



Designation: F 2313 – 08

Standard Specification for Poly(glycolide) and Poly(glycolide-co-lactide) Resins for Surgical Implants with Mole Fractions Greater Than or Equal to 70 % Glycolide¹

This standard is issued under the fixed designation F 2313; 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 specification covers both virgin poly(glycolide) resin and poly(glycolide-co-lactide) resin with mole fractions greater than or equal to 70 % glycolide. This specification is not applicable to glycolide:lactide copolymers with mole fractions exceeding 30 % lactide.

1.2 Since poly(glycolide) is commonly abbreviated as PGA for poly(glycolic acid) and poly(lactide) is commonly abbreviated as PLA for poly(lactic acid), these polymers are commonly referred to as PGA and PLA:PGA resins for the hydrolytic byproducts to which they respectively degrade.

1.3 This specification addresses material characteristics of both virgin poly(glycolide) and poly(>70 % glycolide-co-lactide) resins intended for use in surgical implants and does not apply to packaged and sterilized finished implants fabricated from these materials.

1.4 As with any material, some characteristics may be altered by processing techniques (such as molding, extrusion, machining, assembly, sterilization, and so forth) required for the production of a specific part or device. Therefore, properties of fabricated forms of this resin should be evaluated independently using appropriate test methods to ensure safety and efficacy.

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 may suggest use of hazardous materials, operations, and equipment. 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.*

¹ This specification is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.11 on Polymeric Materials.

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2. Referenced Documents

2.1 ASTM Standards:²

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique

D 2857 Practice for Dilute Solution Viscosity of Polymers

D 3417 Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC)³

D 3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry

D 3536 Test Method for Molecular Weight Averages and Molecular Weight Distribution by Liquid Exclusion Chromatography (Gel Permeation Chromatography—GPC)³

D 3593 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Certain Polymers by Liquid Size-Exclusion Chromatography (Gel Permeation Chromatography—GPC) Using Universal Calibration³

D 4603 Test Method for Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET) by Glass Capillary Viscometer

E 386 Practice for Data Presentation Relating to High-Resolution Nuclear Magnetic Resonance (NMR) Spectroscopy

E 473 Terminology Relating to Thermal Analysis and Rheology

E 793 Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry

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

³ Withdrawn.

- E 794 Test Method for Melting And Crystallization Temperatures By Thermal Analysis
- E 967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers
- E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters
- E 1142 Terminology Relating to Thermophysical Properties
- E 1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis
- E 1356 Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry
- E 1994 Practice for Use of Process Oriented AOQL and LTPD Sampling Plans
- F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

2.2 Other Standards:

- United States Pharmacopeia (USP) Edition 26⁴
- ISO 31-8 Physical Chemistry and Molecular Physics, Part 8: Quantities and Units⁵
- ISO 11357 Plastics—Differential Scanning Calorimetry (DSC)⁵
- ICH Q3C(R3) International Conference on Harmonisation of Technical Requirements for Registration of Pharmaceuticals for Human Use, Quality Guideline: Impurities: Residual Solvents⁶
- 21 CFR 820, United States Code of Federal Regulations, Title 21—Food and Drugs Services, Part 820—Quality System Regulation⁷
- ANSI/ISO/ASQ Q9000-2000, Quality Management Systems; Fundamentals and Vocabulary⁵
- ANSI/ISO/ASQ Q9001-2000, Quality Management Systems; Requirements⁵
- NIST Special Publication SP811 Guide for the Use of the International System of Units (SI)⁸

3. Terminology

3.1 Definition:

3.1.1 *virgin polymer, n*—the initially delivered form of a polymer as synthesized from its monomers and prior to any processing or fabrication into a medical device.

4. Materials and Manufacture

4.1 All raw monomer components and other materials contacting either the raw monomer(s) or resin product shall be of a quality suitable to allow for use of such resin in the manufacture of an implantable medical product. Such quality

includes adequate control of particles and other potential contaminants that may affect either the toxicity of or the cell response to the as-implanted or degrading final product.

4.2 All polymer manufacturing (including monomer handling, synthesis, pelletization/grinding and all subsequent) shall be undertaken under conditions suitable to allow for use of such resin in the manufacture of an implantable medical product.

