Designation: F 1926 – 99

Standard Test Method for Evaluation of the Environmental Stability of Calcium Phosphate Coatings[1]

This standard is issued under the fixed designation F 1926; 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 test method covers calcium phosphate coatings intended for use in surgical implant applications.

1.2 Aspects of the biological response to calcium phosphate materials in soft tissue and bone have been reported from laboratory studies and clinical use **(1-10)**.[2]

1.3 The requirements of this specification apply to calcium phosphate coatings such as calcium hydroxyapatite (see Specification F 1185), beta-tricalcium phosphate (see Specification F 1088), and biphasic mixtures thereof with or without intentional addition of other minor components (< 10 %).

1.4 The coating(s) shall be representative of that produced for sale. It shall have been produced and processed under standard manufacturing conditions.

1.5 The coatings may be applied to porous, nonporous, textured, and other implantable topographical substrate forms representative of the end-use product.

1.6 The calcium phosphate coating may constitute the only coating on a substrate or be one of a multiple coated device.

1.7 This test method is limited to the laboratory evaluation of the dissolution rate of a calcium phosphate coatings. No correlation of the results to in vivo performance is implied.

1.8 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.9 *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:*

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

F 1088 Specification for Beta Tricalcium Phosphate for Surgical Implantation[4]

F 1185 Specification for Ceramic Hydroxylapatite for Surgical Implantation[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *calcium phosphate*—any one of a number of inorganic chemical compounds containing calcium and phosphate ions as its principal constituents.

3.1.2 *coating*—a layer of material mechanically or chemically adhering to the surface of a substrate.

4. Dissolution Media

4.1 Any water used for preparing reagents or dissolution media shall be deionized or distilled and have less then 0.1 ppm each of residual Ca^{++}, phosphorus, and total solids.

4.2 *Unbuffered Water Media*—Deionized or distilled water containing 8×10^{-5} M NaCl, 8×10^{-5} M $CaCl_2$, and 5×10^{-5} M $K_3(PO_4)$.

4.3 *pH 5.5 MES Buffer Media*—1.0 M MES, [2-(N-morphplino)ethanesulfonic acid] having a pH of 5.5 at 37 °C ± 0.5 °C and containing 8×10^{-5} M NaCl, 8×10^{-5} M $CaCl_2$, and 5×10^{-5} M $K_3(PO_4)$.

4.3.1 A buffer concentration of 1.0 M will usually provide sufficient buffer capacity to keep the solution within ± 0.1 pH units of the initial value. If this is not the case, the buffer capacity should be adjusted accordingly.

4.3.2 The pH must be adjusted to 5.5 at 37 ± 0.5° C.

4.4 *pH 7.4 TRIS Buffer Media*—1.0 M TRIS, [Tris(hydroxymethyl)aminomethane] having a pH of 7.4 at 37 °C ± 0.5 °C and containing 8×10^{-5} M NaCl, 8×10^{-5} M $CaCl_2$, and 5×10^{-5} M $K_3(PO_4)$.

4.4.1 A buffer concentration of 1.0 M will usually provide sufficient buffer capacity to keep the solution within ± 0.1 pH

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials .

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[2] The boldface numbers given in parentheses refer to a list of references at the end of the text.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

units of the initial value. If this is not the case, the buffer capacity should be adjusted accordingly.

4.4.2 The pH must be adjusted to 7.4 at 37 ± 0.5° C.

5. Analytical Parameters

5.1 The following procedure should be performed with each of the media listed:

5.1.1 The dissolution rate shall be measured under the conditions of a constant ratio of initial coating mass (mg) to total dissolution media volume (mL), and initial pH. The milligrams of coating to millilitres of dissolution media ratio shall typically be between 1.0 and 0.1.

5.1.2 The pH of the dissolution media (±0.01 pH units), dissolved Ca++ concentration (± 1 ppm), and dissolved phosphorus (as P) concentration (± 1 ppm) shall be measured as soon as practical after the start of the experiment and at appropriate time intervals thereafter to allow the determination of their changes with time.

6. Analytical Procedures

6.1 Make pH measurements with an appropriately calibrated pH meter and probe.

6.2 Measure Ca++ concentrations potentiometrically, colorimetrically, by atomic absorption (AA) or inductively coupled plasma (ICP) spectroscopy, or by inductively coupled plasma mass spectroscopy (ICP/MS).

6.3 Total phosphorus concentrations shall be measured by an appropriate method (for example, inductively coupled plasma (ICP) spectroscopy, inductively coupled plasma mass spectroscopy (ICP/MS), and phosphomolybdate complex method) **(11)**.

6.4 It is recommended that an appropriate bacteriostat (for example, 0.1 v/v % Hibiclens or 0.1 w/v % sodium azide) be added to the dissolution media prior to the start of an experiment.

7. Dissolution Apparatus

7.1 The dissolution vessel (see Fig. 1) shall be of such design to easily accommodate the test specimen (see Fig. 2) and stirrer assembly (see Fig. 3), the specific ion-electrode assembly, and the pH electrode assembly. It shall also be capable of being isolated from the atmosphere by an oxygen and carbon dioxide free inert gas purge.

