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Standard Guide for Evaluation of Thermoplastic Polyurethane Solids and Solutions for Biomedical Applications[1]

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1. Scope

1.1 This guide covers the evaluation of thermoplastic polyurethanes in both solid and solution form for biomedical applications. The polymers have been reacted to completion and require no further chemical processing.

1.2 The tests and methods listed in this guide may be referenced in specification containing minimum required values and tolerances for specific end use products.

1.3 Test values shall be stated in SI units with inch-pound units in parentheses.

1.4 Standard tests for biocompatibility are included to aid in the assessment of safe utilization in biomedical applications. Compliance with these criteria shall not be constructed as an endorsement of implantability. Since many compositions, formulations, and forms of thermoplastic polyurethanes in solid and solution forms are within this material class, the formulator or fabricator must evaluate the biocompatibility of the specific composition or form in the intended use and after completion of all manufacturing processes including sterilization.

1.5 Purchase specifications may be prepared by agreement between the buyer and seller by selection of appropriate tests and methods from those listed applicable to the specific biomedical end use.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[2]

D 150 Test Methods for ac Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[2]

D 257 Test Methods for dc Resistance or Conductance of Insulating Materials[2]

D 395 Test Methods for Rubber Property—Compression Set[3]

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers-Tension[3]

D 570 Test Method for Water Absorption of Plastics[4]

D 575 Test Methods for Rubber Properties in Compression[3]

D 671 Test Method for Flexural Fatigue of Plastics by Constant-Amplitude-of-Force[5]

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[4]

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[4]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[4]

D 1242 Test Methods for Resistance of Plastic Materials to Abrasion[4]

D 1434 Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting[6]

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)[7]

D 1638 Methods of Testing Urethane Foam Isocyanate Raw Materials[8]

D 2124 Test Method for Analysis of Components in Poly-(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique[4]

D 2240 Test Method for Rubber Property—Durometer Hardness[3]

D 2857 Test Method for Dilute Solution Viscosity of Polymers[9]

D 2990 Test Methods for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics[9]

D 3137 Test Method for Rubber Property—Hydrolytic Stability[3]

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[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

[4] *Annual Book of ASTM Standards*, Vol 08.01.

[5] Discontinued; See 2001 *Annual Book of ASTM Standards*, Vol 08.01.

[6] *Annual Book of ASTM Standards*, Vol 15.09.

[7] *Annual Book of ASTM Standards*, Vol 06.01.

[8] Discontinued; See 1998 *Annual Book of ASTM Standards*, Vol 08.01.

[9] *Annual Book of ASTM Standards*, Vol 08.02.

D 3418 Test Method for Transition Temperatures of Polymers by Thermal Analysis[3]
E 96 Test Methods for Water Vapor Transmission of Materials[10]
F 619 Practice for Extraction of Medical Plastics[11]
F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[11]

3. Terminology

3.1 *Definitions:*

3.1.1 *chain extender*—(*1*) an active hydrogen containing compound such as a diol or diamine used to increase the molecular weight of an isocyanate-terminated prepolymer by chemical reaction; (*2*) a diisocyanate used to extend a polyol-terminated polyurethane by chemical reaction.

3.1.2 *chain terminating agent*—an active hydrogen containing a compound such as a monofunctional alcohol, amine, or acid that reacts with the isocyanate group of a prepolymer to prevent further chain growth.

3.1.3 *linear polyurethane*—a polymer whose backbone consists of urethane groups joined by hydrocarbon chains with little or no cross linking.

3.1.4 *segmented polyurethane*—A family of polymers in which ester or ether groups, connected by hydrocarbon chains, occur as blocks that are coupled by urethane and urea groups.

3.1.5 *thermoplastic polyurethane*—linear or segmented polyurethanes that can be melted for processing without significant crosslinking or degradation. They are most frequently synthesized by reacting diols with diisocyanates.

4. Significance and Use

4.1 This guide is intended to aid device fabricators in the selection of proper commercially available polyurethane solids and solutions for their application.

4.2 The polyurethanes covered by this guide may be thermoformed or solution cast into biomedical devices for use as surgical aids or for implantation as determined to be appropriate, based on supporting biocompatibility and physical test data.

5. Descriptive Chemical Information

5.1 *Diols*—Diols that can be used for biomedical applications are as follows:

5.1.1 Poly(oxypropylene).
5.1.2 Poly(oxytetramethylene).
5.1.3 Poly(caprolactone).
5.1.4 Poly(ethylene adipate).
5.1.5 1,4-dihydroxybutane.
5.1.6 Mixture of the above diols.

5.2 *Difunctional Diisocyanates*—Difunctional diisocyanates that can be used are:

5.2.1 Diphenylmethane 4,4-diisocyanate (MDI).
5.2.2 2,4-tolylene diisocyanate (TDI).
5.2.3 1,5-naphthalene diisocyanate.
5.2.4 1,6-hexamethylene diisocyanate (HMDI).

5.3 *Chain Extenders*—Chain extenders that can be used are:

5.3.1 Water.
5.3.2 Glycols.
5.3.3 Aliphatic and aromatic diamines.

5.4 *Chain-Terminating Agents*—Chain-terminating agents suitable for use are:

5.4.1 Monofunctional alcohols, such as methanol or ethanol.
5.4.2 Monofunctional amines, such as dibutylamine or diethylamine.

