Designation: F 641 – 98a (Reapproved 2003)

Standard Specification for Implantable Epoxy Electronic Encapsulants[1]

This standard is issued under the fixed designation F 641; 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 thermoset plastics based on diglycidyl ethers of bisphenol A and amino functional curing agents or amine catalysts.

1.2 The epoxy encapsulants covered by this specification are intended to provide a tissue-compatible protective covering for implantable medical devices such as pulse generators, telemetry devices and RF receivers. The biocompatibility of epoxy plastics has not been established. Epoxy plastic is a generic term relating to the class of polymers formed from epoxy resins, certain curing agents or catalysts and various additives. Since many compositions and formulations fall under this category, it is essential that the fabricator assure safety of implantability of the specific composition or formulation for the intended use by current state-of-the-art test methods. This specification can be used as a basis for standardized evaluation of biocompatibility for such implantable encapsulants.

1.3 The encapsulants covered by this specification are for use in devices intended as long-term implants.

1.4 *Limitations*—This specification covers only the initial qualification of epoxy encapsulants for implantable electronic circuitry. Some of the requirements are not applicable to routine lot to lot quality control.

1.5 *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 570 Test Method for Water Absorption of Plastics[3]

D 638 Test Method for Tensile Properties of Plastics[3]

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[3]

D 883 Terminology Relating to Plastics[3]

D 1042 Test Method for Linear Dimensional Changes of Plastics Under Accelerated Service Conditions[3]

D 1239 Test Method for Resistance of Plastic Films to Extraction by Chemicals[3]

D 1434 Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting[4]

D 1763 Specification for Epoxy Resins[3]

D 1898 Practice for Sampling of Plastics[5]

D 2240 Test Method for Rubber Property—Durometer Hardness[6]

D 2471 Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins[7]

D 2562 Practice for Classifying Visual Defects in Parts Molded from Reinforced Thermosetting Plastics[7]

D 2566 Test Method for Linear Shrinkage of Cured Thermosetting Casting Resins During Cure[8]

D 2734 Test Method for Void Content of Reinforced Plastics[7]

D 3137 Test Method for Rubber Property—Hydrolytic Stability[6]

F 74 Practice for Determining Hydrolytic Stability of Plastic Encapsulants for Electronic Devices[9]

F 135 Test Method for Embedment Stress Caused by Casting Compounds on Glass-Encased Electronic Components[10]

F 602 Criteria for Implantable Thermoset Epoxy Plastics[11]

F 748 Practice For Selecting Generic Biological Test Methods for Materials and Devices[11]

[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.

Current edition approved Apr. 10, 2003. Published May 2003. Originally approved in 1979. Last previous edition approved in 1998 as F 641 – 98a.

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

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

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

[5] Discontinued; See 1997 *Annual Book of ASTM Standards*, Vol 08.01.

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

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

[8] Discontinued; See 1992 *Annual Book of ASTM Standards*, Vol 08.02.

[9] Discontinued; See 1994 *Annual Book of ASTM Standards*, Vol 10.04.

[10] Discontinued; See 1996 *Annual Book of ASTM Standards*, Vol 10.02.

[11] *Annual Book of ASTM Standards*, Vol 13.01.

F 895 Test Method for Agar Diffusion Cell Culture Screening for Cytotoxicity[11]

F 981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone[11]

2.2 *AAMI Standard:*

EOS-D E-O Sterilization Standard[12]

3. Classification

3.1 Encapsulants shall be classified as follows:

3.1.1 *Type I*—Those encapsulants which contact the tissue directly or indirectly.

3.1.2 *Type II*—Those encapsulants used only within hermetically sealed containers. The epoxy encapsulant has no contact with tissues or physiological fluids.

4. Chemical Composition

4.1 *Additives* (Type I Encapsulants Only):

4.1.1 *Reactive Diluents*—The following compounds when used as reactive diluents shall not be used in concentrations greater than 12 parts per hundred resin (phr).

4.1.1.1 Butyl glycidyl ether (BGE).

4.1.1.2 Phenyl glycidyl ether (PGE).

4.1.2 *Other Additives* (see Note 1)—Other additives shall be shown to be nonextractable in 37°C physiological saline for the device design life in concentrations sufficient to significantly affect the properties of the encapsulant or to produce a significant biological reaction.

