	Maximum 10 % above stated value[a]	32	5

[a] For example, if the manufacturer states a percent mass fraction of 5 % of phthalate plasticizers, the content shall not be more than 5,5 %.

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 specimen plates and test specimens

7.1 Laboratory environment

Prepare and test specimens and specimen plates 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 test specimens and specimen plates using the equipment and procedures recommended in the manufacturer's instructions (see 9.3), unless otherwise specified in this part of ISO 20795.

From materials requiring a mixture of two or more ingredients, prepare separate mixes for each test 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 in order to determine compliance with the requirements laid down in 5.1.1.2 and 5.1.2.

Observe the test specimen(s) by visual inspection in order to determine compliance with the requirements laid down in 5.2.2.1, 5.2.2.2, and 5.2.5 and inspect for colour (see 5.2.4) in accordance with ISO 7491.

Observe the test specimen plate(s) by visual inspection in order to determine compliance with the requirements laid down in 5.2.2.3 and 5.2.3.

Inspect visually to determine compliance with Clause 9.

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 orthodontic base polymer 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 Colour

8.2.1 General

Compare a specimen strip prepared in accordance with 8.3.2.3 for compliance with 5.2.4. Inspect its colour visually (see 8.1.1) for compliance with the manufacturer's statement [see 9.2.1 c) and 9.2.2 c)].

8.2.2 Expression of results

Report whether the material passes or fails (see 5.2.4) when tested in accordance with ISO 7491.

8.3 Polishability, freedom from porosity, ultimate flexural strength, and flexural modulus

8.3.1 Polishability

8.3.1.1 Materials

8.3.1.1.1 Wet pumice for polishing, having a grain size of approximately 10 µm to 20 µm.

8.3.1.2 Apparatus

8.3.1.2.1 Model of the specimen plate, in metal or polymer (see Figure 1).

8.3.1.2.2 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.

8.3.1.2.3 Equipment for processing the orthodontic base resin, including gypsum or hydrocolloid investment system [see 9.3 j)].

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

NOTE See ISO 6344-1.

8.3.1.2.5 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.

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

8.3.1.3 Preparation of the mould

For Type 1 and Type 2 polymers, invest the model of the specimen plate (8.3.1.2.1) in the denture flask (8.3.1.2.2) in accordance with the manufacturer's instructions.

8.3.1.4 Procedure

Form and process, according to the manufacturer's instructions, two specimen plates each from a separate mix. Use the material (8.3.1.1), the apparatus (8.3.1.2), and the mould (see 8.3.1.3). Grind and polish the surfaces of the specimen plates for no longer than 1 min with pumice (8.3.1.1.1) and with a wet muslin wheel (8.3.1.2.5) 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 diameter wheel rotating at 3 500 min^{-1} will have a circumferential speed of 1 100 m/min.

Thereafter polish with an unstitched muslin wheel (8.3.1.2.6) with a polishing compound (8.3.1.1.1).

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

8.3.1.4.1 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.

8.3.1.5 Expression of results

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

8.3.2 Freedom from porosity, ultimate flexural strength, and flexural modulus

8.3.2.1 Materials

8.3.2.1.1 Two specimen plates, prepared and tested in accordance with 8.3.1.

8.3.2.2 Apparatus

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

8.3.2.2.2 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.

NOTE A machine with a milling head and a sharp carbide edge is suitable.

8.3.2.2.3 Standard metallographic grinding papers, having a grain size of approximately 30 µm (P500), 18 µm (P1000), and 15 µm (P1200).

NOTE See ISO 6344-1.

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

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

8.3.2.2.6 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 for any load exerted by the deflection instrument when calibrating the machine.

8.3.2.2.7 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.

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

8.3.2.3 Preparation of specimen strips

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 (8.3.2.2.2) 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 (8.3.2.2.3) to the required width and height. Make three measurements of the specimen height along the long axis to an accuracy of ± 0,01 mm using a micrometer and/or dial calliper (8.3.2.2.4). 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.

8.3.2.4 Freedom from porosity

8.3.2.4.1 Procedure and pass/fail determination

Prepare six test specimen strips in accordance with 8.3.2.3 and examine for compliance with 5.2.5.

The material passes only if at least five out of six specimen strips comply with the requirement in 5.2.5.

8.3.2.4.2 Expression of results

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

8.3.2.5 Ultimate flexural strength and flexural modulus

8.3.2.5.1 Procedure

Store five specimen strips, or six in the case of repetition of the test (see 8.3.2.5.2.3 and 8.3.2.5.2.4), prepared in accordance with 8.3.2.3 and complying with 5.2.5, in the container (8.3.2.2.5) 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

(8.3.2.2.7) immersed in the water bath (8.3.2.2.8). 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.

8.3.2.5.2 Calculation and expression of results

8.3.2.5.2.1 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.

