

Proposed American Dental Association
Standard No. 188

Dentistry –
Materials Used to Produce
Orthodontic Sequential
Aligners

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DRAFT

PROPOSED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION STANDARD NO. 188, DENTISTRY – MATERIALS USED TO PRODUCE ORTHODONTIC SEQUENTIAL ALIGNERS

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Standards approved as ADA Standards by an ADA Consensus Body are eligible to be forwarded to the American National Standards Institute (ANSI) for approval as an American National Standard (thus creating an ANSI/ADA Standard).

This document was formulated in ADA Standards Working Group 1.7 on Orthodontic Products which falls under ADA Consensus Body 1.

The scope of ADA Consensus Body 1 is:

Development of standards deliverables for nomenclature, requirements, test methods and acceptance criteria for restorative and orthodontic materials.

ADA Consensus Body 1 has representation from appropriate interests in the United States in the standardization of products and technologies within its scope.

ADA Consensus Body 1 thanks the members of Working Group 1.7 and the organizations with which they were affiliated at the time the standard was developed:

TO COME

This is the first edition of ANSI/ADA Standard No. 188.

**PROPOSED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION
STANDARD NO. 188, DENTISTRY – MATERIALS USED TO PRODUCE ORTHODONTIC
SEQUENTIAL ALIGNERS**

FOREWORD

(This Foreword does not form a part of Proposed ANSI/ADA Standard No. 188 for Dentistry – Materials Used to Produce Orthodontic Sequential Aligners).

This first edition of ANSI/ADA Standard No. 188 for Dentistry – Materials Used to Produce Orthodontic Sequential Aligners has been developed by SCDP Working Group 1.7 on Orthodontic Products to characterize the physical, thermal, and mechanical behavior of materials used in the production of orthodontic sequential aligners.

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STANDARD NO. 188 DENTISTRY – MATERIALS USED TO PRODUCE ORTHODONTIC
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PROPOSED AMERICAN NATIONAL STANDARD/AMERICAN DENTAL ASSOCIATION STANDARD NO. 188 DENTISTRY – MATERIALS USED TO PRODUCE ORTHODONTIC SEQUENTIAL ALIGNERS

1. Scope

This document specifies requirements and test methods for materials used in the production of orthodontic sequential aligners along with packaging and labelling requirements. This document includes materials that are thermoformed into sequential aligners, as well as materials that are used to additively manufacture sequential aligners.

This document does not specify qualitative and quantitative requirements for the evaluation of biocompatibility; which are covered in ANSI/ADA Standard No. 41/ISO 7405, ISO 10993-1, and ANSI/ADA Standard No. 27/ISO 4049.

2. Normative References

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ANSI/ADA 139, *Dental Base Polymers*
(Available at ADAStore.org)

ASTM D6272, *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials*

ASTM D3418, *Standard Test Method for Transition Temperature and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry*
(Available at www.astm.org)

ISO 1942, *Dentistry — Vocabulary*

ISO 6721, *Plastics - Determination of dynamic mechanical properties.*

ISO 13468-1:2019, *Plastics - Determination of the total luminous transmittance of transparent materials - Part 1: Single-beam instrument*

ISO 14782:1999, *Plastics - Determination of haze for transparent materials and ISO 14782:1999/Cor 1:2005 Plastics - Determination of haze for transparent materials - Technical Corrigendum 1.*
(Available at www.ansi.org)

3. Terms and Definitions

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

3.1. aligner

polymeric shell orthodontic appliance that is designed to incrementally reposition teeth from one arrangement to the next

Note 1 to entry: Typically used in series to accomplish larger movements.

4. Classification

For the purpose of this document, materials used in the production of sequential aligners for use in orthodontics are categorized into the following types:

Type 1: Materials that are thermoformed into a sequential aligner for use in orthodontics.

Type 2: Materials that are used to additively manufacture sequential aligners for use in orthodontics

5. Requirements

5.1. Physical and Thermal Behavior

5.1.1. Melting Temperature

When tested in accordance with 6.4.1, the material shall not exhibit a transition temperature or Peak melting temperature in the range from 0 °C to 70 °C, and the recorded transition temperature/Peak melting temperature (T_{pm}) of the material shall not differ by more than ± 5 °C from the value stated in the literature accompanying the material:

- a) T_{pm} = Peak melting temperature, °C;
- b) Materials that are intentionally designed to have transition temperatures in the range from 0 °C to 70 °C must report the transition temperature in the literature accompanying the material along with a statement that “This material is designed to transition in the temperature range from 0 °C to 70 °C”, and this temperature shall not differ by more than ± 5 °C from the reported value.

5.1.2. Light Transmittance

When tested in accordance with 6.4.2, the light transmittance of the material (% transmittance) shall comply with the range stated by the manufacturer.

5.1.3. Haze

When tested in accordance with 6.4.3, the haze of the material (% haze) shall comply with the range stated by the manufacturer.

5.1.4. Water Sorption

When tested in accordance with 6.4.4, the water sorption of the material, w_{sp} (mg/mm³), shall not exceed 20 mg/mm³.

5.1.5. Water Solubility

When tested in accordance with 6.4.4, the water solubility of the material, w_{st} (µg/mm³), shall not exceed 2 µg/mm³.

5.2. Mechanical Behavior

5.2.1. Flexural Strength

When tested in accordance with 6.5.1, the flexural strength of the material, σ_f (MPa), shall comply with the range stated by the manufacturer.

