INTERNATIONAL STANDARD

ISO XXXXX

Dentistry — Extraoral maxillofacial prosthetic materials

Médecine bucco-dentaire — Produits prothèses maxillofaciales extraorales matériel

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials,* Working Group 2.16 on Extraoral Maxillofacial Prosthetic Materials.

This is the first edition of this draft standard and is drawn from several documents with properties similar to existing facial prosthetic materials. But in order to stimulate innovation in the future, the properties in this draft standard are broadly established, to be refined on revisions if necessary. It is also intended to harmonize the definition of long-term use with other international definitions.

Introduction

Extraoral maxillofacial prosthetic materials are for manmade replacement of facial and other human body parts lost due to cancer, trauma, or birth defects. They may be applied, or color corrected directly on the patient, or more commonly, fabricated indirectly in a laboratory. Most are elastic in nature, so this first draft is written as such. Although it is not claimed that any particular level of softness is superior to another, this classification is intended to assist clinicians because clinicians will have more information with which to make an informed choice.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this part of ISO 10139. Information relevant to assessing possible biological or toxicological hazards is given in ISO 7405 and ISO 10993-1.

2 Normative references

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 7619-1, *Rubber, vulcanized or thermoplastic — Determination of indentation hardness — Part 1: Durometer method (Shore hardness)*

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

ISO 10139-2, 3rd ed., 2016-06-15, *Dentistry - Soft lining materials for removable dentures -Part 2: Materials for long-term use)*

Revised American National Standard/ American Dental Association Standard No. 99, *Athletic Mouth Protectors and Materials, 2013.*

ANSI/ADA Standard No. 128/ISO 21563:2013, *Hydrocolloid impression materials,* 2015.

3. Terms and definitions

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

4. Requirements

5.1. Durometer Shore A hardness, 24 h

When 24 h test specimens are subjected to a 5 s Durometer Shore A hardness test in accordance with 7.2.3.2, the material shall conform to the requirements for the relevant type as shown in Table 1. For a material to be classified as a particular type, the mean Shore A hardness for at least two of the three specimens shall conform to the requirements for that type, as specified in Table 1. If the results for two or more specimens are greater than 50, the material shall be deemed not to conform to this part of ISO XXXXX.

Table 1 — Durometer Shore A hardness, 24 h – 5 s

Type	Durometer Shore A
A (soft)(feather edges)	5 ≤ Shore A ≤ 40

B (medium soft)(skin over cartilage)	Shore A 41 ≤ 50

5.2. Test methods

5.3. Test conditions

Unless specified otherwise by the manufacturer, prepare and test all specimens at a temperature of 23 **±** 2 °C. Measurement equipment shall be used in a calibrated condition.

5.4. Apparatus. Shore A hardness equipment, corresponding to ISO 7619-1 with a precision of ±1 HS. Shore Type "A" durometer is required to measure hardness (Shore Instrument and Mfg. Co.; Jamaica, N.Y.). The instrument has a blunt-pointed indenter 0.8 mm in diameter that tapers to a cylindrical shaft of 1.6 mm. The indenter is attached by a lever to a pointer that indicates the hardness on a scale of 0 to 100 durometer.

5.5. Mould, suitable for producing test specimens of at least 35 mm diameter and at least 6 mm thick, constructed using a smooth metal or polymer disc as a template. A mould release agent, *e.g.,* polytetrafluoroethylene (PTFE) spray, may be used to avoid the adherence of material.

5.6. Timing device, accurate to 0.1 s.

5.7. Preparation of test samples. Prepare each test specimen in the mould cavity in accordance with the manufacturer's instructions. Prepare three test specimens.

5.8. Procedure

5.8.1. General. Carry out the test procedure in accordance with 7.2.3.2, 7.2.3.3 and ISO 7619-1 on each of the three test specimens. For the measurements, place the specimens on a flat and solid base and lower the Shore A hardness tester (7.2.1.1) gradually onto the surface of the specimen in such a way that the indenter foot just touches the specimen surface. The surface of the specimens and the contact surface of the Shore A hardness tester shall be coplanar. Ensure that the indenter is normal to the specimen surface. Five measurements shall be made for each of the specimens at each testing time. The loading points shall be uniformly distributed on the surface and shall have a distance of at least 5 mm from the edge of the specimen.