8.3.2.5.2.2 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/deflection curve;

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

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

l, b, and h are as defined in 8.3.2.5.2.1.

8.3.2.5.2.3 Pass/fail determination of ultimate flexural strength

If at least four out of five specimens give results not less than 50 MPa, the material is deemed to have complied with the requirements of 5.2.6.

If at least three of the results are less than 50 MPa, the material is deemed to have failed.

If two of the results are less than 50 MPa, repeat the whole test, but on this occasion, prepare six specimen strips.

If at least five of the results are not less than 50 MPa on the second occasion, the material is deemed to have complied with the requirement of 5.2.6.

8.3.2.5.2.4 Pass/fail determination of flexural modulus

If at least four of the results passed the requirement of 5.2.6 on the first occasion, calculate the flexural modulus according to 8.3.2.5.2.2 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 1 500 MPa, the material is deemed to have complied with the requirements of 5.2.7.

If at least three of the results are less than 1 500 MPa, the material is deemed to have failed.

If two of the results are less than 1 500 MPa, 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 5.2.6 and 5.2.7.

8.3.2.5.2.5 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 5.2.6 and 5.2.7, and whether the material passes.

8.4 Fracture toughness with a modified bending test

8.4.1 Materials

8.4.1.1 Two specimen plates, prepared and tested in accordance with 8.3.1.

8.4.1.2 Glycerol, technical grade, used as a lubricant.

8.4.2 Apparatus

8.4.2.1 Apparatus as described in 8.3.2.2.2, 8.3.2.2.3, 8.3.2.2.4, 8.3.2.2.5, 8.3.2.2.8 plus the following.

8.4.2.2 Motorised saw or **other cutting device**, 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.

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

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

8.4.2.5 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).

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

8.4.2.7 Clean, dry towel.

8.4.2.8 Metal flexural test rig, see 8.3.2.2.7, but with a span length, l_t, of (32,0 ± 0,1) mm (see 8.4.4.1).

8.4.2.9 Machine for testing, calibrated to provide 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/deflection 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.

8.4.3 Procedure

At least 24 h from the beginning of the curing cycle, wet-grind or machine the plates (8.4.1.1) in a milling machine (8.3.2.2.2), equally from both mould surfaces, to obtain flat, parallel surfaces, and so that the thickness of the plates remain slightly oversized. Take care to avoid overheating the specimens.

Cut each plate breadthwise with a cutting device (8.4.2.2) in equal specimen strips 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 (8.3.2.2.3) 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 (P1000) or 15 µm (P1200).

Fix the specimens lengthwise in the holding device (8.4.2.3) 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 (8.4.2.2) to a depth of (3,0 ± 0,2) mm along the marked centreline.

Fix one specimen at a time, in a clamp or holding device (8.4.2.3) so that the specimen cannot be removed by hand/machine force. Wet the pre-crack with a drop of glycerol (8.4.1.2). Set the sharp blade (8.4.2.4) 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 (8.4.2.5) 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 2 a). Measure the width, b_t, and the height, h_t, of the specimen with a micrometer (8.3.2.2.4). See Figure 2 b).

Store 10 selected notched specimens in a container with water (8.3.2.2.5) at (37 ± 1) °C for 7 d ± 2 h. Condition the specimens in a different container of water (8.4.2.6) 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 (8.4.2.7). Place the specimen on the supports of the test rig (8.4.2.8). Place the specimen strip with the notch facing exactly opposite the load plunger [see Figure 2 b)]. Be sure that the notch is placed right in the centre between the supports.

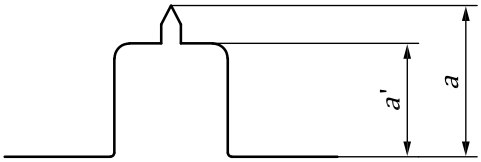
Increase the force of the loading plunger of the testing machine (8.4.2.9) from zero, using a constant displacement rate of (1,0 ± 0,2) mm/min, until maximum load is passed and the crack has almost reached the opposite side of the specimen. The test can be considered finished when the current load is reduced to 5 % of the maximum load or is less than (1,0 ± 0,2) N.

The recording of the whole load/deflection curve is necessary for calculations. Repeat the test for all 10 conditioned specimens.

