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Standard Criteria for Implantable Thermoset Epoxy Plastics[1]

This standard is issued under the fixed designation F602; 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 These criteria cover thermoset plastics based on diglycidyl ethers of bisphenol A (DGEBPA) and appropriate curing agents or catalysts as opposed to thermoplastics based on epoxy structures.

1.2 These criteria are generic and are intended to provide definitions and a standard description of epoxy plastics used in implantable devices. It is also intended to serve as a standard guide for the preparation of more specific documents with values and limits covering specific end uses.

1.3 Compliance with these criteria shall not be construed as an endorsement of implantability. The biocompatibility of epoxy plastics as a class 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 class, it is essential that the formulator or fabricator ensure biocompatibility of the specific composition or formulation in its intended end use. Since these criteria provide guidance for the preparation of more specific documents covering specific end uses, these documents will provide bases for standardized evaluation of biocompatibility appropriate for a specific end use.

1.4 Each of the properties listed shall be considered in selecting materials for specific end uses. A list of selected properties with limiting values assigned is suggested for separate product specifications.

1.5 All of the properties and test methods listed may not be pertinent in any specific situation, nor may all of the tests outlined be required.

1.6 These criteria are limited to functionally or fully cured epoxy plastics. Uncured or incompletely cured formulations are specifically excluded.

1.7 The epoxy plastics covered by this standard are those to be evaluated for use in implantable biomedical devices. The term implantable is herein considered to include devices used in vivo for time periods in excess of 30 days.

1.8 *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:*[2]

D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

D150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation

D257 Test Methods for DC Resistance or Conductance of Insulating Materials

D570 Test Method for Water Absorption of Plastics

D621 Test Methods for Deformation of Plastics Under Load (Withdrawn 1994)[3]

D638 Test Method for Tensile Properties of Plastics

D785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D952 Test Method for Bond or Cohesive Strength of Sheet Plastics and Electrical Insulating Materials

D1042 Test Method for Linear Dimensional Changes of Plastics Caused by Exposure to Heat and Moisture

D1434 Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting

D1763 Specification for Epoxy Resins

D2393 Test Method for Viscosity of Epoxy Resins and Related Components (Withdrawn 1992)[3]

D2471 Practice for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins (Withdrawn 2008)[3]

D2562 Practice for Classifying Visual Defects in Parts

[1] These criteria are under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devicesand are the direct responsibility of Subcommittee F04.11 on Polymeric Materials.

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

Molded from Reinforced Thermosetting Plastics
D2734 Test Methods for Void Content of Reinforced Plastics
D3137 Test Method for Rubber Property—Hydrolytic Stability
E96/E96M Test Methods for Water Vapor Transmission of Materials
F74 Practice for Determining Hydrolytic Stability of Plastic Encapsulants for Electronic Devices (Withdrawn 1994)[3]
F619 Practice for Extraction of Medical Plastics
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

2.2 *AAM1 Standard:*
EOS-D 10/75 Standard for Ethylene Oxide Sterilization[4]

2.3 *ISO Standard:*
ISO 10993 Biological Evaluation of Medical Devices[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *accelerator*—an additive used to increase the rate of cure. An accelerator may also be a catalyst, or it may actually change composition and, therefore, not qualify as a catalyst.

3.1.2 *additive*—a chemical added to epoxy resins or hardeners to modify the handling characteristics or cured properties, or both, of the epoxy-hardener combination.

3.1.2.1 *diluent*—a chemical used in admixture to modify or enhance the properties of either or both the uncured or cured formulations. A primary use is to reduce the viscosity of the mixed system although other properties such as exotherm rate, stiffness, moisture absorption, and so forth, may be modified or enhanced also.

3.1.2.1.1 *nonreactive diluent*—a diluent not containing chemically reactive functional groups.

3.1.2.1.2 *reactive diluent*—a diluent that reacts chemically with the epoxy resin or hardener, or both, during cure.

3.1.2.2 *filler*—a relatively inert solid particulate material added to an epoxy formulation to modify its strength, permanence, working properties, or other qualities, or to lower costs.

3.1.3 *curing agent or hardener*—a compound normally used in a predetermined concentration to react chemically (copolymerize) by means of several different mechanisms (for example, condensation or addition polymerization) with or without heat or pressure in order to change its form from a liquid or fusible, friable, soluble solid to an infusible, insoluble solid having useful and desirable application or end-use properties.

3.1.3.1 *initiator*—an additive used to cause a thermosetting resin to react with itself (polymerize). Usually, these additives—used in relatively very small amounts—initiate homo-polymerization of the epoxy resin resulting in ether linkages.

NOTE 1—The term "catalyst" is frequently misused to denote any material added to a resin to cause a reaction to occur. This usage should be discouraged. The Society of Plastics Industries defines a catalyst as "a compound which alters the speed of a reaction without changing its original composition."