5.8.2. Durometer Shore A hardness test, 24 h specimen. After preparation, measure the Shore A durometer hardness immediately. Record the values 5 s after loading, using a timing device (7.2.1.4). Make all recordings within (2 ± 1) min. Calculate the mean of the five Shore A values for each of the three specimens (results a, b and c).

5.8.2. Expression of results. Record the test results for each of the three specimens in the format illustrated in Table 2.

Table 2 — Durometer Shore A hardness

Age of specimen	Mean Shore A of specimen		
	1	2	3
24 h	a	b	c

6. Water sorption and solubility.

6.1. Materials.

6.1.2. Sheet of polyester film, having a thickness of 50 ± 25 µm to cover the steel mould (6.1.5.1).

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

6.1.4. Water, complying with grade 2 of ISO 3696.

6.1.5. Apparatus

6.1.5.1. Circular stainless steel mould and cover, having the dimensions shown in Figure 1, mounted in gypsum in separate halves of a denture flask.

6.1.5.2. Hydraulic or hand press and clamp, where applicable.

6.1.5.3. Micrometer or dial caliper, accurate to 0.01 mm and fitted with parallel anvils.

6.1.5.4. Rack, to keep the specimens parallel and separated.

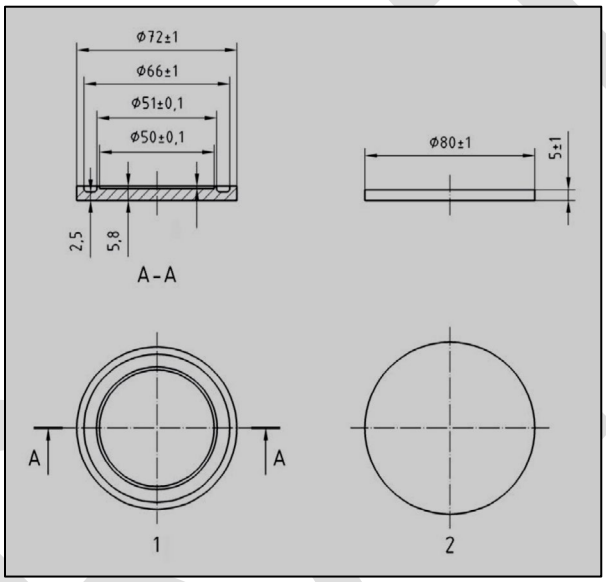
6.1.5.5. Two desiccators.

6.1.5.6. Oven, maintained at 37 ± 1 °C.

6.1.5.7. Polymer-coated tweezers.

6.1.5.8. Towel, clean and dry.

6.1.5.9. Analytical balance, accurate to 0.01 mg.



Key
1 mould
2 cover
Mould depth: 0.5 ± 0.05 mm to form specimen.

Figure 1. — Stainless steel mould and cover for specimen preparation for water sorption and solubility

6.2. Preparation of test specimens. Make five specimens from separate materials or mixes. Pack the material into the mould (6.1.5.1) with the polyester film (6.1.2) 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 with a micrometer or dial calliper (6.1.5.3) 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.

6.3. Procedure.

6.3.1. Conditioned specimens. Place the specimens in the rack (6.1.5.4) inside one of the desiccators (6.1.5.5) containing freshly dried silica gel (6.1.3). Store the desiccator in the oven (6.1.5.6) 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 (6.1.5.9) to weigh the specimen to an accuracy of 0,1 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, measure the diameter and the thickness of each specimen to an accuracy of 0.01 mm. 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.

6.3.2. Wet specimens

Immerse the conditioned specimens in water (6.1.4) at 37 ± 1 °C for 7 d ± 2 h. After this time, remove the discs from the water with polymer-coated tweezers (6.1.5.7), wipe with a clean, dry towel (6.1.5.8) 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 0.1 mg). Record the mass as m_2.

6.3.3. Reconditioned specimens. After this weighing, recondition the specimens to constant mass in the desiccator as described in 6.3.1. 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, using the same number of specimens and the freshly dried silica gel in the desiccators.

6.3.4. Calculation and expression of results.

6.3.4.1. Water sorption. Calculate the value for the water sorption, w_{sp}, for each of the five specimens, expressed in milligrams per cubic meter (mg/m^3) [micrograms per cubic millimetre (µg/mm^3], using Formula (1):

$$w_{sp} = m_2 - m_3 / V \qquad (1)$$

where

m_2 is the mass of the specimen, in milligrams (mg), after immersion in water (see 6.3.2)

m_3 is the reconditioned mass of the specimen, in milligrams (mg) (see 6.3.3);

V is the volume of the specimen, in cubic millimeters (mm^3) (see 6.3.4.1).