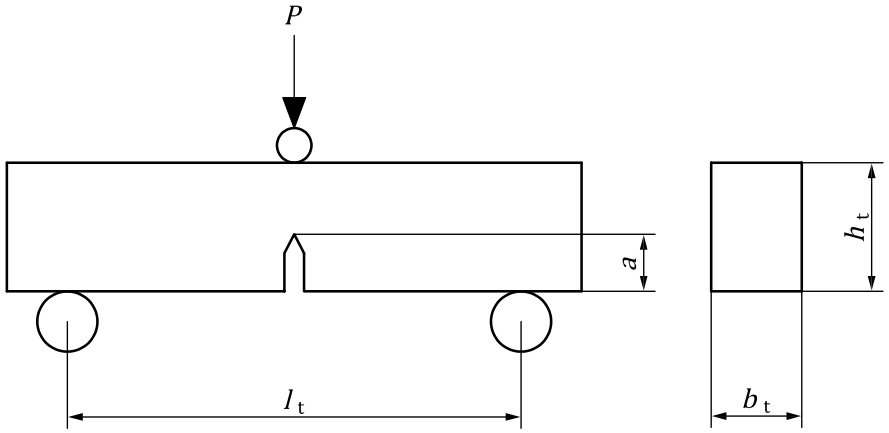
After completing the test, measure the depth of the crack, including the sharp notch, a, in Figure 2, next to the fracture surface with an optical microscope (8.4.2.5).

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 (a_1, a_2, and a_3) 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 3).

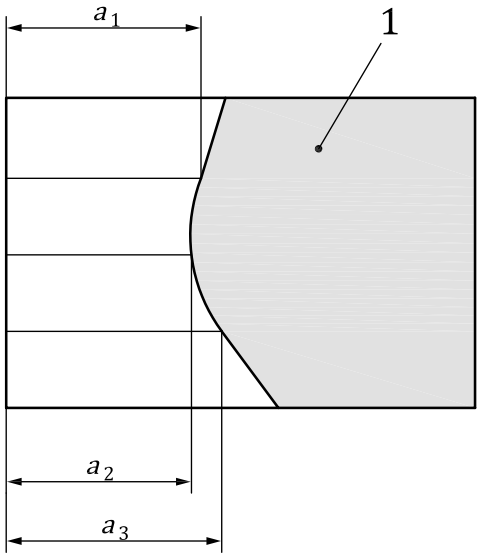


a) Notch arrangement



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

Figure 2 — Fracture toughness test



Key

1 fracture surface

Figure 3 — Determination of the total crack length next to the fracture surface

8.4.4 Calculation and expression of the results

8.4.4.1 Dimensions

Pre-crack	a' = (3,0 ± 0,2) mm
Crack length	a (0,1 mm to 0,4 mm longer than a')
Width	b_t = (4,0 ± 0,2) mm
Height	h_t = (8,0 ± 0,2) mm
Span length	l_t = (32,0 ± 0,1) mm

8.4.4.2 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 expressed in millimetres (see 8.4.4.1).

8.4.4.3 Calculation of the total fracture work

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

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

where

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

$U = \int P d\Delta$ in newton millimetres;

Δ is the measured deflection for load, P;

a, b_t, and h_t are expressed in millimetres (see 8.4.4.1).

NOTE The area under the load/deflection 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.

8.4.4.4 Pass/fail determination of maximum stress intensity factor

If at least eight of the results from 10 specimens are not less than 1,1 MPa $m^{1/2}$, the material complies with the requirements of 5.2.8.

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

If three, four, or five of the results are less than 1,1 MPa $m^{1/2}$, repeat the whole test, but on this occasion, prepare 12 specimen strips.

If at least 10 of the 12 results are not less than 1,1 MPa $m^{1/2}$ on the second occasion, the material complies with the requirement of 5.2.8.

8.4.4.5 Pass/fail determination of total fracture work

If at least eight of the results from 10 specimens are not less than 250 J/m^2, the material complies with the requirements of 5.2.9.

If at least six of the results are less than 250 J/m^2, the material is deemed to have failed.

If three, four, or five of the results are less than 250 J/m^2, repeat the whole test, but on this occasion, prepare 12 specimen strips.

If at least 10 of the 12 results are not less than 250 J/m^2 on the second occasion, the material is deemed to have complied with the requirement of 5.2.9.

8.4.4.6 Expression of results

Report for 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 5.2.8 and 5.2.9, and whether the material passes.

8.5 Residual methyl methacrylate monomer

8.5.1 Principle

Solvent extraction of the methyl methacrylate (MMA) monomer from polymerized orthodontic base polymers 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.

8.5.2 Preparation of test specimen discs

8.5.2.1 Material

8.5.2.1.1 Sheet of polyester film, having a thickness of (50 ± 25) µm, to cover the steel mould (8.5.2.2.1).

8.5.2.2 Apparatus

8.5.2.2.1 Circular stainless steel mould, 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 of ISO 20795-1:2008. Mount the mould in gypsum in separate halves of a denture flask.

8.5.2.2.2 Moulds and/or equipment, recommended by the manufacturer to produce specimens with the dimensions specified in 8.5.2.2.1.

8.5.2.2.3 Standard metallographic grinding papers, with a grain size of approximately 30 µm (P500) and 15 µm (P1200). See Note of 8.3.1.2.4.

