Designation: F 2003 – 0~~0~~2

Standard ~~Guide~~ 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 ~~guide~~ practice to permit an investigator to ~~investigate~~ evaluate the oxidative stability of ~~ultra-high molecular weight polyethylene (UHMWPE)~~ UHMWPE materials as a function of processing and sterilization method. This ~~guide~~ practice describes a laboratory ~~test method~~ procedure for accelerated aging of ultra-high molecular weight polyethylene (UHMWPE) specimens and components for total joint prostheses. The UHMWPE is aged at elevated ~~temperatures and, alternatively,~~ temperature and at elevated ~~partial pressures of oxygen,~~ 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 ~~test methods~~ method described by this ~~guide~~ practice will permit an investigator to compare the oxidative stability of different UHMWPE materials, it is recognized that ~~these test methods~~ this method may not precisely simulate the degradative mechanisms for an implant during real-time shelf aging and ~~implantation. However, these accelerated oxidation methods have been successfully used to rank UHMWPE materials for their long-term oxidative stability.~~ implantation.

1.3 The accelerated aging ~~test methods~~ method specified herein ~~have~~ has been validated based on oxidation levels exhibited by certain shelf-aged UHMWPE components packaged in air and sterilized with gamma radiation. The ~~methods have~~ method has not been shown to be representative of shelf aging when the UHMWPE is packaged in an environment other than air. For example, ~~these test methods have~~ 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]

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[1] This ~~guide~~ practice is under the jurisdiction of ASTM Committee ~~F-4~~ F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on ~~Test~~ Material Test Methods.

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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 ~~guide~~ practice relating to plastics, refer to Terminology D 883. For definitions of terms in this ~~guide~~ 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 ~~guide~~ practice summarizes ~~test methods~~ a method that may be used to accelerate the oxidation of UHMWPE components using elevated ~~temperatures and, alternatively,~~ temperature and elevated ~~partial pressures of oxygen.~~ 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 ~~test methods~~ method outlined in this ~~guide permit~~ practice permits the evaluation of oxidative stability in a relatively short period of time (for example, weeks).

4.2 This ~~guide~~ practice may also be used to ~~precondition~~ oxidize UHMWPE test specimens and joint replacement components prior to characterization of their physical, chemical, and mechanical properties. In particular, this ~~guide~~ practice may be used for ~~preconditioning~~ 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 and Materials~~ Apparatus

5.1 ~~*UHMWPE Test Specimens*—The test specimens shall be prepared in final form in accordance with the requirements~~Combined Apparatus—An oxygen bomb (pressure vessel) apparatus that is capable of ~~any subsequent physical, chemical, or mechanical tests to be performed after preconditioning. For example, if~~ maintaining the ~~specimens will ultimately be subjected to hip joint simulation, they should be prepared in final form in accordance~~ desired temperature with ~~Guide F 1714 and ISO 14242. Because the accelerated oxidation methods outlined in this guide result in inhomogeneous distribution~~ an accuracy of ~~chemical, physical, and hence mechanical properties through the thickness of a preconditioned part, it is not recommended that finished test specimens~~ ±2°C by itself may be ~~machined after preconditioning of (bulk) stock materials. Because this guide is not intended to reproduce~~ used, providing it incorporates the ~~aging~~ requirements of ~~UHMWPE that is stored in a low-oxygen environment, test specimens should be removed from their packaging prior to preconditioning.~~ 5.2-5.4.

5.2 ~~*Preconditioning Chamber*—Accelerated oxidation (preconditioning) of~~Pressure Vessel— If a combined apparatus is not used, it will be necessary to enclose the ~~UHMWPE~~ 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 ~~conducted in~~ manufactured from stainless steel. The pressure vessel shall be equipped with either a ~~convection, air circulating oven that can~~ regulator or a safety release valve to maintain the internal pressure to the desired ~~temperature with~~ value, when at equilibrium, to an accuracy of ~~±2°C. The spatial variation of temperature within the oven shall be measured using thermocouples and verified to be less than ±1°C. The chamber~~ ±7 kPa (±1 psi).

5.3 Because oxygen-air mixtures will ~~need to~~ be ~~sufficiently large to accommodate~~ maintained at elevated temperatures for weeks at a ~~pressure vessel, if~~ time, it is ~~desired to precondition the UHMWPE~~ recommended that a laboratory that is performing aging at ~~an~~ elevated ~~partial~~ pressure take appropriate safety precautions. For this reason, the use of ~~oxygen. An oxygen bomb (pressure vessel) that~~ a commercially available and properly validated "oxygen bomb" is ~~capable~~ recommended.[5] The pressure vessel must be of ~~m~~ suitable construction such that it does not leak, thereby leading to the ~~desired temperature with an accuracy~~ reduction of ~~±2°C by itself may be used.~~ pressure during the two-week aging period.