5.2.2. Flexural Modulus

When tested in accordance with 6.5.1, the flexural modulus of the material, E_B (MPa), shall comply with the range stated by the manufacturer.

5.2.3. Dynamic Mechanical Analysis Stress Relaxation

When tested in accordance with 6.5.2 the dynamic mechanical analysis stress relaxation, $\sigma_{24hr/0hr}$, shall comply with the ranges stated by the manufacturer.

6. Test Methods

6.1. 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.

6.2. Preparation of Test Specimens

6.2.1 Prepare, manipulate, and process materials for making the specimens using the equipment and procedures recommended in the material manufacturer's instructions for use (IFU) unless otherwise specified in this standard.

6.2.2 Specimens shall be fabricated according to their designated Type. For Type 1 materials, specimens shall be produced for each thickness of material that is available as a sequential aligner product. For Type 2 materials, specimens shall be produced at the thicknesses that represent both the minimum and maximum thickness range for their intended use as a sequential aligner product. Type 2 material specimens should be tested within 14 days of fabrication. Type 2 specimens should be stored in a lightproof environment prior to testing.

6.2.3 For Type 1 materials, unless otherwise specified in the procedure for an individual test method, test specimens shall be thermoformed over a mold using the material manufacturer's settings for time, temperature, and pressure. Figure 1 shows an example of an acceptable mold to form specimens (Note that it is acceptable to use a mold that produces one specimen at a time instead of 3 specimens, as shown in Figure 1, as long as the mold dimensions for an individual specimen are followed).

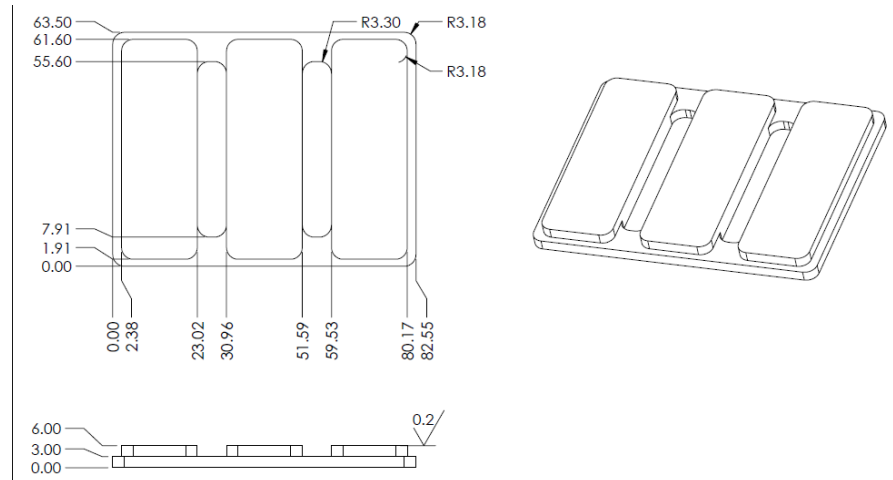


Figure 1 - Thermoforming mold

6.2.3.1 After forming over the mold (6.2.3), Type 1 material specimens shall be cut to 50.8 mm (2 in.) by 12.7 mm (0.5 in) with a tolerance of ± 0.127 mm (± 0.005 in.). Cutting may be performed via computer numerical control (CNC) milling, die punching, shearing or similar types of cutting methods. Specimens should not have cracks, internal stresses, or dimensional irregularities that can yield undesirable data.

6.2.4 For Type 2 materials, unless otherwise specified in the procedure for an individual test method, for the thicknesses that represent both the minimum and maximum thickness range (6.2.2), test specimens shall be produced to 50.8 mm (2 in.) by 12.7 mm (0.5 in) with a tolerance of ± 0.254 mm (± 0.010 in.).

6.3 Conditioning of Test Specimens

6.3.1 Unless otherwise specified in the procedure for an individual test method, for both Type 1 and Type 2 materials, specimens shall be conditioned in water prior to testing. Immerse the specimens in water (complying with grade 2 of ISO 3696:1987) at 37 ± 1 °C for $7 \text{ d} \pm 4 \text{ h}$. After this time, remove the specimens from the water with polymer coated tweezers and air-dry.

NOTE: It is acceptable to use hand dust-blower or source of oil-free compressed air to air-dry specimens.

Specimens shall be tested within 15 minutes of removal from water.

NOTE: Depending on the test method being performed, it may be necessary to stagger the immersion of individual specimens in the water so that all specimens can be tested within 15 minutes of removal from water.

6.4 Physical and Thermal Behavior

6.4.1 Thermal Behavior

The test shall be performed in compliance with ASTM D3418-15 *Standard Test Method for Transition Temperature and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimeter* except as noted in the following Procedure section.

6.4.1.1 Apparatus

6.4.1.1.1 Differential Scanning Calorimeter (DSC) and the essential instrumentation required to provide the minimum differential scanning calorimetric capability for this test method.

6.4.1.1.2 Balance, capable of weighing to ± 0.0001 grams for transition temperatures and to ± 0.00001 grams for determining enthalpies.

6.4.1.1.3 Tweezers

6.4.1.1.4 Paper punch, hypodermic punch, razor blade, or other appropriate means of cutting specimens to fit in the standard aluminum pans (6.4.1.1.5).