8.5.2.2.4 Micrometer screw gauge, accurate to 0,01 mm.

8.5.2.2.5 Dial gauge calliper or **slide calliper**, accurate to 0,01 mm and fitted with parallel anvils.

8.5.2.3 Procedure

Prepare three specimens from three separate mixes. Mix the resin and pack the mixture into the mould (8.5.2.2.1 or 8.5.2.2.2) with the polyester film (8.5.2.1.1) against the cover of the mould. Process the mixture in accordance with the manufacturer's instructions, but retain the polyester film during the processing cycle. 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 (8.5.2.2.3) in turn, to wet-grind material equally (approximately 0,5 mm) from both sides of the specimen disc. Grind the periphery of the specimens against the 15 µm grain metallographic grinding paper until the entire periphery is abraded and smooth. Check with a micrometer or dial calliper (8.5.2.2.4 or 8.5.2.2.5) to ensure that each specimen has a diameter of (50 ± 1) mm and a thickness of (2,0 ± 0,1) mm and that the top and bottom surfaces are flat. 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 a refrigerator, the monomer content remains constant for several days. If the specimens are stored in a 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 monomer.

8.5.3 Extraction of monomer

8.5.3.1 Reagents

8.5.3.1.1 Hydroquinone (HQ).

8.5.3.1.2 Acetone, purity of analytical or HPLC grade.

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

8.5.3.1.4 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.

If the manufacturer has indicated that plasticizer(s) are present additional I.S.(s) may be introduced, the peak of each chosen additional I.S. shall not interfere with any other peak in the sample solution.

8.5.3.2 Apparatus

Ordinary laboratory apparatus and the following:

8.5.3.2.1 Closable one-mark volumetric glass flasks, of capacities 5 ml, 10 ml, and 1 l.

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

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

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

8.5.3.2.5 Glass pipettes.

8.5.3.2.6 Closable glass centrifugation tubes.

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

8.5.3.2.8 Closable glass tubes.

8.5.3.3 Preparation of solutions

8.5.3.3.1 Acetone solution (A)

Weigh approximately 0,02 g HQ (8.5.3.1.1) into a 1 l one-mark volumetric glass flask (8.5.3.2.1). Add acetone (8.5.3.1.2) until the total volume is 1 l.

8.5.3.3.2 Methanol solution (B)

Weigh approximately 0,02 g HQ (8.5.3.1.1) into a 1 l one-mark volumetric glass flask (8.5.3.2.1). Add methanol (8.5.3.1.3) until the total volume is 1 l.

8.5.3.3.3 Methanol/acetone solution (C)

Mix one volume part of solution A (8.5.3.3.1) and four volume parts of solution B (8.5.3.3.2).

8.5.3.3.4 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 for MMA quantification, weigh approximately 350 mg I.S. (8.5.3.1.4) into a 10 ml one-mark volumetric glass flask (8.5.3.2.1).

NOTE If the manufacturer has indicated that plasticizers are present [see 9.3 d)], additional I.S.(s) can be introduced. The mass of additional I.S.(s) is dependent of the mass fraction of plasticizer(s) indicated by the manufacturer.

Add the methanol solution (B) (8.5.3.3.2) 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. (for MMA quantification) 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) (8.5.3.3.1) and the methanol solution (B) (8.5.3.3.2).

8.5.3.3.5 Sample solutions

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

Break each specimen disc (8.5.2) into pieces small enough to pass through the neck of the one-mark 10 ml volumetric glass flasks (8.5.3.2.1). Introduce a sample size of approximately 650 mg into separate one-mark 10 ml volumetric glass flasks (8.5.3.2.1). Weigh the mass with an analytical balance (8.5.3.2.2) and record for each individual sample solution. Add the acetone solution (A) (8.5.3.3.1) until the total volume is 10 ml and then introduce a clean PTFE coated magnetic stirring bar 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 apparatus (8.5.3.2.3) for (72 ± 2) h at room temperature.

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 (8.5.3.2.4) to transfer a 2 ml aliquot of each previously prepared sample solution to each separate one-mark 10 ml volumetric glass flask.

Then add 100 µl of the I.S. solution (8.5.3.3.4) to each flask. Add methanol solution (B) (8.5.3.3.2) to each of these sample solutions to a total volume of 10 ml.

Use separate glass pipettes (8.5.3.2.5) to transfer approximately 5 ml of the polymer and monomer containing slurry from each of the 10 ml flasks to separate glass centrifugation tubes (8.5.3.2.6).

Centrifuge the slurry at 3 000g_n m/s^2 for 15 min in a centrifuge (8.5.3.2.7).

Use separate glass pipettes to transfer approximately a 3 ml aliquot of each centrifuged solution to separate glass tubes (8.5.3.2.8).

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 8.5.1).

8.5.4 Gas chromatography

8.5.4.1 Reagent

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

8.5.4.2 Apparatus

8.5.4.2.1 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.

