



Designation: F1609-03^{ε1} Designation: F 1609 – 08

Standard Specification for Calcium Phosphate Coatings for Implantable Materials¹

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.

~~^{ε1}Note—Mercury warning was editorially added in April 2008.~~

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 of other ceramic or metallic,³ 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.

1.6 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

2. Referenced Documents

2.1 ASTM Standards:⁴

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

F 1044 Test Method for Shear Testing of Calcium Phosphate Coatings and Metallic Coatings

F 1088 Specification for Beta-Tricalcium Phosphate for Surgical Implantation

F 1147 Test Method for Tension Testing of Calcium Phosphate Coatings and Metallic Coatings

F 1160 Test Method for Shear and Bending Fatigue Testing of Calcium Phosphate and Metallic Medical and Composite Calcium Phosphate/Metallic Coatings

F 1185 Specification for Composition of Ceramic-Hydroxylapatite for Surgical Implants

F 1854 Shear and Bending Fatigue Testing of Calcium Phosphate and Metallic Medical and Composite Calcium Phosphate Metallic Coatings Test Method for Stereological Evaluation of Porous Coatings on Medical Implants

F 1926 Test Method for Evaluation of the Environmental Stability of Calcium Phosphate Coatings

F 2024 Practice for X-Ray Diffraction Determination of Phase Content of Plasma-Sprayed Hydroxylapatite Coatings

2.2 Pharmacopeia Convention Documents:⁵

National Formulary XVI, Tribasic Calcium Phosphate

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 Sept. 10, 2003. Published October 2003. Originally approved in 1995. Last previous edition approved in 1995 as F1609-95.

Current edition approved May 1, 2008. Published June 2008. Originally approved in 1995. Last previous edition approved in 2003 as F 1609 – 03^{ε1}.

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

³ 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.

⁴ 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.

⁵ Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

⁶ Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, <http://www.usp.org>.

United States Pharmacopeia:

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

2.3 *Other Documents:*

U.S. Geological Survey Method, Cadmium⁶

U.S. Code of Federal Regulations Title 21 (CFR 21), Part 820—Quality System Regulation⁷

X-Ray Diffraction Analyses³

3. Terminology

3.1 *Definitions:*

3.1.1 *amorphous calcium phosphate*—a non-crystalline calcium phosphate.

3.1.2 *beta tricalcium phosphate*—a calcium phosphate substance of empirical chemical formula, $\text{Ca}_3(\text{PO}_4)_2$ (see Specification F 1088).

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

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

3.1.5 *hydroxylapatite*—a calcium phosphate crystalline compound of empirical chemical formula, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ (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~~shall be consistent with the expected stoichiometry of the specific calcium phosphate compound(s).

4.1.2 *Trace Element Analysis for Hydroxylapatite and Beta Tricalcium Phosphate*—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* U.S. Pharmacopeia (most current) 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, and so forth ~~must~~shall be specified for calcium phosphate coatings.

4.1.5 Calcium to Phosphorus ratio (Ca/P) shall be performed on both the powder and coating forms using a suitable method.

4.2 *Crystallographic Characterization* :

4.2.1 Crystallographic characterization shall be in accordance with Practice F 2024.

4.2.2 Testing shall include quantitative phase analysis and amorphous calcium phosphate content.

4.2.3 FTIR (Fourier Transform Infrared Spectroscopy) shall be performed to identify functional groups.

4.3 *Environmental Stability*—Environmental stability testing shall be performed in accordance with Test Method F 1926 to access the relative dissolution behavior of the material.

5. Physical Characterization

5.1 *Coverage of Substrate:*

5.1.1 Microscopic examination of the surface will be made at 10× magnification; “bare” areas, “pinholes,” cracking, foreign debris, unmelts, chips, delamination and the appearance at the coating/substrate interface, and so forth ~~will~~shall be reported.

5.2 *Thickness*—The thickness ~~will~~shall be measured from cross sections in accordance with Test Method F 1854. If distinct layers exist, they should be reported.

5.2.1 Alternatively, a magnetic field or eddy current technique may be used if it has been shown to be equivalent to Test Method F 1854.

5.3 *Porosity*—The microporosity and macroporosity characterization shall be determined in accordance with Test Method F 1854.

⁶ 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.

⁷ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, Attn: NPODS.