Designation: F 1634 – 95 (Reapproved 2000)

Standard Practice for In-Vitro Environmental Conditioning of Polymer Matrix Composite Materials and Implant Devices[1]

This standard is issued under the fixed designation F 1634; 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 practice covers two procedures for conditioning non-absorbable polymer matrix composite (PMC) materials and implant devices in a liquid environment to obtain a state of saturation.

1.2 The purpose of this practice is to standardize methods and reporting procedures for conditioning PMC materials and implant devices (PMC specimens) in a user selected liquid environment prior to conducting subsequent tests. It is not the purpose of this practice to determine the diffusion coefficients or actual saturation levels of a given liquid into the materials and devices. For these determinations, other procedures, such as Test Method D 5229, may be followed.

1.3 Diffusion of liquid into a solid material is a slow process. While the time necessary to achieve saturation at 37°C may be sufficiently short for thin specimens, such as fracture fixation plates, it may be prohibitively long in thick sections, such as femoral components for hip arthroplasty. However, the diffusion process may be accelerated at an elevated temperature. Consequently, two separate procedures (Procedures A and B) are presented in this practice. Procedure A covers exposing the specimen to the desired conditioning environment at 37°C. Procedure B prescribes a method to accelerate the diffusion process by conditioning the specimen at a selected elevated temperature.

1.4 This practice does not specify the test environment to be used for conditioning. However, the pH value of immersion liquid shall be maintained at 7.4 ± 0.2 to simulate the in vivo environment.

1.5 *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 618 Practice for Conditioning Plastics and Electrical Insulation Materials for Testing[2]

D 756 Practice for Determination of Weight and Shape Changes of Plastics Under Accelerated Service Conditions[2]

D 3878 Terminology of High-Modulus Reinforcing Fibers and Their Composites[3]

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *cumulative moisture content, M_t (%), n*—the amount of absorbed moisture in a material at a given time *t*, expressed as a percentage of the weight of absorbed moisture divided by the initial specimen weight, as follows:

$$M_t, \% = \frac{W_i - W_b}{W_b} \times 100 \quad (1)$$

where:

W_t = current specimen weight, g, and

W_b = initial (baseline) specimen weight at $t = 0$ and standard laboratory atmosphere, g.

3.1.2 *liquid, n*—water, saline solution, calf serum, or any other liquid solution that is used to condition PMC specimens.

3.1.3 *nominal saturated moisture content, M_s(%)*—an approximation of the amount of moisture absorbed by a specimen at saturation, expressed as a percentage of the weight of absorbed moisture at approximate saturation divided by the initial specimen weight, as follows:

$$M_s, \% = \frac{W_s - W_b}{W_b} \times 100 \quad (2)$$

where:

W_s = specimen weight at approximate saturation, g, and

W_b = initial (baseline) specimen weight at $t = 0$ and standard laboratory atmosphere, g.

3.1.4 *standard laboratory atmosphere, n*— a laboratory atmosphere having a temperature of 23 ± 2°C and a relative humidity of 50 ± 10 %.

[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 15.03.

4. Summary of Test Method

4.1 In Procedure A, a specimen is immersed in a liquid bath at 37 ± 1°C with a pH value of 7.4 ± 0.2.

4.2 In Procedure B, conditioning occurs in a liquid bath at a selected elevated temperature.

4.3 Weight change is monitored over time until specimens reach the nominal moisture saturation content.

4.4 Keep specimens in the conditioning bath for storage prior to subsequent tests.

5. Significance and Use

5.1 The conditioning procedures covered in this practice provide methods for saturating PMC specimens in a liquid environment prior to conducting other tests for property evaluation.

5.2 The conditioning may affect geometric and dimensional changes in specimens. In some severe cases, chemical changes may occur in the fiber, polymer and fiber-polymer interphase and interface.

