Designation: F 1609 – 95

Standard Specification for Calcium Phosphate Coatings for Implantable Materials[1]

This standard is issued under the fixed designation F 1609; 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 specification covers the material requirements for calcium phosphate coatings for surgical implant applications.

1.2 In particulate and monolithic form, the calcium phosphate materials system has been well-characterized regarding biological response **(1, 2)**[2] and laboratory characterization **(2–4)**. Several publications **(5–10)** have documented the in vitro and in vivo properties of selected calcium phosphate coating systems.

1.3 This specification includes hydroxylapatite coatings, tricalcium phosphate coatings, or combinations thereof, with or without intentional minor additions (10 % or less, as opposed to trace elements) of other ceramic or metallics, and applied by methods including, but not limited to, the following: (*1*) mechanical capture, (*2*) plasma spray deposition, (*3*) dipping/sintering, (*4*) electrophoretic deposition, (*5*) porcelainizing, and (*6*) sputtering.

1.4 Substrates may include smooth, porous, textured, and other implantable topographical forms.

1.5 This specification excludes organic coatings that may contain calcium and phosphate ionic species.

2. Referenced Documents

2.1 *ASTM Standards:*

C 313 Test Method for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal[3]

C 501 Test Method for Relative Resistance to Wear of Unglazed Ceramic Tile by the Taber Abraser[4]

C 633 Test Method for Adhesion or Cohesive Strength of Flame-Sprayed Coatings[5]

C 674 Test Methods for Flexural Properties of Ceramic Whiteware Materials[4]

C 949 Test Method for Porosity in Vitreous Whitewares by Dye Penetration[4]

E 376 Practice for Measuring Coating Thickness by Magnetic-Field or Eddy-Current (Electromagnetic) Test Methods[6]

F 1044 Test Method for Shear Testing of Porous Metal Coatings[7]

F 1088 Specification for Beta-Tricalcium Phosphate for Surgical Implantation[7]

F 1147 Test Method for Tension Testing of Porous Metal Coatings[7]

F 1185 Specification for Composition of Ceramic Hydroxylapatite for Surgical Implants[7]

2.2 *Pharmacopeia Convention Documents:*[8]

National Formulary XVI, Tribasic Calcium Phosphate

United States Pharmacopeia:

U.S. Pharmacopeia XXI, Chemical Tests CaP (191), Lead <251>, Mercury <261>, Arsenic< 211>, and Heavy Metals <231> Method (1)

2.3 *Other Documents:*

U.S. Geological Survey Method, Cadmium[9]

21 CFR 820[10]

X-Ray Diffraction Analyses[11]

3. Terminology

3.1 *Definitions:*

3.1.1 *beta tricalcium phosphate*—a calcium phosphate substance of empirical chemical formula, $Ca_3(PO_4)_2$ (see Specification F 1088).

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

3.1.3 *coating*—a layer of mechanically or chemically attached material covering a substrate material.

[1] This specification 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.

Current edition approved Aug. 15, 1995. Published September 1995.

[2] The boldface numbers in parentheses refer to the list of references at the end of this specification.

[3] *Discontinued* 1991—See 1990 *Annual Book of ASTM Standards,* Vol 15.02.

[4] *Annual Book of ASTM Standards,* Vol 15.02.

[5] *Annual Book of ASTM Standards,* Vol 02.05.

[6] *Annual Book of ASTM Standards,* Vol 03.03.

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

[8] Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

[9] Crock, J. G., Felichte, F. E., and Briggs, P. H., "Determination of Elements in National Bureau of Standards Geological Reference Materials SRM 278 Obsidian and SRM 688 Basalt by Inductively Coupled Argon Plasma—Atomic Emission Spectrometry," *Geostandards Newsletter,* Vol 7, 1983, pp. 335–340.

[10] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, Attn: NPODS.

[11] The Joint Committee on Powdered Diffraction has established a Powder Diffraction File. The committee operates on an international basis and cooperates closely with the Data Commission of the International Union of Crystallinity and ASTM. Hydroxylapatite data can be found on file card No. 9-432; beta tricalcium phosphate data can be found on file card No. 9-169.

3.1.4 *hydroxylapatite*—A calcium phosphate crystalline compound of empirical chemical formula, $Ca_5(PO_4)_3OH$ (see Specification F 1185).