5. Chemical Composition

5.1 Polymers covered by this specification shall be composed either of glycolide, or of a combination of glycolide and lactide where the lactide content does not exceed 30 % mole fraction (34.7 % by mass). To ensure such composition and the attainment of the desired properties, the following tests are to be conducted.

5.2 Chemical Identification:

5.2.1 The identity of the virgin polymer shall be confirmed either by infrared, ¹H-NMR, or ¹³C-NMR spectroscopy.

5.2.2 Infrared Identification:

5.2.2.1 Identity of either poly(glycolide) homopolymer or poly(glycolide-co-lactide) copolymer may be confirmed through an infrared spectrum exhibiting major absorption bands only at the wavelengths that appear in a suitable reference spectrum. Analysis shall be conducted using practices similar to those described in Practice E 1252. A typical infrared transmission reference spectrum for PGA homopolymer is shown in Fig. 1. A typical infrared transmission reference spectrum for a 90 % PGA:10 % L-PLA copolymer is shown in Fig. 2. While poly(glycolide-co-lactide) copolymers will each have their own respective spectrum that will vary in response to copolymer ratio, this analytic method typically lacks sensitivity sufficient for quantification of copolymer ratio as specified in 7.1.2.

5.2.2.2 Additional spectral bands may be indicative of known or unknown impurities, including residual solvents and catalysts (refer to limits specified in Table 1).

5.2.3 Proton Nuclear Magnetic Resonance (¹H-NMR) Identification:

5.2.3.1 Identity of either poly(glycolide) homopolymer or poly(glycolide-co-lactide) copolymer may be confirmed through sample dissolution, ¹H-NMR spectroscopy, and the use of a suitable reference spectrum. Sample dissolution is in deuterated hexafluoroisopropanol (D-HFIP) or other proton-free solvent able to fully solvate the specimen. Analysis shall be conducted using practices similar to those described in Practice E 386.

5.2.3.2 Additional spectral bands may be indicative of known or unknown impurities, including residual solvents and catalysts (refer to limits specified in Table 1).

5.2.4 Carbon-13 Nuclear Magnetic Resonance (¹³C-NMR) Identification:

5.2.4.1 Identity of either poly(glycolide) homopolymer or poly(glycolide-co-lactide) copolymer may be confirmed in a solid state through ¹³C-NMR spectroscopy and the use of a suitable reference spectrum. Analysis shall be conducted using practices similar to those described in Practice E 386.