3.1.4 *epoxy*—oxirane ring structures.

$$\mathrm{R{-}CH(O){-}CH_2}$$

3.1.4.1 *epoxy plastic*—thermoplastic or thermosetting plastics containing ether or hydroxyalkyl repeating units or both, resulting from the ring-opening reactions of lower molecular weight polyfunctional oxirane resins or compounds, with catalysts or with various polyfunctional acidic or basic coreactants.

3.1.4.2 *epoxy resin*—generally, any resin (liquid or solid) with a chemical structure at least difunctional in oxirane. Specifically for this standard, the diglycidyl ethers of bisphenol A or the equivalent. These compounds are defined as Grade 1 in Specification D1763.

3.1.5 *Terms Relating to Cure: —*

3.1.5.1 *cure, v*—to change the properties of a polymeric system into a final, more stable, usable condition by the use of heat, radiation, or reaction with chemical additives.

3.1.5.2 *cure cycle*—the schedule of time periods at specified conditions to which a reacting thermosetting material is subjected to reach a specified property level.

3.1.5.3 *cure time*—the interval of time from the start of reaction to the time at which specified properties of the reacting thermosetting composition are reached. For materials that react under the conditions of mixing, the start of reaction is the time of initial exposure to the conditions necessary for reaction to occur.

3.1.5.4 *functionally cured*—the term used to denote an epoxy plastic that has attained sufficient cure to achieve stable properties.

3.1.5.5 *fully cured*—the term used to denote total disappearance of epoxy groups as detected by infrared spectroscopy, or other equally sensitive physicochemical methods.

3.1.5.6 *one-component system*—a formulation based on an epoxy resin preblended with a heat, moisture, or otherwise activated curing agent or catalyst. The mixture is storable but cures under the appropriate activation conditions.

3.1.5.7 *postcure*—the additional and separate curing operations to which a "hardened" thermosetting plastic composition is subjected in order to enhance one or more properties. Also used to ensure stabilization of physical properties under use conditions.

3.1.5.8 *two-component system*—a formulation based on an epoxy resin to which a curing agent or catalyst is added just prior to use.

4. Chemical Composition

4.1 *Epoxy Resins*—Oxirane-terminated reaction products of epichlorohydrin and bisphenol A (DGEBPA) or the equivalent.

4.2 *Reactive Diluents*—The following are examples of compounds that may be included as reactive diluents:

4.2.1 Butyl glycidyl ether (BGE).

[4] Available from the Association for the Advancement of Medical Instrumentation, 1901 N. Ft. Myer Dr., Suite 602, Arlington, VA 22209.

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

4.2.2 Phenyl glycidyl ether (PGE).

4.3 *Nonreactive Diluents*—The following are examples of compounds that may be included as nonreactive diluents:
4.3.1 Phthalate esters.
4.3.2 Nonyl phenols.
4.3.3 Miscible polymers.
4.3.4 Flexibilizers.

4.4 *Fillers*—The following are examples of fillers that may be incorporated in the formulations:
4.4.1 Silicas:
4.4.1.1 Fumed silica.
4.4.1.2 Precipitated hydrated silica.
4.4.1.3 Diatomaceous earth.
4.4.2 Carbons.
4.4.3 Certain radiopaque materials:
4.4.3.1 Certain inorganic nonmetallic particles.
4.4.3.2 Certain metallic particles.
4.4.4 Certain pigments.
4.4.5 Glass fibers.
4.4.6 Glass ceramic particles.
4.4.7 Glass or plastic microballoons.

4.5 *Other Additives*—The following are examples of additives that may be used in the formulation:
4.5.1 Slip agents.
4.5.2 Optical brighteners.
4.5.3 Surfactants.

4.6 *Curing Agents*—The following are examples of curing agents for epoxy resins:
4.6.1 Amines (primary, secondary, and tertiary) such as triethylenetetramine (TETA).
4.6.2 Anhydrides, such as phthalic anhydride.
4.6.3 Acids such as phthalic acid.
4.6.4 Amine-terminated polyamides.
4.6.5 Acid or amine-terminated telomers.
4.6.6 Schiff's bases.

4.7 *Catalysts:*
4.7.1 Lewis bases such as tertiary amines.
4.7.2 Lewis acids such as BF_3.

4.8 *Accelerators:*
4.8.1 Tertiary amines.
4.8.2 Phenols.

NOTE 2—Since some curing agents and catalysts may be toxic by themselves, it may be necessary in specific end-use standards to require tests to limit their presence in the final product.