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

8.5.4.3 Preparation of calibration solutions for gas chromatography

Make at least five standard solutions with concentrations of MMA (8.5.4.1.1), 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 (8.5.4.1.1) into separate one-mark volumetric glass flasks of capacity 5 ml (8.5.3.2.1). Add solution C (8.5.3.3.3) until the total volume is 5 ml. Transfer 100 µl of each calibration solution into separate 10 ml one-mark volumetric glass flasks (8.5.3.2.1) together with 100 µl of the I.S. solution (8.5.3.3.4); add solution C (8.5.3.3.3) 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 8.5.3.3.5) does not fit within the extreme MMA concentrations of the calibration graph (see 8.5.5.1.1), make additional calibration points.

8.5.4.4 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.

8.5.4.5 Gas chromatograms of sample and calibration solutions

Depending on the sensitivity of the gas chromatograph used, inject a suitable volume of sample solution (prepared in accordance with 8.5.3.3.5) or the calibration solution (prepared in accordance with 8.5.4.3). 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.

8.5.4.6 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.

8.5.5 Calculation and expression of results

8.5.5.1 Calculation of results from a calibration graph

8.5.5.1.1 Drawing of the calibration graph

Draw a calibration graph by plotting the ratios of the peak area (or height) of methyl methacrylate monomer and the internal standard in the calibration solutions against the respective concentrations of methyl methacrylate expressed in micrograms per millilitre.

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

where

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

$A'_{\mathrm{I.S.}}$ is the peak area (or height) of internal standard (8.5.3.1.4) in the calibration solution.

8.5.5.1.2 Precision of measurements

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

8.5.5.1.3 Determination of the percentage of methyl methacrylate

Use the following ratio to determine the concentration of MMA in the sample solution:

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

where

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

$A_{\mathrm{I.S.}}$ is the peak area (or height) of internal standard (8.5.3.1.4) in the sample solution.

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

The total quantity of MMA in the sample solution, m_{MMA}, in micrograms, is calculated using the following equation:

$$m_{\mathrm{MMA}} = \left(c_{\mathrm{MMA}} \times \frac{10}{2} \times 10 \right)$$

NOTE 1 $\times\frac{10}{2}$: 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 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 $\times 10$: The volume of the original sample solution was 10 ml.

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

where

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

8.5.5.2 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.

8.5.5.3 Expression of results

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

8.6 Plasticiser(s), where applicable

8.6.1 Principle

Solvent extraction of phthalate plasticizer(s) from polymerized orthodontic base polymers is carried out, followed by chromatographic analyses.

A gas chromatographic (GC) method or any other chromatographic method can be used, which gives the same results as with the method of this part of ISO 20795. Verify the results by the GC method described in this part of ISO 20795.

8.6.2 General

The manufacturer shall clearly indicate and identify phthalate plasticizer(s) present in the polymerized material [see 9.3 d)].

8.6.3 Preparation of test specimen discs

See 8.5.2.

8.6.4 Extraction of plasticizer(s)

See 8.5.3.

8.6.4.1 Reagents

Reagents as described in 8.5.3.1 plus the following.

8.6.4.1.1 Phthalate plasticizer(s), indicated by the manufacturer.

8.6.4.1.2 Internal standard(s), purity of analytical grade, whose peak does not interfere with any other peak in the sample solution and which is suitable for quantification of phthalate plasticizer(s).

8.6.4.2 Apparatus

See 8.5.3.2.

8.6.4.3 Preparation of solutions

Solutions as described in 8.5.3.3 for preparation of acetone solution (A), methanol solution (B), and methanol/acetone solution (C) plus the following.

8.6.4.3.1 Internal standard (I.S.) solution

Internal standards that elute near the peak(s) of phthalate plasticizer(s) are preferable.

If additional I.S.(s) is/are not introduced in 8.5.3.3.4, prepare an internal standard solution in order to be able to quantify phthalate plasticizer(s) in orthodontic base polymers.

8.6.4.3.2 Sample solutions

Prepare sample solutions in accordance with 8.5.3.3.5 except that the I.S. solution added to the sample solutions shall be suitable for quantification of phthalate plasticizer(s).

Solutions prepared in accordance with 8.5.3.3.5 for quantification of residual monomer, where an additional I.S. is introduced into the solutions in order to quantify both residual monomer and plasticizers, can be used.

8.6.5 Gas chromatography

8.6.5.1 Reagent

8.6.5.1.1 Phthalate plasticizer(s) indicated by the manufacturer [see 9.3 d)], GC-purity > 99 %.

8.6.5.2 Apparatus

See 8.5.4.2.

8.6.5.3 Preparation of calibration solutions for gas chromatography

Make at least five standard solutions with concentrations of plasticizer(s) appropriate to quantify the sample solutions. Prepare calibration solutions of phthalate plasticizer(s) by weighing into separate one-mark volumetric glass flasks of capacity 5 ml (8.5.3.2.1). Add solution C (8.5.3.3.3) until the total volume is 5 ml. Transfer 100 µl of each calibration solution into separate 10 ml one-mark volumetric glass flasks (8.5.3.2.1) together with 100 µl of the I.S. solution (8.6.4.3.1). Add solution C (8.5.3.3.3) until the total volume is 10 ml.

