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.

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Standard Guide for Silicone Elastomers, Gels, and Foams Used in Medical Applications Part II—Crosslinking and Fabrication[1]

This standard is issued under the fixed designation F2042; 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 guide is intended to educate potential users of silicone elastomers, gels and foams relative to their fabrication and processing. It does not provide information relative to silicone powders, fluids, pressure sensitive adhesives, or other types of silicone products.

1.2 The information provided is offered to guide users in the selection of appropriate processing conditions for specific medical device applications.

1.3 Formulation and selection of appropriate starting materials is covered in the companion document, F2038. This monograph addresses only the curing, post-curing, and processing of elastomers, gels and foams as well as how the resulting product is evaluated.

1.4 Silicone biocompatibility issues can be addressed at several levels, but ultimately the device manufacturer must assess biological suitability relative to intended use. Biocompatibility testing may be done on cured elastomers prior to final fabrication, but the most relevant data are those obtained on the finished device. Data on selected lots of material are only representative when compounding and fabrication are performed under accepted quality systems such as ISO 9001 and current Good Manufacturing Practice Regulations (21 CFR, Parts 210, 211, and 820). Extractables analyses may also be of interest for investigation of biocompatibility, and the procedures for obtaining such data depend on the goal of the study (see ISO 10993–12 and the HIMA Memorandum 7/14/93 for examples of extraction methods).

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered 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.* Users are also advised to refer to Material Safety Data Sheets provided with uncured silicone components.

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]

D395 Test Methods for Rubber Property—Compression Set

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D430 Test Methods for Rubber Deterioration—Dynamic Fatigue

D624 Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers

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

D813 Test Method for Rubber Deterioration—Crack Growth

D814 Test Method for Rubber Property—Vapor Transmission of Volatile Liquids

D926 Test Method for Rubber Property—Plasticity and Recovery (Parallel Plate Method)

D955 Test Method of Measuring Shrinkage from Mold Dimensions of Thermoplastics

D1349 Practice for Rubber—Standard Conditions for Testing

D1566 Terminology Relating to Rubber

D2240 Test Method for Rubber Property—Durometer Hardness

F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

[1] This guide 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] 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.

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2.2 *Other Biocompatibility Standards:*

FDA Guidance on Use of International Standard ISO 10993-1, "Biological evaluation of medical devices—Part 1: Evaluation and testing within a risk management process"—Guidance for Industry and Food and Drug Administration Staff[3]

ISO 10993–1 Biological evaluation of medical devices, Part 1: Evaluation and testing within a risk management process[4]

ISO 10993-12 Biological evaluation of medical devices—Part 12: Sample preparation and reference materials[4]

HIMA Memorandum Guidance for Manufacturers of Silicone Devices Affected by Withdrawal of Dow Corning Silastic Materials, 7/14/93[5]

2.3 *Sterilization Standards:*

ANSI/AAMI ST79 Comprehensive guide to steam sterilization and sterility assurance in health care facilities[4]

ANSI/AAMI ST50 Dry Heat (Heated Air) Sterilizers[4]

ISO 10993-7 Biological evaluation of medical devices—Part 7: Ethylene oxide sterilization residuals[4]

ISO 11137-1 Sterilization of health care products—Radiation Part 1: Requirements for development, validation and routine control of a sterilization process for medical devices[4]

ISO 11137-2 Sterilization of health care products—Radiation Part 2: Requirements for development, validation and routine control of a sterilization process for medical devices[4]

2.4 *Quality Standards:*

ISO 9001 Quality Management Systems—Requirements[4]

21 CFR 820 Quality System Regulation[6]

21 CFR 210 Current Good Manufacturing Practice in Manufacturing, Processing, Packing or Holding of Drugs: General[6]

21 CFR 211 Current Good Manufacturing Practice for Finished Pharmaceuticals[6]

2.5 *Other Standards:*

Dow Corning CTM 0155 (Gel-Like Materials With Modified Penetrometer)

Dow Corning CTM 0813 (Gel-Like Materials With One Inch Diameter Head Penetrometer)

PCB Test Methods such as those used for MRI Project No. 4473, Jan 24, 1997[7]

[3] Available from Food and Drug Administration (FDA), 10903 New Hampshire Ave., Silver Spring, MD 20993-0002, http://www.fda.gov.

