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Standard Practice for Accelerated Aging of Ultra-High Molecular Weight Polyethylene after Gamma Irradiation in Air[1]

This standard is issued under the fixed designation F 2003; 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 It is the intent of this practice to permit an investigator to evaluate the oxidative stability of UHMWPE materials as a function of processing and sterilization method. This practice describes a laboratory procedure for accelerated aging of ultra-high molecular weight polyethylene (UHMWPE) specimens and components for total joint prostheses. The UHMWPE is aged at elevated temperature and at elevated oxygen pressure, to accelerate oxidation of the material and thereby allow for the evaluation of its long-term chemical and mechanical stability.

1.2 Although the accelerated-aging method described by this practice will permit an investigator to compare the oxidative stability of different UHMWPE materials, it is recognized that this method may not precisely simulate the degradative mechanisms for an implant during real-time shelf aging and implantation.

1.3 The accelerated aging method specified herein has been validated based on oxidation levels exhibited by certain shelf-aged UHMWPE components packaged in air and sterilized with gamma radiation. The method has not been shown to be representative of shelf aging when the UHMWPE is packaged in an environment other than air. For example, this practice has not been directly correlated with the shelf life of components that have been sealed in a low-oxygen package, such as nitrogen. This practice is not intended to simulate any change that may occur in UHMWPE following implantation.

1.4 *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 883 Terminology Relating to Plastics[2]

F 648 Specification of Ultra-High Molecular-Weight Polyethylene Powder and Fabricated Form Surgical Implants[3]

F 1714 Guide for Gravimetric Wear Assessment of Prosthetic Hip-Designs in Simulator Devices[3]

F 1715 Guide for Gravimetric Wear Assessment of Prosthetic Knee-Designs in Simulator Devices[3]

2.2 *ISO Standards:*

ISO 5834 Implants for surgery—Ultra-high molecular weight polyethylene[4]

ISO 14242 Implants for surgery—Wear of total hip joint prostheses[4]

ISO 14243 Implants for surgery—Wear of total knee joint prostheses[4]

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice relating to plastics, refer to Terminology D 883. For definitions of terms in this practice relating to UHMWPE, refer to Specification F 648 and ISO 5834.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oxidation*, *n*—the incorporation of oxygen into another molecule (for example, UHMWPE) by means of a chemical reaction, resulting in the formation of a chemical covalent bond.

3.2.2 *oxygen bomb*, *n*—a pressure vessel suitable for preconditioning of UHMWPE at an elevated temperature and partial pressure of oxygen.

4. Significance and Use

4.1 This practice summarizes a method that may be used to accelerate the oxidation of UHMWPE components using elevated temperature and elevated oxygen pressure. Under real-time conditions, such as shelf aging and implantation, oxidative changes to UHMWPE after sterilization using high energy radiation may take months or years to produce changes that may result in deleterious mechanical performance. The

[1] This practice 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.

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

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

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

method outlined in this practice permits the evaluation of oxidative stability in a relatively short period of time (for example, weeks).

4.2 This practice may also be used to oxidize UHMWPE test specimens and joint replacement components prior to characterization of their physical, chemical, and mechanical properties. In particular, this practice may be used for accelerated aging of UHMWPE components prior to evaluation in a hip or knee joint wear simulator as outlined in Guide F 1714 (hip wear), Guide F 1715 (knee wear), ISO 14242 (hip wear), or ISO 14243 (knee wear), or combination thereof.

5. Apparatus

5.1 *Combined Apparatus*—An oxygen bomb (pressure vessel) apparatus that is capable of maintaining the desired temperature with an accuracy of ±2°C by itself may be used, providing it incorporates the requirements of 5.2-5.4.

5.2 *Pressure Vessel*—If a combined apparatus is not used, it will be necessary to enclose the specimens within a pressure vessel, also known as an "oxygen bomb," capable of withstanding a static pressure of 690 kPa (100 psi). The pressure vessel shall be manufactured from stainless steel. The pressure vessel shall be equipped with either a regulator or a safety release valve to maintain the internal pressure to the desired value, when at equilibrium, to an accuracy of ±7 kPa (±1 psi).