Record the mass of phthalate plasticizer(s) for each individual calibration solution and calculate the final concentrations, in micrograms per millilitre.

If the plasticizer content of the sample solutions (8.6.4.3.2) does not fit within the extreme phthalate plasticizer concentrations of the calibration graph (see 8.6.6.1.1), make additional calibration points.

8.6.5.4 Gas chromatographic equipment, gases, and operating conditions

See 8.5.4.4.

The conditions can be altered in order to be suitable for quantification of the phthalate plasticizer(s).

Use an isothermal period where both MMA and I.S. (chosen for quantification of MMA) elute, then raise the column temperature until a temperature suitable for quantification of the phthalate plasticizer(s) is reached. The plasticizer(s) should elute in a new isothermal period together with the I.S. chosen for quantification of the phthalate plasticizer(s).

8.6.5.5 Gas chromatograms of sample and calibration solutions

Depending on the sensitivity of the gas chromatograph used, inject a suitable volume of sample solution (prepared in accordance with 8.6.4.3.2) or the calibration solution (prepared in accordance with 8.6.5.3). 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 phthalate plasticizer(s) content in the sample solutions, and ensure good separation of all substances by using appropriate column oven temperature profiles.

8.6.5.6 Evaluation of peaks of gas chromatograph

Identify the phthalate plasticizer(s) in the sample solution by chromatographic methods. Determine the retention times of phthalate plasticizer(s) and the I.S.(s) chosen, at least in relation to each other. Determine the peak area(s) or height(s) of phthalate plasticizer(s) and I.S.(s) by electronic registration and integration.

8.6.6 Calculation and expression of results

8.6.6.1 Calculation of results from calibration graph(s)

8.6.6.1.1 Drawing of calibration graph(s)

Draw a calibration graph for each individual phthalate plasticizer by plotting the ratio of the peak area (or height) against the concentrations:

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

where

$A'_{\text{PLASTICIZER}}$ is the peak area (or height) of the phthalate plasticizer in the calibration solution;

$A'_{\text{I.S.}}$ is the peak area (or height) of internal standard (8.6.4.1.2) in the calibration solution.

8.6.6.1.2 Precision of measurements

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

8.6.6.1.3 Determination of the percentage of phthalate plasticizer(s)

Determine the percentage of phthalate plasticizer(s) with the corresponding ratio:

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

where

$A_{\text{PLASTICIZER}}$ is the peak area (or height) of the phthalate plasticizer in the sample solution;

$A_{\text{I.S.}}$ is the peak area (or height) of internal standard (8.6.4.1.2) in the sample solution.

Use the calibration graph(s) to determine the concentration of each phthalate plasticizer, $c_{\text{PLASTICIZER}}$, in micrograms per millilitre in the analysed sample solution.

The quantity of each individual phthalate plasticizer in the sample solution, $m_{\text{PLASTICIZER}}$, in micrograms is calculated using the following equation:

$$m_{\text{PLASTICIZER}} = \left(c_{\text{PLASTICIZER}} \times \frac{10}{2} \times 10 \right)$$

NOTE 1 $\times\frac{10}{2}$: 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 (100 µl of an additional I.S. may also be added) in a volumetric 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 $\times 10$: The volume of the original sample solution was 10 ml.

The quantity of each individual phthalate plasticizer in the sample solution is summarized and divided with the original mass of each individual sample.

Percent mass fraction of phthalate plasticizer for each individual sample is given by:

$$\text{Phtalate plasticizer (\% mass fraction)} = \frac{m_{\text{TPC}}}{m_{\text{SAMPLE}}} \times 100$$

where

m_{TPC} is the total phthalate plasticizer content, in micrograms;

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

8.6.6.2 Pass/fail determination

If results obtained for at least seven of the sample solutions comply with the requirements stated in 5.2.11, the material passes.

If four or fewer of the sample solutions comply with the requirement stated in 5.2.11, 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 5.2.11, the material passes.

8.6.6.3 Expression of results

Report the number of sample solutions evaluated, all results for total extractable phthalate plasticizer(s) content, and whether the material passes.

8.7 Water sorption and solubility

8.7.1 Materials

8.7.1.1 Sheet of polyester film, having a thickness of (50 ± 25) µm, to cover the steel mould (8.7.2.1).

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

8.7.1.3 Water, complying with grade 2 of ISO 3696.

8.7.2 Apparatus

8.7.2.1 Circular stainless steel mould and cover, having the dimensions shown in Figure 2 of ISO 20795-1:2008, mounted in gypsum in separate halves of a denture flask.