5. Physical Requirements

5.1 *Uncured Properties*—The following test methods may be conducted on the uncured mixed formulation or appropriate components:
5.1.1 *Peak Exotherm Temperature*—Test Method D2471.
5.1.2 *Gel Time*—Test Method D2471.
5.1.3 *Mix Ratio*—The mix ratio shall be calculated and maintained at the ratio recommended by the manufacturer of the formulation.
5.1.4 *Viscosity*—Test Method D2393.

5.2 *Cured Properties* (Required)—The following test methods shall be conducted on the fully cured and properly conditioned material.
5.2.1 *Extraction*—Practice F619.
5.2.2 *Foreign Particles*—Upon careful visual examination, the epoxy plastic shall be free of any extraneous debris that may adversely affect its safety, efficacy, or reliability.
5.2.3 *Hydrolytic Stability*—Practice F74 or Test Method D3137.
5.2.4 *Biocompatibility Testing*—Biocompatibility of polymers and implant devices made using these materials shall be determined in accordance with Practice F748 or the ISO 10993 series, unless otherwise agreed upon between the supplier and the consumer and regulating bodies. These standards recommend specific biocompatibility testing, depending on the intended use of the device.
5.2.4.1 Biocompatibility testing should be performed on specimens that have been processed and sterilized using the methods intended for the final device.
5.2.5 *Sterilant Residues and Sterilization Effects*—Testing regarding applicable methods of sterilization should be documented. In addition to degassing time necessary for EtO sterilization, the stability of the epoxy under steam and radiation sterilization should be specified if these types of sterilization are called for.
5.2.5.1 Sterilant residues shall be tested according to appropriate methods, such as AAMI EOS-D. The concentration of ethylene oxide, ethylene chlorohydrin, ethylene glycol, and dichlorodifluoromethane (or the equivalents) at the time of implantation shall be shown to be within safe limits prescribed by the device manufacturer. Cell culture tests can be used to show the absence of sterilant residues. When materials are sterilized by radiation, materials subjected to maximum radiation dose shall be qualified by performance tests.
5.2.6 *Water Absorption*—Test Method D570.

5.3 *Cured Properties* (Optional)—The following test methods shall be conducted on the fully cured and properly conditioned material as is appropriate for the end use:
5.3.1 *Adhesion*—Test Method D952.
5.3.2 *Bacteriostasis and Fungistasis*—Sterility Tests.[6]
5.3.3 *Compression Set*—Test Methods D621.
5.3.4 *Dielectric Constant*—Test Methods D150.
5.3.5 *Dielectric Strength*—Test Method D149.
5.3.6 *Dissipation Factor*—Test Methods D150.
5.3.7 *Elongation*—Test Method D638.
5.3.8 *Flexural Strength*—Test Method D1434.
5.3.9 *Gas Permeation*—Test Method D1434.
5.3.10 *Hardness*—Test Method D785.
5.3.11 *Moisture Vapor Transmission*—Test Methods E96/E96M.
5.3.12 *Prothrombin Time*—The effect of the material on the Prothrombin time shall be tested by appropriate methods.[7]
5.3.13 *Specific Gravity*—Test Methods D792.

[6] *United States Pharmacopeia,* available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, http://www.usp.org.
[7] *Human Blood Coagulation, Haemostasis, and Thrombosis*, Rosemary Biggs, ed., Blackwell Scientific Publications, 1972.

5.3.14 *Stability* (dimensional)—Test Method D1042.

5.3.15 *Stypven Time*—The effect of the material on the Stypven time shall be tested by appropriate methods.[6]

5.3.16 *Surface Resistivity*—Test Methods D257.

5.3.17 *Tangent Modulus*—Test Method D638.

5.3.18 *Tensile Strength*—Test Method D638.

5.3.19 *Visual Defects*—Practice D2562.

5.3.20 *Voids*—Test Method D2734.

5.3.21 *Volume Resistivity*—Test Method D257.

6. Identification

6.1 The following analytical methods may be used to characterize the materials:

6.1.1 Infrared spectroscopy.

6.1.2 Spectrographic analysis.

6.1.3 X-ray emission or diffraction.

7. Marking

7.1 The labels shall bear appropriate statements as to safety and handling precautions for each component and shall bear appropriate lot numbers.

8. Packaging

8.1 Packaging shall provide appropriate protection for the compound(s).

9. Keywords

9.1 epoxy (EP) plastics-surgical implants; plastics (thermosetting); plastic surgical devices/applications; polymers-surgical applications; resins-epoxy

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 This document provides definitions and a standard description for thermoset epoxy plastics based on diglycidyl ethers of bisphenol A and appropriate curing agents and catalysts, compositions which are used in the manufacture of implantable devices. The guide enumerates relevant test methods and describes generic criteria which should assist in developing more specific specifications for implantable devices containing epoxy resins with values and limits covering end-use applications. This document should also help the fabricator to select ingredients for the medical device that ensure its biocompatibility.

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 F748 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.