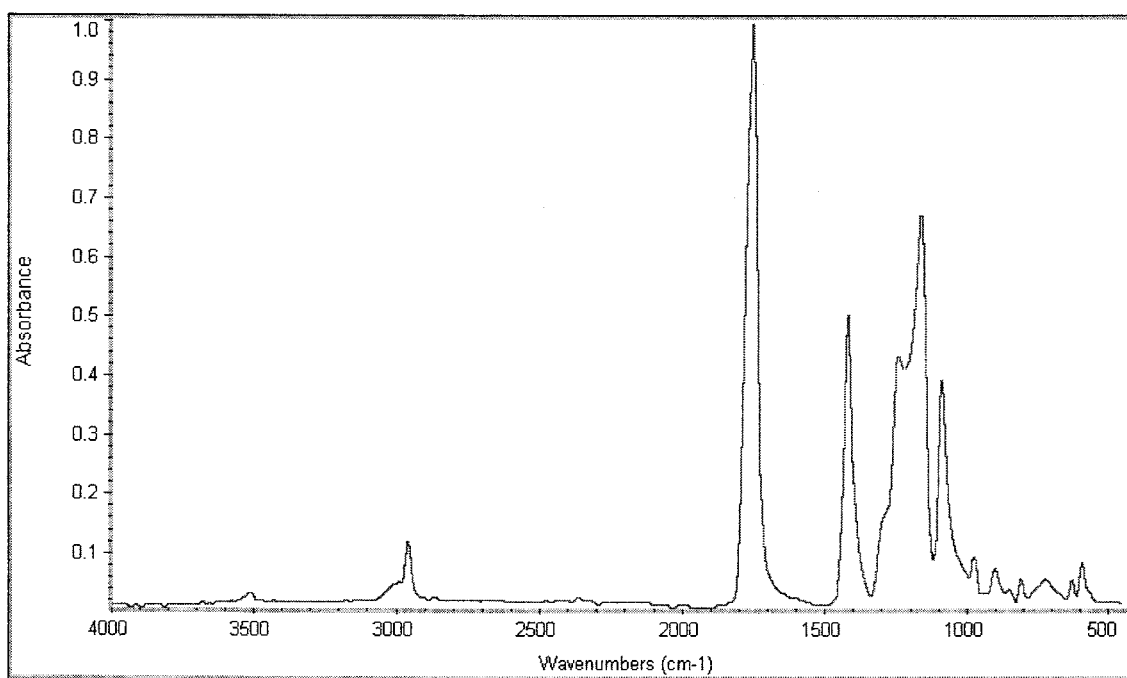
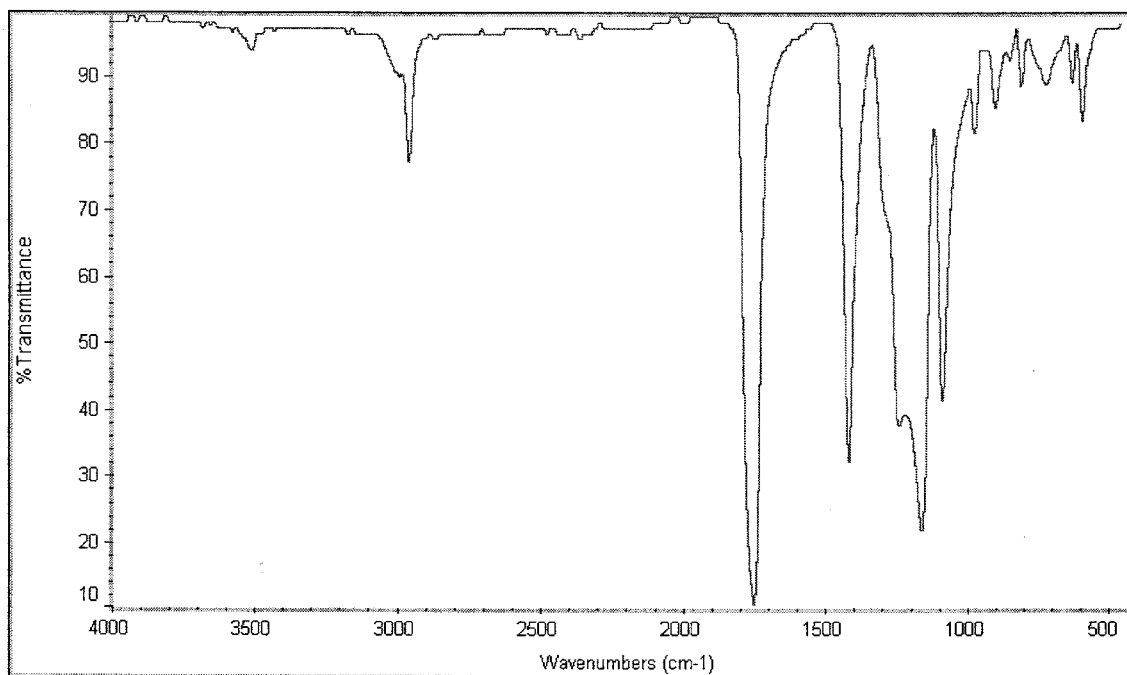
⁴ Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, <http://www.usp.org>.

⁵ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁶ Available from ICH Secretariat, c/o IFPMA, 30 rue de St-Jean, P.O. Box 758, 1211 Geneva 13, Switzerland. Available online at <http://www.ich.org/LOB/media/MEDIA423.pdf>

⁷ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

⁸ 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>.



NOTE—Supplied example infra-red spectrum is of “Dexon Medical Suture (beige)” as acquired from the Hummel Polymer Library, available from: Thermo Nicolet Corporation, 5225 Verona Road, Madison, WI 53711-4495, USA.

FIG. 1 Poly(glycolide) Resin Infrared Spectrum