6.4.1.1.5 Standard Aluminum pan with non-hermetic lid, or other compatible containers.

6.4.1.2 Preparation of Test Specimens

6.4.1.2.1 For Type 1 materials, for each product thickness, prepare five specimens, as described in 6.2. Next, for each of these specimens, cut five individual specimens with a punch (or other appropriate cutting method) into the appropriate size to make suitable contact with the bottom of the standard pans (6.4.1.1.5) chosen for use with the DSC. Each of the individual specimens shall also meet the approximate weight requirements (6.4.1.2.3).

NOTE: To make suitable contact with the bottom of the standard pan (i.e., specimen covering as much of the surface area of the bottom of the pan as possible) and meet the approximate weight requirements, it may be necessary to cut the individual specimens into multiple pieces and stack them in the pan. ASTM D3418 states that "intimate thermal contact between the pan and specimen is essential for reproducible results."

6.4.1.2.2 For Type 2 materials, for each of the thicknesses that represent both the minimum and maximum thickness range (6.2.2), prepare five individual specimens such that each specimen covers as much of the surface area of the bottom of the respective standard pan (6.4.1.1.5) as possible while adhering to the approximate weight requirements (6.4.1.2.3).

6.4.1.2.3 Specimens shall weigh approximately 10 mg, and shall be weighed using the balance (6.4.1.1.2) to an accuracy of ± 10 μg for both Type 1 and Type 2 materials.

6.4.1.3 Conditioning of Test Specimens

For both Type 1 and Type 2 materials, the specimens shall be conditioned prior to testing in accordance with 6.3.

6.4.1.4 Procedure

6.4.1.4.1 The purge gas shall be used during testing. The flow rate of the gas should be the same as

used in the calibration of the equipment.

6.4.1.4.2 Weigh the specimen to an accuracy of $\pm 10 \mu\text{g}$, specimens shall weigh approximately 10 mg for both Type 1 and Type 2 materials.

6.4.1.4.3 Place one specimen per pan using tweezers to avoid contamination. Special attention should be used to make sure the specimen is in full contact with the bottom of the pan.

6.4.1.4.4 Crimp a metal cover against the pan with the specimen sandwiched in between to ensure good heat transfer. Intimate thermal contact between the pan and the specimen is essential for reproducible results.

6.4.1.4.5 Perform and record a preliminary thermal cycle by heating the specimen at a rate of $20 \text{ }^\circ\text{C}/\text{min}$ under inert purge gas atmosphere from at least $50 \text{ }^\circ\text{C}$ below to $30 \text{ }^\circ\text{C}$ above the expected melting/transition temperature.

NOTE: Since the moisture introduced to the specimen during the conditioning step may produce an endotherm in the DSC scan that may be mistaken for a melting transition, it is necessary to perform a preliminary thermal cycle. Performing a second thermal cycle will help confirm whether or not such an endotherm was caused by moisture, or if it was indeed indicative of a meaningful transition in the material.

6.4.1.4.6 Hold the temperature for 5 min.

NOTE: ASTM D3418 permits the use of a shorter hold time in cases where a high-temperature hold causes polymer degradation, but the deviation shall be reported.

6.4.1.4.7 Cool to at least $50 \text{ }^\circ\text{C}$ below the transition temperature of interest using the same rate that was used for heating and record the cooling curve.

NOTE: If the transition temperature of interest is a glass transition, ASTM D3418 allows quench cooling to at least $50 \text{ }^\circ\text{C}$ below the transition temperature of interest.

6.4.1.4.8 Hold the temperature for 5 min.

6.4.1.4.9 Repeat heating at a rate of $20 \text{ }^\circ\text{C}/\text{min}$ and record the heating curve until all desired transitions have been completed.

Measure the temperature for the desired point on the curve(s) as T_{pm} or other known transition temperature. Confirm that transitions still appear on the second thermal cycle; transitions that disappear during the second thermal cycle are most likely due to moisture being driven from the specimen and may be ignored. If more than one transition temperature is observed, report all observed transition temperatures.

Where:

T_{pm} = melting peak temperature, $^\circ\text{C}$

NOTE: ASTM D3418 notes that the actual temperature displayed on the temperature axis depends upon the instrument type, and it is, therefore, suggested to follow any recommended procedures or

guidelines of the instrument manufacturer to obtain specimen temperature at the point of interest.

6.4.1.5 Expression of Results

Calculate and report the average melting/transition temperature(s) of the material as the mean of the values of the five specimens to the nearest 1 °C, and whether the material passes the requirements in 5.1.1.

Report any side reaction (for example, crosslinking, thermal degradation, or oxidation), and the reaction identified if possible.

6.4.2 Light Transmittance

The test shall be performed in compliance with ISO 13468-1:2019 *Plastics - Determination of the total luminous transmittance of transparent materials - Part 1: Single-beam instrument*

6.4.2.1 Preparation of Test Specimens

6.4.2.1.1 For Type 1 materials, test specimens shall be thermoformed over a mold using the material manufacturer's settings for time, temperature, and pressure. The mold shall be such that it produces specimens that are large enough to cover the port in the chosen measurement instrumentation, as described in the Apparatus section (6.4.2.3), and the mold shall conform to the surface finish requirements specified in Figure 1 (i.e., a maximum of 0.2 μm). After forming over the mold, specimens shall be cut to properly fit in the chosen measurement instrumentation, as described in the Apparatus section. Cutting may be performed via computer numerical control (CNC) milling, die punching, shearing or similar types of cutting methods. Specimens should not have cracks, internal stresses, or dimensional irregularities that can yield undesirable data. A minimum of five specimens shall be produced for each thickness of material that is available as a sequential aligner product.