4. Chemical or Crystallographic Requirements, or Both

4.1 *Chemical*:

4.1.1 Elemental analysis for calcium and phosphorous and intentional additions (other than trace elements) will be consistent with the expected stoichiometry of the specific calcium phosphate compound(s).

4.1.2 *Trace Element Analysis*—The concentration of trace elements in the coating shall be limited as follows:

Element	ppm, max
As	3
Cd	5
Hg	5
Pb	30
total heavy metals (as lead)	50

For reference purposes, the *U.S. Pharmacopeia XXI* and *U.S. Geological Survey Method, Cadmium*, shall be used.

4.1.3 The analysis of other trace elements may be required, based on the conditions, apparatus, or environments specific to the coating application technique used.

4.1.4 The analysis of intentional additional elements or compounds such as fluorine, manganese, magnesium, carbonate, etc. must be specified for calcium phosphate coatings.

4.2 *Crystallographic Characterization*:

4.2.1 This involves the degree of crystallinity, determined as a weight percent of the total substance.

4.2.2 This involves the identification of crystalline species and their weight percentages, expressed as a percent of the total substance.

4.2.3 This involves the identification of functional groups by infrared analysis.

4.3 *Environmental Stability*—The characterization of environmental stability shall be consistent with prior referenced methods using solutions best representing the potential in-service device environments.

5. Physical Characterization

5.1 *Coverage of Substrate*:

5.1.1 Microscopic examination of the surface will be made at 10× magnification; flaws, “bare” areas, “pinholes,” and coating borders, etc. will be reported for those observed at 10× magnification.

5.1.2 Visible impurities shall be reported if observed at 10× magnification.

5.2 *Thickness*—The thickness will be measured from cross sections and will be reported as the mean and range of thickness. If distinct layers are noted, each layer should be so characterized.

5.3 *Porosity*—The microporosity and macroporosity characterization shall be determined.

5.4 *Color*—The color should be uniform and consistent in consideration of the specific process, substrate material and geometry, and coating thickness.

5.5 *Surface Topography*—the surface topography shall be measured using equipment designed to determine surface roughness dimensions as microinches or micrometres. Characterization of the surface topography of the underlying substrate may be required if applicable for the specific coating method. Scanning electron microscopy shall be used to establish the coating surface and cross-section morphology.

5.6 *Other Characterizations*—Other characterizations may be required or applicable, depending on the end-use application. Standard test methods should be used in all cases, where available, or acceptable alternatives developed. An additional ASTM test that may apply is Test Methods C 674.

6. Mechanical Characterization

6.1 The following mechanical characterizations may be applicable to a coating, depending on the substrate material or geometry, coating thickness or location, and coating methods(s), or some combination thereof. Characterization reports shall contain sufficient information regarding the test techniques, procedures, and standards used and details such as specimen orientation and proportional depth of thickness in order to represent the analysis accurately.

6.1.1 The tensile bond strength of the coating to the substrate shall be measured using a non-penetrating adhesive such as FM-1000 Adhesive Film. The measurement technique shall follow the general procedure of Test Methods C 633 and F 1147. The tensile test shall be considered invalid if any portion of the metal substrate is exposed to the surface before testing, allowing direct bonding of the adhesive to the metal.

6.1.2 Shear strength determinations shall be consistent with Test Method F 1044, or a demonstrated equivalent technique, as long as the adhesive is demonstrated to be non-penetrating and the test technique allows for the coating to be placed in a pure shear condition, minimizing “peeling stress” modes.

6.1.3 The fatigue strength shall be determined on an applicable full-scale device and as a materials system using appropriate testing methods applicable to the various service conditions.

6.1.4 Abrasion resistance shall be determined using a comparative model such as Test Method C 501, or a demonstrated equivalent, with two standards of comparison, one more abrasive and one less abrasive. A function-related installation abrasion test, specific to the device and its associated instrumentation, may additionally be required.

6.1.5 Other mechanical evaluations of calcium phosphate coatings shall be made, depending on service environments, installation techniques, or other load considerations.

7. Test Specimen Fabrication

7.1 All test specimens for coating characterizations shall be prepared from indicative coating lots and samples from the same production feedstock lots and prepared on the same equipment, representative of the coating as applied to the devices.

7.2 For device characterization, all test specimens should be subjected to the same processing and sterilization as the finished device, if applicable.

8. Quality Program Requirements

8.1 The manufacture of calcium phosphate coatings will conform to good manufacturing practices (21 CFR 820).