5.5 *Catalysts*—Stannous octoate is suitable for prepolymer preparation.

5.6 *Optional Additives*:

5.6.1 Pigments and dyes, such as titanium dioxide and copper phthalocyanine blue.
5.6.2 Radiopaque materials, such as barium sulfate.
5.6.3 Antiblocking agents and lubricants, such as natural and synthetic waxes.
5.6.4 Optical brighteners, antioxidants, and light and heat stabilizers.
5.6.5 The basic polymer bought may contain, as agreed upon between the purchaser and supplier, optional adjunct substances required in the production of the polymer or intended end use product, provided these substances are in minor amounts and are biocompatible in the recommended concentrations.

5.7 *Descriptive Information on Polyurethane Solutions*:

5.7.1 Solvent or solvents.
5.7.2 Percent solids.[12]
5.7.3 Solution viscosity—Test Method D 2857.
5.7.4 Infrared identity test—Test Method D 2124.
5.7.5 Recommended diluents.
5.7.6 Gardner color test—Test Method D 1544.

6. Processing Recommendations for Fabrication (by Supplier)

6.1 Drying conditions before fabrication.

6.2 Suggested optimum time, temperature, and pressure for injection molding, extrusion, or thermoforming to desired shapes.

6.3 Polymer stability at elevated temperatures of dry polymer in air or under an inert atmosphere.

6.4 Recommended storage conditions.

7. Suggested Physical Tests on a Solid Specimen or Film

7.1 *Tensile Strength*—Test Methods D 412.

7.2 *Ultimate Elongation*—Test Methods D 412.

7.3 *Compression Deflection Characteristics*—Test Methods D 575.

7.4 *Tensile Stress at 100, 200, and 300 % Strain*—Test Methods D 412.

7.5 *Flexural Strength*—Test Methods D 790.

7.6 *Indentation Hardness* (*Durometer*)—Test Method D 2240.

[10] *Annual Book of ASTM Standards*, Vol 04.06.
[11] *Annual Book of ASTM Standards*, Vol 13.01.
[12] David and Staley, *Analytical Chemistry of the Polyurethanes*, Wiley Interscience Publishers, 1969, p. 359.

7.7 *Flow Rate by Extrusion Plastometer*—Test Method D 1238.
7.8 *Compression Set*—Test Methods D 395.
7.9 *Abrasion Resistance* (*Taber*)—Test Methods D 1242.
7.10 *Specific Gravity*—Test Methods D 792.
7.11 *Hydrolytic Stability of Elastomers*—Test Method D 3137.
7.12 *Tensile Creep and Creep Rupture Test*—Test Methods D 2990.
7.13 *Gas Transmission Rate of Plastic Film*—Test Method D 1434.
7.14 *Water Vapor Transmission of Plastic Film*—Test Methods E 96.
7.15 *Water Absorption of Plastics*—Test Method D 570.
7.16 *Flexural Fatigue of Plastics*—Test Method D 671.
7.17 *Thermal Analyses*—Test Method D 3418.

8. Suggested Chemical Tests

8.1 *Isocyanate Group Content*—Methods D 1638.
8.2 *Amine Equivalent Weight*. [13]
8.3 *Ash*. [14]

9. Suggested Electrical Tests

9.1 *Volume Resistivity*—Test Methods D 257.
9.2 *Surface Resistivity*—Test Methods D 257.
9.3 *Dielectric Strength*—Test Method D 149.
9.4 *Dielectric Constant*—Test Methods D 150.

10. Suggested Biological Tests

10.1 The following tests are suggested for each product line offered for sale as a raw material for fabricating biomedical devices. Additional tests may be advised for certain devices as described by Practices F 619 and F 748 and USP Class VI testing guidelines.

10.1.1 Toxicological tests on individual batches of product may be made upon agreement between the purchaser and supplier.

11. Packaging, Labeling, and Preservation

11.1 The solid polymer should be packaged in a suitable container to avoid contamination and deterioration. An airtight container, capable of excluding moisture, should be used when necessary.

11.2 The polyurethane solutions should be packaged in nonreactive, noncontaminating containers to ensure product integrity.

11.3 The product should be properly identified including product and lot numbers, and, if necessary, a storage expiration date and conditions.

12. Keywords

12.1 plastic surgical devices/applications; polymers-surgical applications; polyurethane-medical applications

[13] *Ibid.*, pp. 87–89.
[14] *U.S. Pharmacopeia*, Vol 23, 1995.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 This guide provides definitions and a standard description for thermoplastic polyurethanes in both solid and solution forms for biomedical applications. The guide enumerates relevant test methods and describes generic criteria which should assist in developing more specific specifications for implantable devices containing thermoplastic polyurethanes with values and limits covering end-use applications.

X2. BIOCOMPATIBILITY

X2.1 The suitability of these materials from a human implant perspective is dependent on the specific application. The biologic tests appropriate for the specific site, such as recommended in Practice 748 should be used as a guideline.

X2.2 No known surgical implant material has ever been shown to be completely free of adverse reactions in the human body. However, long-term clinical experience of use of specific compositions and formulations of this material class referred to in this standard has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.