[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 Advanced Medical Technology Association, 1200 G St. N.W. Suite 400 Washington, D.C. 20005–3814, http://www.advamed.org.

[6] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

[7] Available from Midwest Research Institute, 425 Volker Blvd., Kansas City, MO 64110–2299, Ph: (816) 753-7600.

3. Terminology

3.1 The classification of silicone elastomers is based upon a number of interrelated factors which include the chemical system used to crosslink the elastomer, the physical characteristics of the uncured elastomer, and the methods used to fabricate the elastomers. Additional pertinent terms are defined in standard D1566.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *manufacture*—the process which occurs in the supplier's facility in which the various components of the elastomer are brought together, allowed to interact, and are packaged to provide the uncured elastomer for sale.

3.2.2 *fabrication*—the process by which the uncured elastomer is converted into a fully vulcanized elastomer of the desired size and shape. This process may occur in the same facility as the manufacture of the uncured elastomer but is more typically performed at the facility of a customer of the silicone manufacturer.

3.2.2.1 *injection molding*—fabrication of elastomers into forms defined by molds constructed so that the uncured elastomer can be transferred by pumping into the closed mold. This method requires venting of the mold in some manner. The elastomer may be vulcanized by heating the mold after it is filled but more typically the molding conditions (temperature and filling rate) are adjusted so that uncured elastomer can be added to a pre-heated mold in which it will then cure. The mold is then opened and the part removed and post-cured, if necessary.

3.2.2.2 *compression molding*—a process in which the uncured elastomer is placed in an open mold. The mold is closed and pressure applied to the mold to fill the cavity. Heat is applied to vulcanize the elstomer, the mold is then opened and the fabricated part is removed.

3.2.2.3 *freshening*—because of the interaction that can occur between the fumed silica and silicone polymers, thick uncured high consistency elastomers can become so stiff over time that they are very difficult to process. To overcome this problem, a two–roll mill is used to disrupt this interaction, resulting in a material which is easier to fabricate. This process is called freshening and is typically done immediately before catalyzation.

3.2.2.4 *transfer molding*—a process in which the mixed, uncured elastomer is placed in a compartment connected to the mold. The compartment is then closed, pressure is applied to transfer the uncured elastomer to the mold, filling the cavity. Heat and pressure are applied to the mold to vulcanize the elastomer, the mold is then opened, and the fabricated part is removed.

3.2.2.5 *extrusion*—a continuous process in which the mixed, uncured elastomer is forced through an orifice having the desired cross-sectional profile. The elastomer is then vulcanized by passing it through either a hot air or radiant heat oven. The most common application of extrusion processing is the fabrication of tubing but it can be used to produce other items as well.

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3.2.2.6 *post-cure*—the process of subjecting a vulcanized elastomer to elevated temperature, usually in a hot-air oven, after its initial fabrication. This process step is done to complete cross-linking of the object, remove peroxide by-products, and eliminate changes in its physical properties. Post-cure is often necessary when the component is only partially cross-liked by molding; it is performed in an attempt to accelerate the molding process, and increase its output.

3.2.2.7 *calendaring*—the process of forming an uncured, mixed elastomer into a thin sheet or film by passing it between two rolls.

3.2.2.8 *dispersion*—the process of placing an uncured elastomer in a solvent. This lowers the viscosity of the material and is usually done to allow the fabrication of thinner films than can be obtained by calendaring or to form coatings. Following dispersion use, the solvent must be removed either before or during the vulcanization process. Care must be taken to assure that the solvent is compatible with the elastomer, to prevent preferential settling of the components of the formulation by excessive dilution of the elastomer.

3.2.3 *one-part elastomer*—an elastomer supplied in the uncured form in one package containing all of the formulation components. It does not require mixing before fabrication.