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

8.7.2.3 Micrometer screw gauge, accurate to 0,01 mm.

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

8.7.2.5 Rack, to keep the specimens parallel and separated.

8.7.2.6 Two desiccators.

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

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

8.7.2.9 Water bath, capable of maintaining constant temperatures, where applicable.

8.7.2.10 Tweezers, polymer coated.

8.7.2.11 Clean dry towel.

8.7.2.12 Timer, accurate to 1s.

8.7.3 Preparation of test specimens

Make five specimens from separate mixes. Mix the resin and pack the mixture into the mould (8.7.2.1) with the polyester film (8.7.1.1) 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.

Check, using a micrometer or dial calliper (8.7.2.3 or 8.7.2.4), 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.

8.7.4 Procedure

8.7.4.1 Conditioned specimens

Place the specimens in the rack (8.7.2.5) inside one of the desiccators (8.7.2.6) containing freshly dried silica gel (8.7.1.2). Store the desiccator in the oven (8.7.2.7) 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 (8.7.2.8) 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.

8.7.4.2 Wet specimens

Immerse the conditioned specimens in water (8.7.1.3) at (37 ± 1) °C for 7 d ± 2 h. After this time, remove the discs from the water using polymer coated tweezers (8.7.2.10), wipe with a clean, dry towel (8.7.2.11) 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.

8.7.4.3 Reconditioned specimens

After this weighing, recondition the specimens to constant mass in the desiccator as described in 8.7.4.1. 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 8.7.4.1), using the same number of specimens and the freshly dried silica gel in the desiccators.

8.7.5 Calculation and expression of results

8.7.5.1 Water sorption

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

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

where

m_2 is the mass of the specimen (see 8.7.4.2) in micrograms, after immersion in water;

m_3 is the reconditioned mass of the specimen (see 8.7.4.3) in micrograms;

V is the volume of the specimen (see 8.7.4.1) in cubic millimetres.

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

8.7.5.2 Water solubility

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

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

where

m_1 is the "conditioned" mass of the specimen (see 8.7.4.1), in micrograms;

m_3 and V are as given in 8.7.5.1.

Round off the values calculated for water solubility to the nearest 0,1 µg/mm^3.

8.7.5.3 Pass/fail determination of water sorption

If at least four of the water sorption results comply with the requirement stated in 5.2.12, the material passes.

If at least three of the water sorption results do not comply with the requirement stated in 5.2.12, the material is deemed to have failed.

If only three of the water sorption results comply with the requirement stated in 5.2.12, 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 5.2.12, the material is deemed to have passed.

8.7.5.4 Pass/fail determination of water solubility

If at least four of the water solubility results comply with the requirement stated in 5.2.13, the material passes.

If at least three of the water solubility results do not comply with the requirement stated in 5.2.13, the material is deemed to have failed.

If only three of the water solubility results comply with the requirement stated in 5.2.13, prepare and test a series of six additional specimens. If at least five of the water solubility results of the second series comply with the requirement stated in 5.2.13, the material is deemed to have passed.

8.7.5.5 Expression of results

Report the number of specimens evaluated, all results for water sorption and water solubility with the number of specimens complying with the requirements of 5.2.12 and 5.2.13, and whether the material passes.

9 Requirements for labelling, marking, packaging, and instructions supplied by manufacturer

9.1 Packaging

The material shall be supplied in properly sealed containers made of materials that neither contaminate nor allow contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during transit and storage. The liquid shall be contained in a dark-coloured bottle or opaque container. An outer package may be used to present one or more immediate containers for retail marketing.

9.2 Marking of outer packages and containers

9.2.1 Outer packages

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

a) the trade or brand name of the material;

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

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

d) expiry date (year and month) expressed in accordance with ISO 8601;

e) recommended conditions of storage;

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

g) cautionary statements with regard to flammability and flashpoint of the liquid (when applicable);

h) cautionary statements with regard to toxic, hazardous, or irritating characteristics;

i) identification of any pharmaceutically active ingredients present in the material and referred to in product information and instructions provided by the manufacturer;

j) the manufacturer's batch reference.

9.2.2 All immediate containers

All immediate containers shall be clearly marked with the following information:

a) the trade or brand name of the material;

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

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

d) expiry date (year and month) expressed in accordance with ISO 8601;

e) recommended conditions of storage;

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

g) cautionary statements with regard to flammability and flashpoint of the liquid (when applicable);

h) cautionary statements with regard to toxic, hazardous, or irritating characteristics;

i) identification of any pharmaceutically active ingredients present in the material and referred to in product information and instructions provided by the manufacturer;

j) the manufacturer's batch reference.

9.2.3 Container of liquid

The flashpoint of the liquid shall be clearly marked on each container of liquid.

