Designation: F1635 – 24

Standard Test Method for *in vitro* Degradation Testing of Hydrolytically Degradable Polymer Resins and Fabricated Forms for Surgical Implants[1]

This standard is issued under the fixed designation F1635; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers *in vitro* degradation of hydrolytically degradable polymers (HDP) intended for use in surgical implants. It provides a detailed methodology for conditioning samples and suggests quantitative techniques for evaluating changes in properties (for example, mass, molar mass, and mechanical strength) over time.

1.1.1 For many types of HDPs, a correlation has been established between *in vitro* degradation per this practice and *in vivo* degradation for unloaded specimens (see X1.1.1).

1.2 The requirements of this test method apply to HDPs in various forms:

1.2.1 Virgin polymer resins, or

1.2.2 Any form fabricated from virgin polymer such as a semi-finished component of a finished product, a finished product, which may include packaged and sterilized implants, or a specially fabricated test specimen.

1.2.2.1 Use of this test method for conditioning virgin resins, semi-finished forms, or special test specimens may provide information that is useful for material and/or product development. However, those results are not sufficient for predicting the degradation behavior of the final, sterilized implant. Subsections 5.5, 8.2, and X1.2 provide additional guidance.

1.3 This standard provides guidance for mechanical loading or fluid flow, or both, when relevant to the device being evaluated. The specifics of loading type, magnitude, and frequency for a given application are beyond the scope of this test method.

1.4 This standard is not applicable to conditioning of polymers that degrade primarily through mechanisms other than hydrolysis (for example, enzymatic or oxidative degradation). See X1.1.1 for more information.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D638 Test Method for Tensile Properties of Plastics

D671 Test Method for Flexural Fatigue of Plastics by Constant-Amplitude-of-Force (Withdrawn 2002)[3]

D695 Test Method for Compressive Properties of Rigid Plastics

D747 Test Method for Apparent Bending Modulus of Plastics by Means of a Cantilever Beam (Withdrawn 2019)[3]

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D882 Test Method for Tensile Properties of Thin Plastic Sheeting

D1708 Test Method for Tensile Properties of Plastics by Use of Microtensile Specimens

D1822 Test Method for Determining the Tensile-Impact Resistance of Plastics

D2857 Practice for Dilute Solution Viscosity of Polymers

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

Current edition approved Dec. 1, 2024. Published December 2024. Originally approved in 1995. Last previous edition approved in 2016 as F1635 – 16. DOI: 10.1520/F1635-24.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

2.2 *ISO Standards:*[4]

ISO 31-8 Physical Chemistry and Molecular Physics—Part 8: Quantities and Units
ISO 10993-1 Biological Evaluation of Medical Devices—Part 1: Evaluation and Testing Within a Risk Management Process
ISO 10993-9 Biological Evaluation of Medical Devices—Part 9: Framework for Identification and Quantification of Potential Degradation Products
ISO 13781 Poly(L-lactide) resins and fabricated forms for surgical implants—In vitro degradation testing

2.3 *NIST Standard:*[5]

NIST Special Publication SP811 Guide for the Use of the International System of Units (SI)

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbable, adj—in the body*, an initially distinct foreign material or substance that either directly or through intended degradation can pass through or be assimilated by cells and/or tissue.

NOTE 1—See Appendix X3 for a discussion regarding the usage of absorbable and other related terms.

3.1.2 *hydrolytically degradable polymer (HDP)*—any polymeric material in which the primary mechanism of chemical degradation in the body is by hydrolysis (water reacting with the polymer resulting in cleavage of the chain).

3.1.3 *resin*—any polymer that is a basic material for plastics.[6]

4. Summary of Test Method

4.1 Samples of polymer resins, semi-finished components, finished surgical implants, or specially designed test specimens fabricated from those resins are placed in a buffered soaking solution at physiological pH and temperatures. Samples are periodically removed and tested for various material or mechanical properties at specified intervals. The required test intervals may vary greatly depending on the specific polymeric composition. For example, poly(L-lactide) and poly(*e*-caprolactone) degrade very slowly and can require two or more years for complete degradation. Polymers based substantially on glycolide can completely degrade in two to three months depending on the exact composition and on the size of the specimen. Degradation time is also strongly affected by specimen size, polymer molar mass, and crystallinity.

NOTE 2—The term molecular weight (abbreviated MW) is obsolete and should be replaced by the SI (Système Internationale) equivalent of either relative molecular mass (M_r), which reflects the dimensionless ratio of the mass of a single molecule to an atomic mass unit (see ISO 31-8), or molar mass (M), which refers to the mass of a mole of a substance and is typically expressed as grams/mole. For polymers and other macromolecules, use of the symbols M_w, M_n, and M_z continue, referring to mass-average molar mass, number-average molar mass, and z-average molar mass, respectively. For more information regarding proper utilization of SI units, see NIST Special Publication SP811.

5. Significance and Use

5.1 This test method is intended to help assess the degradation rates (that is, the mass loss rate) and changes in material or structural properties, or both, of HDP materials used in surgical implants. Polymers that are known to degrade primarily by hydrolysis include but are not limited to homopolymers and copolymers of *l*-lactide, *d*-lactide, *d,l*-lactide glycolide, caprolactone, and *p*-dioxanone.[7]

5.2 This test method may not be appropriate for all types of implant applications or for all known absorbable polymers. The user is cautioned to consider the appropriateness of the test method in view of the materials being tested and their potential application (see X1.1.1).

5.3 Since it is well known that mechanical loading can increase the degradation rate of absorbable polymers, the presence and extent of such loading needs to be considered when comparing *in vitro* behavior with that expected or observed *in vivo*.

5.3.1 *Mechanically Unloaded Hydrolytic Evaluation*—Conditioning of a hydrolysable device under mechanically unchallenged hydrolytic conditions at 37 °C in buffered physiological soaking solution is a common means to obtain a first approximation of the degradation profile of an absorbable material or device. It does not necessarily represent actual *in vivo* service conditions, which can include mechanical loading in a variety of forms (for example, static tensile, cyclic tensile, shear, bending, and so forth). If the performance of a device under its indicated use includes loading, hydrolytic aging alone is NOT sufficient to fully characterize the device.

5.3.2 *Mechanically Loaded Hydrolytic Evaluation*—The objective of loading is to approximate the expected device service conditions so as to better understand potential physicochemical changes that may occur. Such testing can be considered as necessary if loading can be reasonably expected under *in vivo* service conditions. When feasible, test specimens should be loaded in a manner that simulates *in vivo* conditions, both in magnitude and type of loading. Clinically relevant cyclic load tests may include testing to failure or for a specified number of cycles followed by testing to evaluate physicochemical properties.

5.3.2.1 *Static Loading*—It is notable that for some polymeric materials it has been shown that a constant load results in the same failure mechanism (for example, creep) and is the worst case when compared to a cyclic load (where the maximum amplitude of the cyclic load is equal to the constant load). Thus, in specific cases it may be acceptable to simplify the test by using a constant load even when the anticipated *in*

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, at http://physics.nist.gov/cuu/Units/bibliography.html.

[6] *Polymer Technology Dictionary*, Tony Whelan ed., Chapman & Hall, 1994.

[7] *Handbook of Biodegradable Polymers*, A. J. Domb ed., Harwood Academic Publishers, 1997.