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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), ~~11~~ 25 W. ~~42nd~~ 43rd St., ~~13th~~ 4th Floor, New York, NY 10036.
~~[5] The boldface numbers in parentheses refer to the list of references at the end of~~
[5] Pressure vessels available from Advantec MFS, Inc., 6691 Owens Drive, Pleasanton CA, 94588-3335 have been found satisfactory for this ~~standard.~~ purpose.

NOTE 1—It may be desirable 1—Oxygen flow and test interruption have been shown to use an oven with significantly influence the capability outcome of h accelerated aging studies. Consequently, the test specimen at a controlled heating rate with an accuracy of ±0.1°C/min.

5.3 *Pressure Vessel*— When preconditioning UHMWPE at elevated partial pressures of oxygen, it will be necessary to enclose the specimens within a pressure vessel, also known as an oxygen bomb, capable of withstanding a static vessel must maintain nearly constant pressure of 690 (that is, within ±7 kPa (100 psi). The pressure vessel shall or 1 psi) throughout the duration of the testing period, or the results may not be manufactured from stainless steel reproducible or aluminum. The pressure vessel may be unreliable.

5.4 *Thermal Chamber*— If a combined apparatus is not used, accelerated aging of the UHMWPE shall be equipped with either conducted using a regulator or a safety release valve to thermal chamber that can maintain the internal pressure to the desired value, when at equilibrium, to temperature with an accuracy of ±7 kPa (±1 psi). Because oxygen-air mixtures will ±2°C. The spatial variation of temperature within the thermal chamber shall be maintained at elevated temperatures for weeks at a time, it is recommended measured using thermocouples and verified to be less than ±1°C. Note that appropriate safety precautions the thermal chamber will need to be taken by a laboratory performing preconditioning at elevated partial pressures. For this reason, sufficiently large to accommodate the use of a commercially available and properly validated oxygen bomb is recommended.

6. Preconditioning Test Methods pressure vessel, described in 5.2.

5.5 *Test Method A (Ambient Air Preconditioning)*—Conduct Test Method A in a suitableTemperature Controller—The combined apparatus or thermal chamber. Precondition test specimens at chamber shall be equipped with a constant temperature controller, capable of 80°C for 3 weeks prior to subsequent testing. controlling the heating rate with an accuracy of 0.1°C/min.[6]

NOTE 2—To maximize the extent 2—Temperature stability and penetration of oxidative degradation into bulk hip or knee components using Test Method A, it is recommended that specimens be inserted into the thermal chamber while at room temperature, and that the chamber be elevated test interruption has been shown to significantly influence the constant preconditioning temperature at a slow constant heating rate, ranging from 0.1 to 0.6°C/min **(1)**.[5]

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

6.2 *Test Method B (Preconditioning at Elevated Partial Pressures outcome of Oxygen)*—Conduct Test Method B using an oxygen bomb placed inside a suitable thermal chamber. Precondition test specimens at a accelerated aging studies. Consequently, the pressure vessel must maintain nearly constant temperature (that is, within ±1°C) throughout the duration of 70°C and at an equilibrium pressure of 503 kPa (73 psi, 5 atmospheres) of pure oxygen for 2 weeks prior to subsequent testing.

6.3 Regardless of the preconditioning test method used (Test Method A testing period, or B), array the results may not be reproducible or may be unreliable.

6. Test Specimens

6.1 The test specimens within shall be prepared in final form according to the test chamber requirements of any subsequent physical, chemical, or oxygen bomb such that all relevant surfaces have equivalent access mechanical tests to oxygen during the test. be performed after accelerated aging. For example, with hip and knee components, if the articulating surface which may subsequently specimens will ultimately be subjected to wear simulation 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 obstructed or covered by other parts or materials that might interfere with uniform access machined after accelerated aging of (bulk) stock materials, because the surface to oxygen.

6.4 For both Test Methods A accelerated oxidation procedure outlined in this practice will result in an inhomogeneous distribution of chemical, physical, and B, maintain hence mechanical properties through the test chamber or oxygen bomb, or both, at ambient humidity. The user should thickness of an aged part.

6.3 Test specimens shall be aware that adding water removed from their packaging prior to accelerated aging, because this practice is not intended to reproduce the test chamber may affect the oxidation mechanism during the preconditioning process. aging of UHMWPE that is stored in a low oxygen environment.

7. ReportValidation of Specimen Preparation and Test Conditions

7.1 It is important that details regarding Apparatus

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

7.1.1 Verify the calibration of the preconditioning, temperature sensor(s) that will be used to validate the storage thermal conditions for in the test samples, and the test method used accelerating aging apparatus. The temperature sensor shall be recorded calibrated as defined in the manufacturepor'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 surface which may subsequently be subjected to wear simulation shall not be obstructed or covered by other parts or materials that might interfere with uniform access of the surface to oxygen.