9.3 Manufacturer's instructions

The instructions needed for safe and effective use of the material shall be included in each individual package. All processing methods described in the manufacturer's instructions shall result in an orthodontic base polymer that fulfils the requirements laid down in this part of ISO 20795. As a minimum they shall include (as applicable) the following information:

a) details of the composition of the material;

b) maximum residual monomer content (percent mass fraction) when tested in accordance with 8.5;

c) if a residual monomer content is claimed to be less than 1 % mass fraction, the curing process necessary to achieve this result shall be given;

d) in clear language, the identity and maximum quantity of extractable phthalate plasticizer(s) (percent mass fraction) used in the polymerized material;

e) recommended storage conditions for unprocessed material;

f) cautions against prolonged skin contact with the unpolymerized gel or liquid and against inhalation of the monomer;

g) if applicable, powder/liquid ratio (mass per unit volume or mass fraction);

h) if applicable, time, temperature, and procedures to prepare the material for packing;

i) if applicable, time over which packing may be effectively conducted;

j) equipment and material needed to prepare the mould (e.g. type of flask, gypsum, hydrocolloid investment system);

k) recommended separation media;

l) temperature of the flask during packing;

m) detailed procedure for activating and completing polymerization of the material;

n) post-processing treatment of the processed material (cooling and storage after deflasking).

Annex A
(normative)

HPLC method for determination of MMA content

A.1 General

Several items needed for the HPLC (high performance liquid chromatography) method are identical to some of those required for use in the GC (gas chromatography) method (see 8.5).

A.2 Preparation of test specimens

See 8.5.2.

A.3 Extraction of monomer

A.3.1 Reagents

Reagents as described in 8.5.3.1 plus the following.

A.3.1.1 Tetrahydrofurane (THF), of analytical or HPLC grade.

A.3.1.2 Water, complying with grade 2 of ISO 3696.

A.3.2 Apparatus

See 8.5.3.2.

A.3.3 Preparation of solutions

See 8.5.3.3.

NOTE THF can be substituted for acetone. An internal standard (I.S.) solution (see 8.5.3.3.4) is not required. Therefore, the addition of the I.S. to the sample solutions (see 8.5.3.3.5) and the addition of the I.S. to the calibration solutions (see 8.5.4.3) is not required.

A.3.4 High performance liquid chromatography (HPLC)

A.3.4.1 Reagent

See 8.5.4.1.

A.3.4.2 Apparatus

A.3.4.2.1 High performance liquid chromatograph, with ultraviolet spectroscopy detector capable of measuring at 205 nm (or an equivalent detector) and a recording system.

A.3.4.2.2 Injection loop, of capacity e.g. 20 µl.

A.3.4.3 Preparation of calibration solutions

See 8.5.4.3, except that I.S. is not required and that THF can be substituted for acetone.

A.3.4.4 HPLC equipment and operating conditions

a) Column: octadecyl silanized, 5 µm particle size, 250 mm length, and 4 mm to 5 mm internal diameter.

b) Mobile phase: 66 % CH_3OH:34 % H_2O, isocratic elution.

c) Flowrate: 0,8 ml/min.

d) Detection: UV wavelength 205 nm.

e) Temperature: constant room temperature.

NOTE The operating conditions can be altered if satisfactory separation is achieved. A different mobile phase system, e.g. acetonitrile/water (CH_3CN/H_2O), can be used if separation is satisfactory.

A.3.4.5 HPLC chromatograms of sample and calibration solutions

The wavelength of 205 nm is suitable for low concentrations of MMA in the sample solution. The calibration graph shall be linear. If the concentration of the sample solution is too high, quantitative dilution of sample and calibration solutions is required, or a different choice of wavelength, e.g. 225 nm, can be made.

To ensure that a constant volume of the sample solutions and the calibration solutions is injected, a loop with a fixed volume (e.g. 20 µl) is used.

To ensure correct quantification of the MMA content in the sample solutions, good separation of all substances shall be secured by selecting an appropriate mobile-phase composition.

Operate the high performance liquid chromatograph (A.3.4.2.1) until all components are completely eluted.

A.3.4.6 Evaluation of peaks from HPLC chromatograms

Determine the retention time of MMA. The retention time shall be stable during the analyses of sample solutions and calibration solutions. The retention time is dependent upon the column and mobile phase composition.

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

A.3.5 Calculation and expression of results

A.3.5.1 Calculation of results from a calibration graph

A.3.5.1.1 Calibration graph drawing

Draw a calibration graph by plotting the peak area (or height) of methyl methacrylate monomer in the calibration solution against the respective concentrations of MMA expressed in micrograms per millilitre.

A.3.5.1.2 Precision of measurements

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

A.3.5.1.3 Determination of the percentage of methyl methacrylate

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

Total quantity of MMA in the sample solution, m_{MMA}, in micrograms is calculated as in 8.5.5.1.3.

A.3.5.2 Pass/fail determinations

See 8.5.5.2.

A.3.5.3 Expression of results

See 8.5.5.3.

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