6.4.2.1.2 For Type 2 materials, specimens shall be produced at the thicknesses that represent both the minimum and maximum thickness range for their intended use as a sequential aligner product and with dimensions that properly fit the chosen measurement instrumentation, as described in the Apparatus section (6.4.2.3). A minimum of five specimens shall be produced for both the minimum and maximum thickness range. Type 2 material specimens should be tested within 14 days of fabrication. Type 2 specimens should be stored in a lightproof environment prior to testing.

6.4.2.2 Conditioning of Test Specimens

For both Type 1 and Type 2 materials, the specimens shall be conditioned prior to testing in accordance with 6.3.

6.4.2.3 Apparatus

6.4.2.3.1 See apparatus section in ISO 13468-1:2019 for a description of the necessary equipment to perform the test method.

6.4.2.4 Procedure

6.4.2.4.1 Perform the test according to the Procedure set forth in ISO 13468-1:2019.

6.4.2.5 Expression of Results

Calculate and report the light transmittance of the material (% transmittance) as the mean of the values of the five specimens tested to the nearest percent, and whether the material passes the requirement in 5.1.2.

6.4.3 Haze

The test shall be performed in accordance with ISO 14782:1999 *Plastics - Determination of haze for transparent materials*, and ISO 14782:1999/Cor 1:2005 *Plastics — Determination of haze for transparent materials - Technical Corrigendum 1*

6.4.3.1 Preparation of Test Specimens

6.4.3.1.1 For Type 1 materials, test specimens shall be thermoformed over a mold using the material manufacturer's settings for time, temperature, and pressure. The mold shall be such that it produces specimens that are large enough to cover the port in the chosen measurement instrumentation, as described in the Apparatus section (6.4.3.3), and the mold shall conform to the surface finish requirements specified in Figure 1 (i.e., a maximum of 0.2 μm). After forming over the mold, specimens shall be cut to properly fit in the chosen measurement instrumentation, as described in the Apparatus section. Cutting may be performed via computer numerical control (CNC) milling, die punching, shearing or similar types of cutting methods. Specimens should not have cracks, internal stresses, or dimensional irregularities that can yield undesirable data. A minimum of five specimens shall be produced for each thickness of material that is available as a sequential aligner product.

6.4.3.1.2 For Type 2 materials, specimens shall be produced at the thicknesses that represent both the minimum and maximum thickness range for their intended use as a sequential aligner product and with dimensions that properly fit the chosen measurement instrumentation, as described in the Apparatus section (6.4.3.3). Type 2 material specimens should be tested within 14 days of fabrication. Type 2 specimens should be stored in a lightproof environment prior to testing. A minimum of five specimens shall be produced for both the minimum and maximum thickness range.

6.4.3.2 Conditioning of Test Specimens

For both Type 1 and Type 2 materials, the specimens shall be conditioned prior to testing in accordance with 6.3.

6.4.3.3 Apparatus

6.4.3.3.1 The apparatus shall consist of a stabilized light source, an associated optical system, an integrating sphere with ports, and a photometer comprised of a photodetector, signal processor, and a display unit or recorder.

6.4.3.3.2 The light source and the photometer shall be used in conjunction with a filter to provide an output corresponding to a photopic standard luminous efficiency which is identical to the color-

matching function specified in ISO/CIE 10527 under CIE standard illuminant d65, as specified in ISO/CIE 10526. The output of the photodetector shall be proportional to the incident flux, to within 1% of the incident flux, over the range used.

6.4.3.3.3 The light source and its associated optical system shall produce a parallel light beam, no ray of which makes an angle of more than 0.05 rad (3°) with the beam axis. This beam shall not exhibit vignetting at either port of the integrating sphere.

6.4.3.3.4 The design of the instrument shall be such that the reading is zero in the absence of the light beam.

6.4.3.3.5 The integrating sphere used to collect the transmitted light may be of any diameter (but preferably no less than 150 mm in order to be able to accommodate large specimens), as long as the total port area does not exceed 3.0% of the internal reflecting area of the sphere.

6.4.3.3.6 The integrating sphere shall have an entrance port, an exit port, a compensation port, and a photodetector port. The entrance and exit ports shall be centered on the same great circle of the sphere, and there shall be an angle of 3.14 ± 0.03 rad $180 \text{ deg} \pm 2$ between the centers of the ports.

6.4.3.4 Procedure

6.4.3.4.1 Allow the apparatus sufficient time to reach thermal equilibrium before measurements are made.

6.4.3.4.2 Mount a specimen in the specimen holder.

6.4.3.4.3 Make the four measurements given in Table 1.

6.4.3.4.4 Measure the thickness of the specimen in three places to an accuracy of 0.02 mm.

6.4.3.4.5 Carry out the procedure on each of the specimens in turn.

Table 1 - Haze Measurements

	Entrance Port	Exit Port	Compensation Port
τ_1		White reference	Light trap
τ_2	Specimen	White reference	Light trap
τ_3		Light trap	White reference
τ_4	Specimen	Light trap	White reference

6.4.3.5 Expression of Results

Calculate the haze, in percent, using the following equation:

$$\text{Haze} = \left(\frac{\tau_4}{\tau_2} - \frac{\tau_3}{\tau_1} \right) \times 100$$

Where

- τ_1 is the intensity of the incident light;
- τ_2 is the intensity of the light transmitted by the specimen;
- τ_3 is the intensity of the light scattered by the instrument;
- τ_4 is the intensity of the light scattered by the instrument and the specimen.