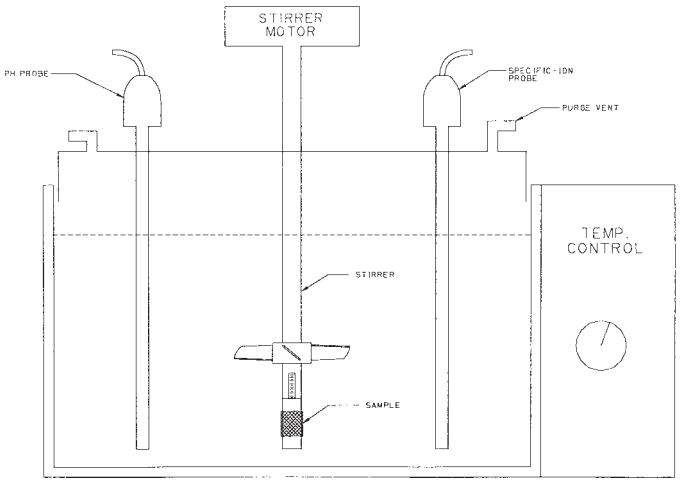

FIG. 1 Dissolution Apparatus

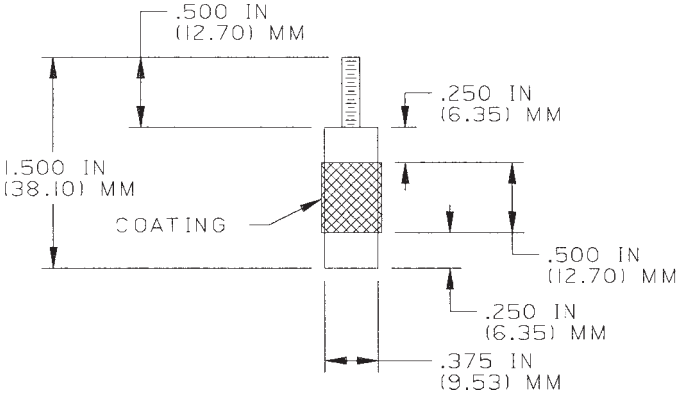

FIG. 2 Test Specimen and Coating

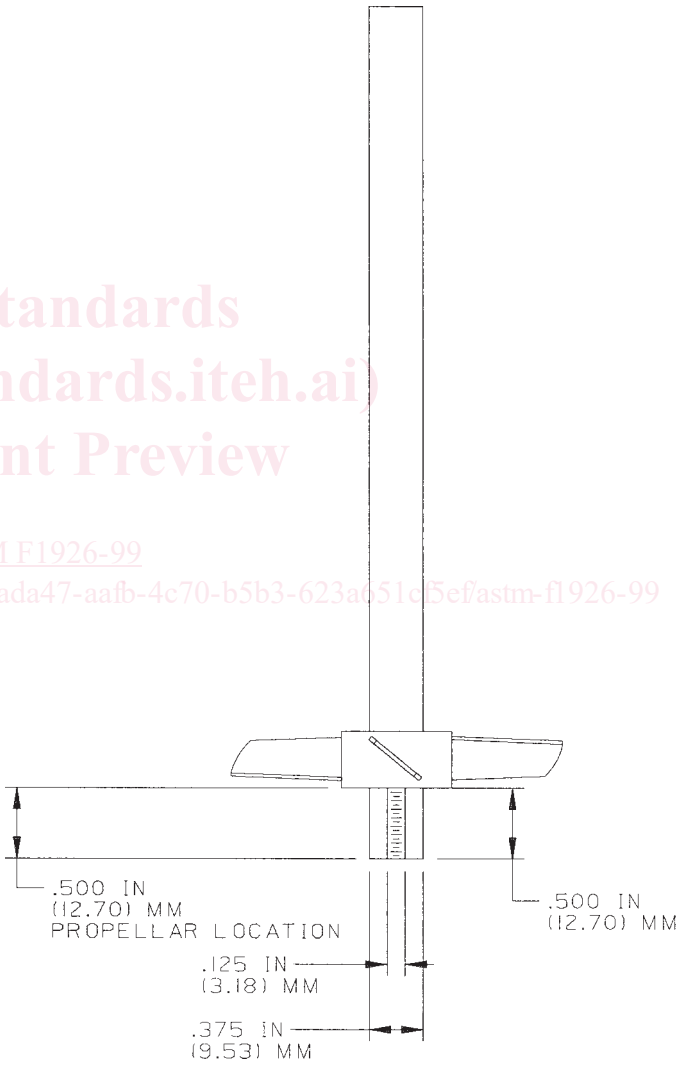

FIG. 3 Stirrer Shaft Assembly

7.2 It shall be of appropriate dimensions to contain the required volume of dissolution media at a level to facilitate sufficient stirring action from the stirrer blades.

7.3 The stirrer assembly shall consist of a stirrer motor capable of maintaining a constant stirring rate of at least 80