6.3.4.2. **Water solubility.** Calculate the soluble matter per unit volume, w_{sl}, leached out during immersion, expressed in milligrams per cubic metre (µg/mm^3), for each of the five specimens using Formula (2):

$$w_{sl} = m_1 - m_3 / V \qquad (2)$$

where

m_1 is the conditioned mass of the specimen, in milligrams (mg), after immersion in water (see 6.3.3)

m_3 and V are as given in Formula 1.

Round off the values calculated for water solubility to the nearest 0.1 mg/m^3 (µg/mm^3).

7. **Tear strength test**

7.1 **Apparatus and material** .

a) **Specimen sheet forming mold** (Figure A.9) having a depth that will provide for a specimen thickness of 4.0 + 0.5 mm

NOTE The specimen thickness may vary within the stated tolerance depending upon the capacity of the gripping mechanism available for the test. Use of the optional tear test specimen preparation procedure described in Annex A for fitting the test specimen for gripping in the test instrument will allow accommodation of any specimen thickness within the specified tolerance.

b) **Polyethylene sheets**, approximately 0.035 mm thick and having length/width dimensions approximating those for the mold cavity floor and the mold cover, (Figure A.9)

c) **High vacuum grease**, such as a silicone grease [7.1.1 b)]

d) **Oven**, held at 35+2 °C.

e) **Two water baths** {7.3.1 h)]

f) **Specimen sheet support pad**, on which to place the formed specimen sheet during use of the die [7.7.1g)] for precision cut-out of the specimen. Length/width dimensions of the pad shall approximate those of the specimen forming sheet.

NOTE: The pad, which may consist of layers of water proof paper, or polymer or wax sheets, may need to vary in thickness depending upon resistance to cutting exhibited by the specimen sheet and pad forming materials.

g) ASTM D624, Die C, for cutting specimens to dimensions shown in Figure A.8

NOTE Satisfactory tear strength test specimens, such as depicted in Figure A.8, can also be produced using a machined or molded polymeric specimen forming mold plate.

h) Specimen thickness measuring instrument, such as a dial indicator mounted on a conventional dial indicator support stand. The dial indicator shall have graduations of 0.01 mm, a measuring range in excess of 10 mm, a disk-like contact point about 10 mm in diameter, and a spindle travel distance controlled such that the measuring force applied by the contact point does not exceed 22 kPa.

i) Test instrument, capable of measuring a tensile force of at least 50 N at a rate of 500 mm/min

7.7.2 Specimen preparation

7.7.2.1 Prepare a minimum of five specimens

7.7.2.2 Advance preparation steps

7.7.2.2.1 For all specimens

— Apply a thin film of the high vacuum grease [7.7.1 c)] to the under surface of the mold cavity cover for the specimen sheet forming mold (Figure A.9, Key item 4)

— adapt a wrinkle free polyethylene sheet [7.7.1 b)] to the grease covered surface cavity cover

— condition the specimen sheet forming mold, without the cover, in the oven [7.7.1 d)] for at least 15 min

7.7.2.3 Specimen sheet forming steps

a) Verify the readiness of the material.

b) Remove the specimen sheet forming mold from the oven

c) Within 60s after removing the mold from the oven complete the following three steps: Slightly overfill the specimen sheet forming mold cavity with material. Press the polyethylene covered surface of the specimen sheet mold cover into contact with the mold cavity borders to expel the excess and give the specimen sheet its final form. Condition the specimen sheet forming assembly.

7.7.2.4. Specimen shaping and further preparatory steps. Accomplish the following steps within 90s after removal of the specimen forming assembly from the water bath conditioning.

a) Separate the formed specimen sheet from the mold and place it on the specimen sheet support pad [7.7.1 f)]

b) Use ASTM Tear Die C [7.7.1 g)] to cut the specimen to the desired shape (Figure A.8)

NOTE 1: If a specimen forming mold plate such as the one described in the Note appearing under 7.7.1.g) is used to form the specimen, the resulting specimen will be ready for the thickness measurement and testing immediately after separation from the mold plate

CAUTION — Handle specimens very carefully during subsequent steps to avoid stressing the notched area of the specimen before the test load is applied.

c) Use the instrument [7.7.1 h)] to measure thickness of the specimen at a point centered and just inside the apex of the 90° angle notch and record the measurement.