NOTE: If using equipment that just reports a Haze value without the individual parameters (τ_1 , τ_2 , τ_3 , and τ_4), then it is acceptable to record the single Haze value for the individual specimen to be used in the calculation of the mean of the values of the five specimens.

Calculate and report the Haze of the material as the mean of the values of the five specimens tested to the nearest percent, and whether the material passes the requirement in 5.1.3.

6.4.4 Water Sorption and Solubility

Water sorption and solubility (Adapted from ANSI/ADA Standard No. 139 *Dental Base Polymers, ISO 20795-2 Dentistry – Base Polymers – Part 2: Orthodontic base polymers*)

6.4.4.1 Materials

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

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

6.4.4.2 Apparatus

6.4.4.2.1 Rack, to keep the specimens parallel and separated.

6.4.4.2.2 Desiccators, two.

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

6.4.4.2.4 Tweezers, polymer coated.

6.4.4.2.5 Towel, clean and dry.

6.4.4.2.6 Micrometer screw gauge, accurate to 0.01 mm.

6.4.4.2.7 Dial gauge caliper or slide caliper, accurate to 0.01 mm.

6.4.4.2.8 Analytical Balance, accurate to 0.1 mg.

6.4.4.3 Preparation of Test Specimens

For both Type 1 and Type 2 materials, prepare a minimum of five specimens, as described in 6.2.

6.4.4.4 Procedure

6.4.4.4.1 Dry conditioned mass (m_1) and volume (V)

6.4.4.4.1.1 Place the specimens in/on the rack (6.4.4.2.1) inside one of the desiccators (6.4.4.2.2) containing freshly dried silica gel (6.4.4.1.1). Store the desiccator in the oven (6.4.4.2.3) at 37 ± 1 °C for 24 ± 1 h and then remove the desiccator from the oven.

6.4.4.4.1.2 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.

6.4.4.4.1.3 Use an analytical balance (6.4.4.2.8) 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 weightings.

6.4.4.4.1.4 At this point calculate the volume, V , of each rectangular specimen, using the mean of three width measurements, the mean of three length measurements, and the mean of three thickness measurements made across the specimen corresponding to approximately a first edge, a middle, and a second edge.

6.4.4.4.2 Wet conditioned mass (m_2)

Immerse the conditioned specimens in water (6.4.4.1.2) at 37 ± 1 °C for $7 \text{ d} \pm 2 \text{ h}$. After this time, remove the specimens from the water with polymer coated tweezers (6.4.4.2.4), wipe with a clean dry towel (6.4.4.2.5) or blow with compressed air until free from visible moisture, wave in the air for $15 \pm 1 \text{ s}$ and weigh $60 \pm 0 \text{ s}$ after removal from the water to an accuracy of 0.2 mg. Record the mass as m_2 .

6.4.4.4.3 Reconditioned dry mass (m_3)

After this weighing, recondition the specimens to constant mass in the desiccator as described in 6.4.4.4.1.1 to 6.4.4.4.1.3. 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 6.4.4.4.1), using the same number of specimens and the freshly dried silica gel in the desiccators.

6.4.4.5 Calculation and expression of results

6.4.4.5.1 Water sorption

Calculate the value for the water sorption, w_{sp} , for each specimen, expressed in milligrams per cubic millimeter from the following equation:

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

where

m_2 is the wet conditioned mass of the specimen (see 5.2.5.4.2), in milligrams

m_3 is the reconditioned mass of the specimen (see 5.2.5.4.3), in milligrams

V is the volume of the dry conditioned specimen (see 5.2.5.4.1), in cubic millimeters.

Record the calculated value of each specimen for water sorption to the nearest milligram per cubic millimeter.

6.4.4.5.2 Water solubility

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

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

where

m_1 is the dry conditioned mass of the specimen (see 5.2.5.4.1), in micrograms

m_3 is the reconditioned mass of the specimen (see 5.2.5.4.3), in micrograms

V is the volume of the dry conditioned specimen (see 5.2.5.4.1), in cubic millimeters

Round off the values calculated for water solubility to the nearest 0.1 microgram per cubic millimeter.

6.4.4.5.3 Pass/fail determination of water sorption

If at least four of the water sorption results comply with the requirement stated in 5.1.4, the material passes.

If at least three of the water sorption results do not comply with the requirement stated in 5.1.4, the material is deemed to have failed.

If only three of the water sorption results comply with the requirement stated in 5.1.4, 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 5.1.4, the material is deemed to have passed.

6.4.4.5.4 Pass/fail determination of water solubility

If at least four of the water solubility results comply with the requirement stated in 5.1.5, the material passes.

If at least three of the water solubility results do not comply with the requirement stated in 5.1.5, the material is deemed to have failed.

If only three of the water solubility results comply with the requirement stated in 5.1.5, 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 5.1.5, the material is deemed to have passed.

