Designation: F3336 – 22

Standard Practice for Lipid Preconditioning of Ultra-High-Molecular-Weight Polyethylene for Accelerated Aging[1]

This standard is issued under the fixed designation F3336; 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 incorporate lipids found in the synovial environment into polymeric specimens. This can be used as a preconditioning step to evaluate the oxidative stability of ultra-high-molecular-weight polyethylene (UHMWPE) materials. This practice describes a laboratory procedure for preconditioning of UHMWPE specimens.

1.2 The preconditioned UHMWPE can be aged at elevated temperature and at elevated oxygen pressure following methods of accelerated aging described in Practice F2003, to accelerate oxidation of the material and thereby allow for the evaluation of its long-term chemical stability.

1.3 The preconditioned UHMWPE can be tested without further aging using a method to evaluate oxidative stability such as oxidation induction time as described in Test Method D3895.

1.4 The methods of this practice may be used on any type of UHMWPE material intended for use in total joint arthroplasty in a synovial joint (for example, conventional, cross-linked, antioxidant stabilized, etc.). See Appendix X1.

1.5 Although the preconditioning method followed by accelerated aging described by this practice will permit an investigator to compare the oxidative stability of different UHMWPE materials, it is recognized that this method is not known to simulate the degradative mechanisms for an implant during real-time shelf aging or *in vivo*. The described methods have not been evaluated for mechanical testing under cyclic loading.

1.6 The preconditioning and accelerated aging methods specified herein are intended to rank the resistance to oxidation of materials as a result of the absorption of lipids, which may occur in UHMWPE following implantation, and to determine susceptibility to oxidative changes. The methods have not been evaluated for use in preconditioning of UHMWPE components for subsequent testing of mechanical or wear properties. Procedure A should not be used for preconditioning of UHMWPE components for subsequent testing of mechanical or wear properties.

1.7 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 for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *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]

D883 Terminology Relating to Plastics

D3895 Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry

F648 Specification for Ultra-High-Molecular-Weight Polyethylene Powder and Fabricated Form for Surgical Implants

F2003 Practice for Accelerated Aging of Ultra-High Molecular Weight Polyethylene after Gamma Irradiation in Air

F2102 Guide for Evaluating the Extent of Oxidation in

[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] 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.

Polyethylene Fabricated Forms Intended for Surgical Implants

2.2 *ISO Standards:*[3]

ISO 5834 Implants for Surgery—Ultra-High Molecular Weight Polyethylene

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice relating to plastics, refer to Terminology D883. For definitions of terms in this practice relating to UHMWPE, refer to Specification F648 and ISO 5834.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *lipids, n*—lipophilic compounds which have been shown to absorb into UHMWPE from the synovial fluid (**1**).[4]

3.2.2 *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.

3.2.3 *oxidative stability, n*—the resistance to oxidation in a material. A specific material can be described to have more oxidative stability if the change in its oxidation under described conditions is less than that of another material under the same conditions.

4. Significance and Use

4.1 This practice summarizes two methods that may be used to precondition UHMWPE by the absorption of lipids to differentiate the simulated *in vitro* oxidative stability of UHMWPEs, after lipid exposure.

4.1.1 *Procedure A, High Squalene Absorption*—This method of preconditioning with lipids may be used for comparative oxidative stability testing to screen different materials under aggressive conditions.

4.1.2 *Procedure B, Mixed Lipid Absorption*—This method of preconditioning may be used for comparative oxidative stability testing under mild conditions that more closely simulate *in-vivo* conditions.

4.2 This practice may be used to accelerate the oxidation of UHMWPE components when using elevated temperature and elevated oxygen pressure according to the methods of Practice F2003. Under real-time conditions such as implantation, oxidative changes to UHMWPE formulations may take months or years to produce changes that may result in deleterious mechanical performance. The method outlined in this practice permits the preparation of UHMWPE for evaluation of oxidative stability in a relatively short period of time (for example, weeks).

4.3 This practice may also be used to precondition UHMWPE test specimens prior to characterization of their physical and chemical properties. In particular, this practice may be used for preconditioning with lipids prior to oxidation induction time (OIT) testing as outlined in Test Method D3895.

5. Apparatus

5.1 *Absorption Vessel*—A glass container such as a beaker or an Erlenmeyer flask that can contain the desired volume of the absorption medium and that can be stirred and heated safely up to 120 °C, with an accuracy of 2 °C.

5.2 *Heating and Stirring Apparatus*—A hotplate equipped with a stirring function. For the high absorption procedure performed at 120 °C, it will be necessary to stir the medium during absorption to maintain homogeneity of the sample surface exposure. An oil bath shall be placed on the heating and stirring apparatus to create a heating environment which can maintain the temperature of the absorption medium at 120 °C, with an accuracy of ±1 °C. Alternative heating methods may be used so long as the required accuracy is obtained.