9.2 *Pressurization*—The pressure vessel shall be filled at room temperature and purged with oxygen at least three times prior to starting the aging experiment. For example, at a standard laboratory environment, a change in pressure of 62.5 ± 1 psi will be needed, such that the pressure increases to 503 kPa (73 psi) as the at the target aging temperature of 70°C is reached.

9.3 *Standard Relative Humidity*—Water shall not be added to the pressure vessel during accelerated aging. The user should be aware that adding water to the test chamber may affect the oxidation mechanism during the accelerated aging process.

9.4 *Initial Temperature and Heating Rate*—The pressure vessel is initially at standard laboratory temperature (23 ± 2°C) and will be gradually raised to the aging temperature of 70°C. The initial heating rate will be 1.0 ± 0.1°C/min.

9.5 *Accelerated Aging*—Specimens are to be aged at a constant temperature of 70° C and at an equilibrium gage pressure of 503 kPa (73 psi, 5 atmospheres) of pure oxygen for 336 ± 1 h (14 days) prior to subsequent testing. There are to be no interruptions of the aging period (that is, no opening of the pressure vessel).

9.6 *Recording During the Test*—Temperature and pressure recordings should be logged daily during the test period to note any potential changes of the experimental conditions.

9.7 *Guide for Subsequent Testing*—Specimens shall be subjected to further testing within two weeks after accelerated aging.

10. Reporting of Specimen Preparation and Test Conditions

10.1 The written report shall include details regarding the preparation of the test samples, the chronology of the accelerated aging, and the storage conditions for the test samples.

10.2 *Test Sample Preparation*—~~Report~~—The investigator shall list the size, shape, and method of manufacture of the test samples. ~~R~~ The report shall also contain the type of resin used, the manufacturer/supplier of the UHMWPE, and any subsequent processes that were performed on the test articles after manufacture, such as sterilization or high- energy irradiation.

~~7~~10.3 *Chronology*—~~R~~The report shall list the time at which the test specimens were manufactured, subsequently sterilized, and later ~~preconditioned. Report~~ aged. The report will also report the time that any subsequent analysis or testing was performed on the ~~preconditioning~~ aged items.

~~7~~10.4 *Test Sample Storage Conditions* —It is important to document the storage conditions of the test samples before and after ~~preconditioning. Report~~ accelerated aging. The report shall indicate the environmental conditions (that is, storage in air versus nitrogen) and temperature under which the specimens were stored.

~~7.5 *Test Method*—Report the preconditioning test method that was used (A or B), the heating rate, the test temperature, and the test duration.~~

~~**8.**~~

11. Keywords

~~8~~11.1 aging; oxidation; preconditioning; stability; UHMWPE; UHMW PE; ultra-high molecular weight polyethylene

APPENDIX

(Nonmandatory Information)

X1. Rationale

X1.1 Post-irradiation aging results in degradative changes to the physical, chemical, and mechanical properties of UHMWPE. **(2, 3)**~~.~~ Even under ambient conditions, oxidation of irradiated UHMWPE evolves at a slow pace, with a degradation rate measured in years. **(2)**~~.~~ As a result, ~~preconditioning~~ accelerated aging test methods have been developed in the past four years to accelerate the oxidation process in UHMWPE and thereby assess oxidative stability during a comparatively short time period.

X1.2 Oxidation of UHMWPE proceeds in a complex cascade of chemical reactions, which may be accelerated by increasing the temperature or by increasing the concentration of available oxygen, or both. **(4)**~~.~~ Consequently, in several studies, post-irradiation aging has been simulated using a combination of thermal ~~preconditioning~~ oxidation and elevated oxygen ~~partial pressures~~ pressure. **(1, 5, 6)**~~.~~ Despite the variation in test conditions reported by these studies, accelerated oxidation protocols have increasingly been employed not only to characterize the effects of gamma sterilization in air, but also to evaluate the oxidation resistance of UHMWPE sterilized by alternative methods.

X1.3 Accelerated oxidation test methods for UHMWPE are not without their limitations. Even though the ~~protocols~~ method outlined in this ~~guide are~~ practice is now widely used for ~~preconditioning~~ accelerated aging UHMWPE specimens prior to mechanical testing, the question remains as to whether or not the thermal technique~~s~~ precisely recreate~~s~~ the morphology and mechanical properties of shelf-aged UHMWPE. **(7, 8)**~~.~~ Although research is still needed to elucidate the differences between thermal conditioning and long-term shelf-aging, this ~~guide~~ practice is intended to provide ~~information about established test methods~~ a specific procedure for evaluating the oxidative stability of UHMWPE specimens.

X1.4 Round robin studies based on an earlier version of this practice, published by ASTM in 2000, revealed interlaboratory variability in excess of 100 % for certain groups of aged test specimens. **(9)** Consequently, revision of this practice was initiated in 2001 in response to available data **(9, 10)** highlighting procedural details which may influence the outcome of accelerated aging studies.

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