6.4.4.5.5 Expression of results

Report the number of specimens evaluated, all results for water sorption and water solubility with the numbers of specimens complying with the requirements of 5.1.4 and 5.1.5, and whether the material passes.

6.5 Mechanical Behavior

6.5.1 Flexural Strength and Flexural Modulus

The flexural strength and flexural modulus procedures are adapted from ASTM D6272 *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point bending*, Procedure A for materials with a thickness of less than 1.6 mm.

NOTE: Meira et al.¹¹ noted that test specimens of aligner materials which exceed the maximum strain of 5.0% does not apply to the requirements of the three-point loading system that is described in ASTM790.

6.5.1.1 Apparatus

6.5.1.1.1 Testing machine – a properly calibrated mechanical test system with a crosshead that can operate at a constant rate of motion over the range indicated in the procedure. The testing system shall be equipped with a load sensor, and its error shall not exceed $\pm 1\%$ of the maximum load expected to be measured. Furthermore, the load sensor shall be essentially free from inertial lag at the crosshead rate used. Also, the testing machine shall be equipped with a deflection measuring device. The stiffness of the testing machine shall be such that the total elastic deformation of the system does not exceed 1% of the total deflection of the test specimen during testing.

6.5.1.1.2 Four-point loading apparatus – The loading noses and supports of the apparatus shall have cylindrical surfaces. The radii of the loading noses and supports shall be 3.2 ± 0.1 mm with a support span (L) between supports of 25.4 ± 0.3 mm.

6.5.1.1.3 Micrometer - Suitable micrometer for measuring the width and thickness of the test specimen to an incremental discrimination of at least 0.001 mm should be used.

6.5.1.2 Preparation of Test Specimens

For both Type 1 and Type 2 materials, prepare a minimum of ten specimens, as described in 6.2.

6.5.1.3 Conditioning of Test Specimens

For both Type 1 and Type 2 materials, the specimens shall be conditioned prior to testing in accordance with 6.3.

6.5.1.4 Procedure

Test at least 10 specimens for each material.

Use an untested specimen for each measurement. Measure the width and depth (thickness) of each specimen to the nearest 0.003 mm at the center of the support span.

For flexural fixtures that have continuously adjustable spans, measure the span accurately to the nearest 0.1 mm. Use the actual measured span for all calculations. For flexural fixtures that have fixed span positions, verify the span distance, the same as for adjustable spans, and use the actual measured span for all calculations.

Calculate the rate of crosshead motion according to the Procedure A set forth in ASTM D6272, which employs a strain rate of 0.01 mm/mm/min, and set the testing machine (6.5.1.1.1) for the calculated rate of crosshead motion. During loading, the actual crosshead rate shall not differ from that calculated using ASTM D6272 by more than $\pm 10\%$. According to ASTM D6272 (Note 7), for materials that do not yield or break within the 5 % strain limit when tested by Procedure A, the strain rate of 0.10 mm/mm/min specified by Procedure B is allowed, which may induce the specimen to yield or break, or both within the required 5 % strain limit.

NOTE: According to ASTM D6272, beyond 5 % strain, this test method is not applicable. Materials that do not rupture by the maximum strain allowed under this test method may be more suited for tensile testing.

Align the loading noses and supports so that the axes of the cylindrical surfaces are parallel and the loading span is one third of the support span. The parallelism of the apparatus may be checked by means of a plate with parallel grooves into which the loading noses and supports will fit when properly aligned. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading noses and supports. The loading noses assembly shall be the type which does not rotate.

Apply the load to the specimen at the specified crosshead rate, and collect simultaneous load-deflection data. Measure deflection using a gage under the specimen in contact with it at the center of the support span. Do not use the movement of loading noses relative to the supports. Load-deflection curves may be plotted to determine the following: flexural strength; the secant modulus or the tangent modulus of elasticity; and the total work (optional), as measured by the area under the load-deflection curve. Perform the necessary toe compensation to correct for seating and indentation of the specimen and deflections in the machine.

6.5.1.5 Calculation and Expression of Results

6.5.1.5.1 Flexural Strength

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

$$\sigma_f = PL/bd^2$$

σ_f = Stress in the outer fibers at maximum load, MPa;
 P = Maximum load on the load deflection curve, N;
 L = Support Span, mm;
 b = Average Width of specimen tested, mm;
 d = Average Depth (thickness) of specimen tested, mm.

Some materials that do not break at strains of up to 5% may give a load deflection curve that shows a point at which the load does not increase with an increase in strain, which is the yield point, Y. The flexural strength may then be calculated for those materials by letting P equal the load at the yield point, Y.

According to ASTM D6272, for beam specimens tested at large support spans, such that the support span-to-specimen depth ratio is greater than 16 to 1 and deflections exceed 10% of the support span, calculate the ultimate flexural strength, σ_f , in megapascals using the following equation:

$$\sigma_f = (PL/bd^2)[1 + (4.70D^2/L^2) - (7.04dD/L^2)]$$

where: σ_f , P, L, b, and d are the same as above, and
 D = deflection of the centerline of the specimen at the middle of the support span at the maximum load, P, in mm.

NOTE: ASTM D6272 notes that when large support span-to-depth ratios are used, significant end forces are developed at the support noses, which will affect the moment in a simple supported beam. The above equation includes additional terms that are an approximate correction factor for the influence of these end forces in large support span-to-depth ratio beams where relatively large deflections exist.