NOTE 1—Other additives, as indicated in Criteria F 602, include compounds such as nonreactive diluents, fillers, release agents, and the like.

4.1.3 *Phthalate Esters*—Phthalate esters such as dibutyl phthalate shall not be used in concentrations ≥10 phr.

4.2 *Mix Ratios* (Type I and Type II Encapsulants):

4.2.1 *Amines*—The mix ratio shall be maintained at ±5 equivalent % of stoichiometry.

4.2.2 *Catalysts*—The mix ratio shall be maintained within the ranges recommended by the formulator.

4.3 *Carbonates* (Type I and Type II Encapsulants)—The encapsulant shall be poured under conditions such that the formation of amine carbonates is minimized. The device manufacturer may specify maximum limits of carbon dioxide or water vapor, or both, in the atmosphere in which the encapsulant is being mixed or poured.

5. Physical Properties

5.1 *Type I Encapsulants*:

5.1.1 *Peak Exotherm Temperature* (Test Method D 2471)—The peak exotherm temperature during cure shall be kept below the maximum acceptable value for the lowest temperature rated component of the device.

5.1.2 *Fully Cured Specimens*—The required properties measured on fully cured specimens conditioned as in 6.1 are as follows:

5.1.2.1 *Transparency*—In cases where no fillers or reinforcements are used, the encapsulant shall have sufficient transparency so that the circuitry may be visually inspected after encapsulation.

5.1.2.2 *Foreign Particles*—No foreign particles, particulate matter and gross contamination shall be observed when checked under 2× wide field magnification.

5.1.2.3 *USP Biological Tests Plastic Containers, Class IV*[13]—Pass.

5.1.2.4 USP Pyrogen Test[14] or other Pyrogen methods which have been demonstrated to be of equal or greater sensitivity—Pass.

5.1.2.5 *Sterilant Residues* (AAMI EOS-D)—Where applicable, the concentration of ethylene oxide, ethylene chlorohydrin, ethylene glycol, and dichlorodifluoromethane (or the equivalents) at the time of implant shall be shown to be within safe limits prescribed by the device manufacturer.

5.1.2.6 The cure shrinkage (Test Method D 2566) or embedment stress (Test Method F 135) shall be ≤2%. The stress shall not exceed the limits of the most pressure-sensitive components.

5.1.2.7 Tissue Culture Test (Agar Overlay)[15] or Test Method F 895—Pass.

5.1.2.8 While cell culture methods as described in Test Method F 895 may be appropriated for the batch-to-batch screening of fully cured specimens, the basic recipe used should have been qualified for its overall tissue response by methods such as those suggested in Practice F 748 for "Implanted Devices Principally Contacting Tissue and Tissue Fluid" including testing according to Practice F 981.

5.1.3 *Required Cured Properties Measured in Long-Term Immersion Tests for Type I Encapsulants*—The property values prescribed in Table 1 shall be obtained at 22 ± 3°C and 50 ± 10 % relative humidity on specimens conditioned as in 6.3. Samples shall be wiped dry prior to test with a lint-free tissue, as appropriate.

5.1.4 Optional cured properties measured after accelerated immersion for Type I encapsulants may be determined for screening purposes after conditioning as in 6.2.

5.2 *Type II Encapsulants*:

5.2.1 *Peak Exotherm Temperature* (Test Method D 2471)—The peak exotherm temperature during cure shall be kept below the maximum acceptable value for the lowest temperature rated component of the device.

5.2.2 The property values prescribed in Table 2 shall be determined at 22 ± 3°C (71.6 ± 5.7°F) and 50± 10 % relative humidity on fully cured samples conditioned as in 6.1.

6. Specimen Preparation

6.1 *Preparation*—Prepare specimens used for evaluation of properties of the cured material in the same manner as the intended product. Such conditioning shall include all specified relevant variables for the product prior to implant including

[12] Available from Association for Advancement of Medical Instrumentation, 1500 Wilson Blvd., Suite 417, Arlington, VA 22209.

[13] *U.S. Pharmacopeia*, XXIII, 1995, pp. 1783-1787.

[14] *Ibid.*, pp. 1696-1697.

[15] Guess, W. L., et al., *Journal of Pharmaceutical Sciences*, Vol 54, 1965, pp. 1545–1547.