NOTE 1—Because elevated temperatures can cause lipid contamination of the surfaces of the aging vessels, it is recommended that an apparatus be dedicated to the aging of lipid-containing specimens.

6. Test Specimens

6.1 Specimens shall be first machined and preconditioned in this form. Cubes (1 cm × 1 cm × 1 cm) are recommended.

6.2 Following preconditioning, physical or chemical characterization tests such as the measurement of cross-link density may be performed on smaller samples cut from the original (preconditioned) sample.

7. Conditioning

7.1 If samples to be used have been previously processed by radiation, then samples shall be removed from their inert/vacuum packaging immediately prior to the procedure.

7.2 After completing the preconditioning procedure, specimens shall be maintained at 23 ± 2 °C (73.4 ± 3.6 °F) for not more than seven days, starting from the end date of incorporation, before performing further testing or accelerated aging.

8. Procedure

8.1 *Specimen Orientation*—Test specimens shall be arrayed within the absorption vessel such that all relevant surfaces have equivalent access to the medium containing the lipids during the test, preferably by stirring.

8.2 *Procedure A, High Squalene Absorption*—For preconditioning with lipids for comparative oxidative stability testing under aggressive conditions, cubes of test material (1 cm × 1 cm × 1 cm) shall be immersed in preheated squalene (see Annex A1) at 120 °C for 2 h. After 2 h, remove the samples, allow them to cool to room temperature, and wipe them clean with a dry, lint-free wipe prior to storage or further testing.

8.3 *Procedure B, Mixed Lipid Emulsion*—For preconditioning with lipids for comparative oxidative stability testing under mild conditions, cubes of test material (1 cm × 1 cm × 1 cm) shall be immersed in a mixed lipid emulsion (see Annex A2) at 40 °C for three weeks or until 1.0 ± 0.1 mg of weight absorption is measured, whichever occurs first. Measure the absorbed mass once per week. Wipe the samples clean with a dry, lint-free wipe prior to weighing. Replace with fresh emulsion when replacing the samples after weighing.

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

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

8.4 *Stability of Lipid Absorption Media*—The lipids used in the procedures are often sensitive to environmental changes. For Procedure A, squalene shall not be reused. It shall be removed from the manufacturer or supplier's container immediately before the experiment and preheated to 120 °C as quickly as possible, preferably within 2 h. The mixed lipid emulsions can be prepared and kept at room temperature for up to seven days prior to the procedure.

8.5 *Homogeneous Sample-to-Volume Ratios*—It is recommended that the absorption vessel shall not have too many samples per volume of medium to prevent inhomogeneity of sample exposure to the medium. It is recommended that up to twelve cubes be used in 150 mL of medium.

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

8.7 *Guide for Subsequent Testing*—Specimens shall be subjected to further testing within two weeks after preconditioning. Samples shall be stored at 4 °C in the time between preparation and subsequent testing.

8.8 *Measurement of Oxidation Levels and Reporting of Oxidative Stability*—Fourier transform infrared spectroscopy methods described in Guide F2102 may be used to calculate oxidation index in the samples. Extraction of extractable lipids by using hexane or heptane as described in Guide F2102 shall be performed to isolate oxidation of the polymer. Change in oxidation index shall be reported, which may be calculated as surface, bulk, and maximum oxidation index, as described in Guide F2102.

9. Reporting of Specimen Preparation and Test Conditions

9.1 The written report shall include details regarding the preparation of the test samples, the preparation of the absorption media, the lipid absorption procedures, and the storage conditions for the test samples.

9.2 *Test Sample Preparation*—The investigator shall list the method of manufacture of the test samples. 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). The report shall also contain the type of lipids used in the absorption medium, the manufacturer/supplier information, and catalog numbers for the lipids and emulsifiers.

9.3 *Chronology*—The report shall list the times at which the test specimens were manufactured, subsequently processed, preconditioned and, if applicable, later aged. The report shall also report the time that any subsequent analysis or testing was performed on the preconditioned items.

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

10. Keywords

10.1 aging; oxidation; preconditioning; stability; UHMWPE; ultra-high molecular weight polyethylene

ANNEXES

(Mandatory Information)

A1. SQUALENE IMMERSION

A1.1 *Source*—The following lipids should be used: squalene (Sigma Aldrich, S3626).

A1.2 The samples shall be immersed in squalene as received (≥98 %, determined by gas chromatography) and the squalene shall not be diluted.