NOTE 2: To avoid pre-test stress of the specimen during the thickness measurement it may be necessary to increase dimensions of the measuring instrument base to provide complete support for the underside of the specimen.

d) Align and secure the specimen in the gripping mechanism of the test instrument [7.7.1 i)] for testing taking the following factors into account

— Experience seems to indicate that the optimum air pressure for use in pneumatic gripping of the material specimens is about 83 kPa (12 PSI).

— Depending upon the type of grip face surfacing, it may be necessary to cover gripping surfaces of the test instrument with adhesive backed abrasive paper (about ISO grit number P280) in order to achieve effective gripping without significant pre test stressing of the specimen.

NOTE: Fitting the specimens for gripping according to the option described in Annex B helps reduce possibilities for pre-test stressing of the specimen.

7.7.3 Test procedure

Immediately after securing the specimen in the gripping mechanism, apply a tensile test load at a speed of 500 mm/min until rupture of the specimen. Record the force required to achieve rupture.

7.7.4 **Calculation of results.**

Use the following equation to calculate the tear strength for each specimen to the nearest 0.01 N/mm:

$$T_s = F / d$$

where T_s is the tear strength mN/m (N/mm)

F is the maximum force, in newtons, applied to cause rupture of the specimen

d is the specimen thickness (mm)

7.7.5. Pass/fail determinations and expression of results

7.7 6. Tear strength mN.m (N/mm) (minimum) 75

8. Requirements for packaging, marking and instructions supplied by manufacturer

4.3 Tear Strength The tear strength of ASTM Tear Die C specimens, when tested in accordance with Clause 6.2.3, shall be greater than 200 cm-N for Type III Class 1 and Class 2 materials, and the liner materials for Type II Class 1 and Class 2 materials.

8.1. Packaging

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

An outer package may also be used to present the immediate containers as a single unit.

8.2. Marking

The outer packages and, if appropriate, the immediate containers or wrappings of the components, shall be clearly marked with the following information:

a) the trade name of the product;

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

c) the description of the contents including the following:

1) the type of material, according to Durometer Shore A hardness, as determined in accordance with 7.2;

2) the number of this part of ISO 10139, i.e. ISO 10139-2;

3) the chemical nature of the system, for example, heat-polymerizable, auto-polymerizable, acrylic polymer, silicone, thermoplastic, rigid material;

4) a statement that the product is a soft lining material for long-term use in removable dentures or maxillofacial prostheses;

5) the amount and type of solubles (if the solubility of the material is greater than 3 mg/m^3 ($\mu g/mm^3$), see 5.5);

d) the net content of the components, expressed in grams or millilitres;

e) the batch code (lot number) of the material, and the date of manufacture;

f) the expiry date, expressed according to ISO 8601, beyond which the material might not exhibit its required properties (year, month);

g) the recommended conditions of storage;

h) any hazard warnings, where appropriate, for toxic, hazardous, inflammable or irritating characteristics and flash point of liquid;

i) any pharmaceutically active ingredient present and referred to in the material claim for use.

In those cases where the size of the immediate container or package is too small to fit in all the details, reference shall be made on the outer package to a leaflet inside which the additional information shall be provided.

8.3. Manufacturer's instructions for use

Instructions for use shall accompany each package and shall include at least the following information:

a) the information listed in 8.2;

b) the fields of application;

c) the contraindications, side-effects and interactions with other substances, if appropriate;

d) a detailed description of the working procedure including the following information, where appropriate:

1) an indication of how to prepare the prosthesis;

2) the procedure for mixing or preparing the material, as appropriate, including information on the mixing ratio of the components and, if applicable, the mixing time and working time;

3) the procedure for application to the mould, flasking and packing;

4) all details of the application procedure, packing, curing procedure, time, temperature profile, pressure application, cooling, deflasking and any need for specialized equipment, where applicable;

5) the instructions for finishing and polishing;

6) a statement of any procedure or method to be employed to extrinsically color, if appropriate; and

7) statement of compatibility with various classes of skin adhesives.

e) any information on the care of the lined denture by the patient and recommendations for cleaning, including reference to any method or material which would be inappropriate for cleaning the lining;

f) any information on environmental conditions which may adversely affect the material, such as temperature, humidity or ambient light, and the disposal of waste, if precautions are necessary;

g) the (chemical) type of material(s).

Bibliography

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[6] American National Standard/American Dental Association Specification No. 99 for *Athletic Mouth Protectors and Materials*, 2001.

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