3.2.4 *two-part elastomer*—an elastomer supplied in two packages which must be mixed in specified proportions before fabrication.

3.2.5 *liquid silicone rubber or low consistency silicone rubber (LSR)*—an elastomer having a viscosity such that it can be moved or transferred by readily available pumping equipment. LSRs are typically used in injection molding operations.

3.2.6 *high consistency rubber (HCR)*—an elastomer having a viscosity such that it cannot be moved or transferred by readily available pumping equipment. These elastomers are fabricated using high shear equipment such as a two-roll mill and cannot be injection molded. They are typically used in compression or transfer molding and extrusion processes.

3.2.7 *room temperature vulcanization (RTV)*—a one-part elastomer which cures in the presence of atmospheric moisture. Little, if any, acceleration of cure rate is realized by increasing temperature. Because cure is dependent upon diffusion of water into the elastomer, cure in depths of greater than 0.64 cm is not recommended.

3.2.8 *gel*—a lightly crosslinked material having no or relatively low levels of reinforcement beyond that provided by the crosslinked polymer. Gels are usually two-part formulations utilizing a platinum-catalyzed addition cure system. The hardness of the gel can be adjusted within wide limits. The material is not usually designed to bear a heavy load but rather to conform to an irregular surface providing intimate contact. As a result, loads are distributed over a wider area. These materials may also be used to provide protection from environmental contaminants.

3.2.9 *foam*—a crosslinked material which has a component added to it which generates a volatile gas that creates gas-filled cells as the material is being vulcanized. This vulcanization process results in a material with a relatively low density. Foams are usually two-part formulations utilizing a platinum-catalyzed addition cure system. They conform as they expand to irregular surfaces just as gels do to provide intimate contact and protection from the environment but are more rigid and provide more strength than gels. Since foams are expanded elastomers, on a weight basis, they are highly crosslinked relative to gels. Most cure conditions will result in a closed cell foam.

4. Significance and Use

4.1 This guide is intended to provide guidance for the specification and selection of fabrication methods for silicones used in medical devices. It also provides guidance relative to testing that might be done to qualify lots of acceptable material, based on desired performance properties.

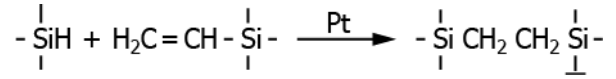
4.2 Silicone manufacturers supplying material to the medical device industry should readily provide information regarding non-proprietary product formulation to their customers either directly or through the US FDA Master File program.

5. Crosslinking Chemistry

5.1 Silicone elastomers used in medical applications are typically crosslinked by one of three commonly used cure systems. These involve the platinum-catalyzed addition of a silylhydride to an unsaturated site, the generation of free radicals by a peroxide, or the reaction of an easily hydrolyzable group of silicon.

5.1.1 *addition cure*—this cure system utilizes the addition of a silylhydride to a site of unsaturation, usually a vinyl group. As shown in Fig. 1, this reaction is catalyzed by a platinum complex. The catalyst will be present at a level such that the concentration of platinum is in the range of 5 to 20 ppm but is more typically present at a level of about 7.5 ppm. When multiple silylhydrides are present in the same molecule, for example in a crosslinker molecule, and they react with vinyl groups attached to a silicon in a silicone polymer, a crosslinked network results.

Elastomers using this cure system are two-part elastomers and are utilized in both LSRs and HCRs. In practice, the platinum catalyst, an inhibitor, and vinyl functionality on the silicone backbone are present in one part of the formulation and the crosslinker in the presence of vinyl functionality on the silicone backbone is present in the other. These two parts are intimately mixed shortly before they are intended to be used. At room temperature a certain amount of working time (time before the crosslink network builds to unacceptable levels) is provided to allow time to fabricate the silicone part. Heat is then applied to activate the platinum, the crosslinking reaction occurs, and the elastomer is vulcanized. The amount of working time and rate of cure are determined by the amount of crosslinker, catalyst, and inhibitors used in the formulation.



NOTE 1—Si=Silicon
Pt=Platinum

FIG. 1 Silylhydride Addition Cure Reaction