5.3 Caution must be taken if Procedure B (10.2, Procedure B—Accelerated Moisture Saturation at Elevated Temperature) is followed to condition PMC specimens at an elevated temperature. Physical and chemical reactions in materials are normally temperature dependent. An increase in experimental temperature may accelerate a desirable moisture diffusion process. However, elevated temperatures above 37°C may also cause undesirable reactions that do not represent appropriate responses of materials at 37°C. Consequently, a pilot study is recommended in Procedure B to determine if a selected elevated temperature can be used for accelerated conditioning. If the properties of materials are determined to be irreversibly affected by this pilot study at the selected elevated temperature, then either an appropriate lower elevated temperature should be determined by repeating the pilot study, or Procedure B should not be used.

6. Apparatus

6.1 *Balance*—An analytical balance capable of measuring weight of specimens to within a resolution of at least 0.005 % of the total specimen weight.

6.2 *Conditioning Bath*—A temperature-controlled liquid bath shall be capable of maintaining the required temperature to within ±1°C. The bath shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

6.3 *Specimen Bag*— A sealable, flexible, moisture-proof bag made of material suitable for exposure to specimens that have been removed from the conditioning bath for cooling prior to weighing. Bags that meet the requirement of MIL-B-131 have been found to be satisfactory for use in such applications.

6.4 *Absorbent Cloth*— Clean, non-linting absorbent cloth for use in wiping excess liquid from surface of specimens.

6.5 *Gloves*—Clean, non-linting gloves for use when handling specimens.

6.6 *pH Measurement System*—An analytical system capable of measuring pH to within ±0.1.

6.7 *Differential Scanning Calorimeter*—An analytical system capable of heating a specimen at a controlled rate while measuring heat input and temperature.

7. Sampling and Test Specimens

7.1 *Preparation*— Precaution shall be taken to avoid the entrapment of moisture in uneven surfaces, or delamination due to inappropriate machining and manufacturing processes.

7.2 *Labeling*—Label the specimen so as to be distinct from each other in a manner that will both be unaffected by the test and not influence the test and, furthermore, will not be removed during conditioning.

8. Measurements of Test Specimens

8.1 The following measurements shall be made on specimens prior to immersion, after conditioning at the end of a test procedure, and at any intermediate stage as prescribed in the test procedures:

8.1.1 *Weight*—The weight within 0.005 % of specimen weight.

8.1.2 Characteristic dimensions of specimens may be measured as a function of immersion time to determine the amount of swelling induced by moisture absorption.

9. Visual Examination

9.1 Noticeable qualitative changes in surfaces, outline, and general appearance of the test specimen shall be recorded after each stage of the testing procedure. These changes include color, surface irregularities, odor, surface voids, delamination and cracking. The immersion liquid should also be observed for evidence of material that has leached from specimens or holders, and evidence of bacterial or fungal contamination. If bacterial or fungal contamination is found, specimens should be removed from the solution, washed with detergent and water, rinsed, and placed in fresh solution. If contamination is a recurring problem, antibacterial or antifungal agents must be added to the solution; minimal amounts should be used as they may affect specimen properties.

10. Procedures

10.1 *Procedure A—Moisture Saturation Determination at 37°C*:

10.1.1 *Specimen Preconditioning*—Bring the test specimens to a uniform 23 ± 2°C after manufacturing process.

10.1.2 *Moisture Absorption*:

10.1.2.1 Record the initial (baseline) weight, W_b.

10.1.2.2 Place the specimen in the conditioning bath, which has previously reached the specified temperature 37 ± 1°C. The pH value of immersion liquid used shall be maintained at 7.4 ± 0.2 throughout the conditioning process and monitored at least once a week. If the solution pH falls outside the designated range, the solution should be changed. The pH should not be maintained by repeatedly adding buffer to the same solution. This will change solution composition and may affect specimen properties. Evaporation losses should be made up with sterile deionized water if saline, serum, plasma, or other hydrous medium is used as the conditioning environment.