5.3 Because oxygen-air mixtures will be maintained at elevated temperatures for weeks at a time, it is recommended that a laboratory that is performing aging at elevated pressure take appropriate safety precautions. For this reason, the use of a commercially available and properly validated "oxygen bomb" is recommended.[5] The pressure vessel must be of suitable construction such that it does not leak, thereby leading to the reduction of pressure during the two-week aging period.

NOTE 1—Oxygen flow and test interruption have been shown to significantly influence the outcome of accelerated aging studies. Consequently, the pressure vessel must maintain nearly constant pressure (that is, within ±7 kPa or 1 psi) throughout the duration of the testing period, or the results may not be reproducible or may be unreliable.

5.4 *Thermal Chamber*—If a combined apparatus is not used, accelerated aging of the UHMWPE shall be conducted using a thermal chamber that can maintain the desired temperature with an accuracy of ±2°C. The spatial variation of temperature within the thermal chamber shall be measured using thermocouples and verified to be less than ±1°C. Note that the thermal chamber will need to be sufficiently large to accommodate the pressure vessel, described in 5.2.

5.5 *Temperature Controller*—The combined apparatus or thermal chamber shall be equipped with a temperature controller, capable of controlling the heating rate with an accuracy of 0.1°C/min.[6]

NOTE 2—Temperature stability and test interruption has been shown to significantly influence the outcome of accelerated aging studies. Consequently, the pressure vessel must maintain nearly constant temperature (that is, within ±1°C) throughout the duration of the testing period, or the results may not be reproducible or may be unreliable.

6. Test Specimens

6.1 The test specimens shall be prepared in final form according to the requirements of any subsequent physical, chemical, or mechanical tests to be performed after accelerated aging. For example, if the specimens will ultimately be subjected to hip joint simulation, they should be prepared in final form according to Guide F 1714 and ISO 14242.

6.2 Finished specimens shall not be machined after accelerated aging of (bulk) stock materials, because the accelerated oxidation procedure outlined in this practice will result in an inhomogeneous distribution of chemical, physical, and hence mechanical properties through the thickness of an aged part.

6.3 Test specimens shall be removed from their packaging prior to accelerated aging, because this practice is not intended to reproduce the aging of UHMWPE that is stored in a low oxygen environment.

7. Validation of Apparatus

7.1 *Thermal Chamber Validation*—Using the calibrated temperature sensor, validate the temperature of the accelerated aging apparatus to within ±1°C of the aging temperature.

7.1.1 Verify the calibration of the temperature sensor(s) that will be used to validate the thermal conditions in the accelerating aging apparatus. The temperature sensor shall be calibrated as defined in the manufacturer's instructions.

7.2 *Pressure Vessel Validation*—Verify the integrity of the pressure vessel to within ±7 kPa (±1 psi) by conducting the following 14-day (336 ± 1 h) validation test:

7.2.1 Increase the pressure of pure oxygen inside the vessel by 503 kPa (73 psi) at 70 ± 1°C.

7.2.2 Throughout the duration of the validation test, the gage pressure shall not vary by ±7 kPa (±1 psi).

7.2.3 Pressure vessels that are not capable of maintaining the target gage pressure within the stated tolerance shall be considered invalid for the purposes of accelerated aging until the excessive leaking has been rectified.

7.3 The thermal chamber and pressure vessel shall be validated at least once per year, unless otherwise indicated by a specification or customer.

8. Conditioning

8.1 After high energy irradiation, specimens shall be maintained at 23 ± 2°C (73.4 ± 3.6°F) for 28 days, starting from the date of irradiation, prior to commencing accelerated aging, unless otherwise directed by the customer.

8.2 After irradiation, specimens shall remain in their original packaging during the preconditioning period.

8.3 Unirradiated specimens shall be maintained in a standard laboratory environment of 23 ± 2°C (73.4 ± 3.6°F) for 40 ± 1 h prior to commencing accelerated aging.

9. Procedure

9.1 *Specimen Orientation*—Test specimens shall be arrayed within the test chamber or oxygen bomb such that all relevant surfaces have equivalent access to oxygen during the test. For example, with hip and knee components, the articulating

[5] Pressure vessels available from Advantec MFS, Inc., 6691 Owens Drive, Pleasanton CA, 94588-3335 have been found satisfactory for this purpose.

[6] Air convection ovens available from Cole Parmer Instrument Company, http://www.coleparmer.com have been found satisfactory for this purpose.