6.5.1.5.2 Flexural Modulus

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

$$E_B = 0.21L^3m/bd^3$$

Where:

E_B = Modulus of elasticity in bending, MPa;
 L = Support span, mm;
 b = Average Width of specimen tested, mm;
 d = Average Depth (thickness) of specimen tested, mm;
 m = slope of tangent to the initial straight-line portion of the load deflection curve, N/mm of deflection.

The secant modulus may also be determined using the equation above by letting m equal the slope of the secant to the load-deflection curve as described in ASTM D6272. If the secant modulus is calculated, the chosen stress point used for the determination of the secant shall be reported.

6.5.1.5.3 Expression of results for flexural strength

Calculate and report the flexural strength of the material as the mean of the values of the ten specimens tested to the nearest MPa, and whether the material passes the requirement in 5.2.1.

6.5.1.5.4 Expression of results for flexural modulus

Calculate and report the flexural modulus of the material as the mean of the values of the ten specimens tested to the nearest MPa, and whether the material passes the requirement in 5.2.2. Also, report the method of modulus of elasticity determination (tangent or secant) and the stress level used if secant modulus.

6.5.2 Stress Relaxation Test Method

The test shall be performed per ISO 6721 *Plastics - Determination of dynamic mechanical properties*. The stress relaxation and strain recovery (SR-SR) behaviors of a specimen of uniform geometry is analyzed under isothermal and constant humidity conditions using a single cantilever configuration. The single cantilever configuration imparts a bending stress on the test specimen.

6.5.2.1 Apparatus

6.5.2.1.1 A Dynamic Mechanical Analyzer (such as the TA Instruments Q800 or DMA 850), with a relative humidity (RH) generator, shall be used.

NOTE: This information about the Q800 or DMA 850 instrumentation is given for the convenience of users of this document and does not constitute an endorsement by ANSI or ADA of this product.

6.5.2.1.2 The specimen will be held in a single cantilever configuration as shown in Figure 2.

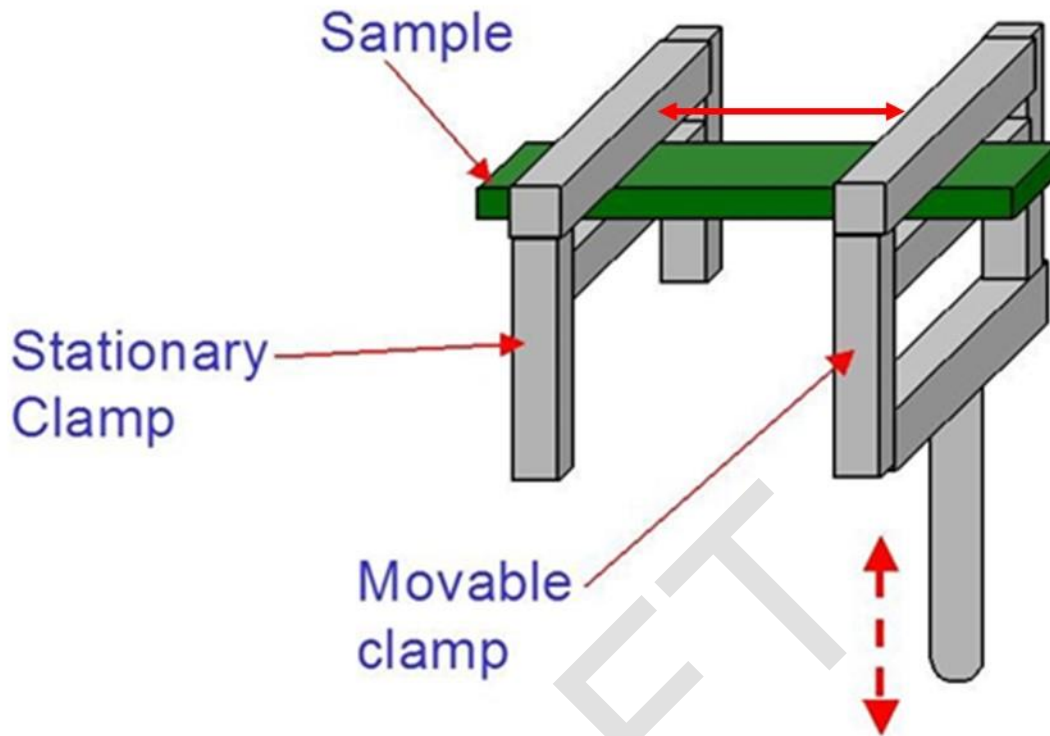


Figure 2 – Illustration demonstrating single cantilever configuration

6.5.2.1.3 Measure and record the effective length of the specimen as the distance between the inner facing surfaces of the clamps, as shown in Figure 2.

NOTE: On the Q800 and DMA 850, the effective length distance is 17.5 mm; however, this distance may vary for other DMA instrumentation.

6.5.2.2 Preparation of Test Specimens

For both Type 1 and Type 2 materials, prepare a minimum of five specimens, as described in 6.2.

6.5.2.3 Conditioning of test specimens

For both Type 1 and Type 2 materials, the specimens shall be conditioned prior to testing in accordance with 6.3.

6.5.2.4 Procedure

6.5.2.4.1 Test shall be run in Stress Relaxation Mode with 1.0 % strain.

6.5.2.4.2 Test shall be run at 95% relative humidity.

6.5.2.4.3 Measure the width of each test specimen to within ± 0.01 mm and the thickness to within ± 0.001 mm.

6.5.2.4.4 With the moveable clamp in the float position, slide the test specimen first through the stationary clamp then through the movable clamp. Ensure that the specimen is perpendicular lengthwise to each of the clamps.

6.5.2.4.5 Tighten the screw of the stationary clamp first at a torque of 5 ft lbs. Hand tighten the screw of the moveable clamp.

6.5.2.4.6 Lock the movable clamp and retighten the screw of the moveable clamp to 5 ft lbs.

6.5.2.4.7 Untighten the stationary clamp screw and retighten to 5 ft lbs. This will alleviate stress on the specimen due to clamping.

6.5.2.4.8 Close the instrument specimen chamber.

6.5.2.4.9 Allow specimen to equilibrate until it remains at 37 °C at 95% humidity for 15 minutes.

6.5.2.4.10 Displace specimen to 1.0 % strain.

6.5.2.4.11 Hold at 1.0 % strain for 24 hours while measuring stress.

6.5.2.4.12 Release strain and allow specimen chamber to return to ambient conditions.

6.5.2.5 Calculation and Expression of Results

Stress relaxation will be expressed as a ratio of the stress measured at 24 hours divided by the stress measured at 0 hours. Calculate and report the stress relaxation of the material as the mean of the values of the five specimens tested to the nearest whole number, and whether the material passes the requirement in 5.2.3

7. Packaging, Marking, Instructions, and Information to be Supplied by the Manufacturer

7.1. General requirements

The manufacturer shall make the following readily available in the catalogue, packaging insert, labeling or other readily accessible means:

- a) thermal behavior which shall be in accordance with 5.1.1;
- b) light transmission which shall be in accordance with 5.1.2;
- c) haze which shall be in accordance with 5.1.3;
- d) water sorption which shall be in accordance with 5.1.4;
- e) water solubility which shall be in accordance with 5.1.5;
- f) flexural strength which shall be in accordance with 5.2.1;
- g) flexural modulus which shall be in accordance with 5.2.2;

h) dynamic mechanical analysis which shall be in accordance with 5.2.3.

NOTE: Additional information may be included at the discretion of the manufacturer or as required by legislation.

7.2. Outer Packaging

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

- a) the name and address of the manufacturer or distributor/agent, or both in the country of sale;
- b) the name or trade/brand name of the material;
- c) the intended use;
- d) the "Manufacture Date" and "Expiry Date" year and month, expressed in accordance with ISO 8601;
- e) the quantity;
- f) recommended conditions of storage;

7.3. Packaging

Adequate containment and protection from contamination during transit and storage shall be provided in accordance with acceptable commercial practice.

7.4. All immediate containers

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

- g) the name and address of the manufacturer or distributor/agent, or both in the country of sale;
- h) the name or trade/brand name of the material;
- i) the intended use;
- j) the "Manufacture Date" and "Expiry Date" year and month, expressed in accordance with ISO 8601;
- k) the quantity;
- l) recommended conditions of storage;

7.5. Manufacturer's Instructions

The instructions for safe and effective use of the material shall be included in each package or otherwise readily available. As a minimum they shall include (as applicable) the following information:

- a) warnings or precautions regarding processing of the material;
- b) details of the composition of the material;
- c) time, temperature, and procedures required for processing the material for use;
- d) time over which processing of material may be conducted;

- e) equipment and material needed to process material;
- f) recommended storage conditions for unprocessed material;
- g) post-processing treatment of the processed material.

7.6. Declaration of Components

The manufacturer shall provide, either in the instructions for use or by means of a Safety Data Sheet, the composition and information on components present in the materials ≥ 1 % by mass (irrespective of hazard potential) and any ingredient that is classified as a carcinogen, mutagen or reproductive toxicant (CMR) present in the material ≥ 0.1 % by mass.

The list shall include a chemical name or its common abbreviation if available. The Chemical Abstracts Service registry number (CAS number) may be used.

Either the mass percent range for each listed component or a listing in the order of mass from the highest to the lowest concentration shall be provided.

NOTE CMR is defined in authoritative lists, for example, by the International Agency for Research on Cancer (IARC), National Toxicology Program (NTP), American Conference of Governmental Industrial Hygienists (ACGIH), EC Annex VI CMR to Regulation (EC) 1272/2008.

The manufacturer may include additional information at their discretion.

Bibliography

- [1] ANSI/ADA Standard No. 41, *Evaluation of Biocompatibility of Medical Devices*
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- [3] ASTM D790 *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating*
- [4] ASTM D3418 *Standard Test Method for Transition Temperature and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry*
- [5] ISO 1942, *Dentistry - Vocabulary*
- [6] ISO 6721, *Plastics - Determination of dynamic mechanical properties*
- [7] ISO 7405, *Dentistry - Evaluation of biocompatibility of medical devices used in dentistry.*
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- [11] R. Miera, H. Lukic, and S.J. Megremis, "Characterization of Materials Used to Produce Orthodontic Sequential Aligners," *Journal of Dental Research*, 2022; Vol.101, Special Issue A. <https://iadr.abstractarchives.com/abstract/51am-3666425/characterization-of-materials-used-to-produce-orthodontic-sequential-aligners>

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