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## Standard Specification for Composition of Ceramic Hydroxylapatite for Surgical Implants<sup>1</sup>

This standard is issued under the fixed designation F 1185; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

~~<sup>ε1</sup>Note—Keywords were added and Section 3 editorially corrected in March 1993.~~

### 1. Scope

1.1 This specification covers ~~material~~ chemical and crystallographic requirements for ~~ceramic~~ hydroxylapatite intended for surgical implants. For a material to be called ~~ceramic~~ hydroxylapatite, it must conform to this specification. (See Appendix X1.)

1.2 The biological response to ~~ceramic~~ hydroxylapatite in soft tissue and bone has been characterized by a history of clinical use (1-3)<sup>2</sup> and by laboratory studies (4-6) .

~~1.3 This specification specifically excludes hydroxylapatite coatings, non-ceramic hydroxylapatite, ceramic-glasses, tribasic calcium phosphate, whitlockite, and alpha- and beta-tricalcium phosphate. (See Specification F1088~~

1.3 This specification includes powder, particulate, and forms intended for use as surgical implants, components of surgical implants, or as raw materials for manufacturing processes such as thermal spray coating, electrophoretic deposition, physical vapor deposition, and so forth.

1.4 This specification specifically excludes hydroxylapatite coatings, amorphous calcium phosphate, ceramic-glasses, tribasic calcium phosphate, whitlockite, and alpha- and beta-tricalcium phosphate. (See Specification F 1088.)

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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:<sup>3</sup>

F 748 [Practice for Selecting Generic Biological Test Methods for Materials and Devices](#)

F 981 [Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone](#)

F 1088 [Specification for Beta-Tricalcium Phosphate for Surgical Implantation](#)

~~2.2 Specification for Beta-Tricalcium Phosphate for Surgical Implantation~~

F 2024 [Practice for X-ray Diffraction Determination of Phase Content of Plasma-Sprayed Hydroxyapatite Coatings](#)

#### 2.2 Code of Federal Regulations:<sup>4</sup>

Title 21, Part 820.

#### 2.3 National Formulary:<sup>5</sup>

Tribasic Calcium Phosphate

#### 2.4 United States Pharmacopeia:<sup>6</sup>

Identification Tests for Calcium and Phosphate <191>

Lead <251>

Mercury <261>

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee F4 F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials.

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

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards*, Vol 13.01, volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Available from U.S. Government Printing Office, Washington, DC 20402.

<sup>4</sup> Available from U.S. Government Printing Office, N. Capitol and H St., NW, Washington, DC 20402.

<sup>5</sup> National Formulary XVI. Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

<sup>6</sup> United States Pharmacopeia XXI. Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

Arsenic <211>

Heavy Metals <231> Method 1

2.5 *U. S. Geological Survey Method*.<sup>7</sup>

Cadmium

2.6 *American Society for Quality*.<sup>8</sup>

C1 Specification of General Requirements for a Quality Program

### 3. Terminology

3.1 *Descriptions of Terms Specific to This Standard:*

3.1.1 *calcining*—the heat treatment of a ceramic precursor for the purpose of eliminating volatile constituents. Calcining is also accompanied by some surface area and bulk volume reductions. Increases in mechanical properties are not usually significant.

3.1.2 *ceramic hydroxylapatite*—hydroxylapatite which has been fired at sintering temperatures. Firing time is mass dependent, and should be sufficiently long to cause significant densification and formation of a biologically stable form.

3.1.3 *hydroxylapatite*—the chemical substance having the empirical formula  $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ .

3.1.4 *sintering*—an integration of time and temperature of a ceramic precursor which develops a coherent body with useful properties. Sintering is a non-melting process accompanied by significant surface area and bulk volume reductions (densification), grain growth, and increases in mechanical properties.<sup>9</sup>

### 4. Chemical Requirements

4.1 Elemental analysis for calcium and phosphorus will be consistent with the expected stoichiometry of hydroxylapatite.

4.2 A quantitative X-ray diffraction analysis shall indicate a minimum hydroxylapatite content of 95% (7)

4.1 Elemental analysis for calcium and phosphorus will be consistent with the expected stoichiometry of hydroxylapatite. The calcium and phosphorus contents shall be determined using a suitable method such as ion chromatography.

4.2 A quantitative X-ray diffraction analysis shall indicate a minimum hydroxylapatite content of 95 % as determined in accordance with Practice F 2024. Analysis of relative peak intensities shall be consistent with published data.

4.3 The concentration of trace elements in the hydroxylapatite shall be limited as follows:<sup>10</sup>

4.3 For hydroxylapatite derived from natural sources, the concentration of trace elements shall be limited as follows:

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

For referee purposes, methods in

Either inductively coupled plasma/mass spectroscopy (ICP/MS), atomic absorption (AAS), or the methods listed in 2.4 and 2.5 shall be used.

4.4 The maximum allowable limit of all heavy metals determined as lead will be 50 ppm as described in

4.3.1 The analysis of other trace elements may be required, based on the conditions, apparatus, or environments specific to the manufacturing techniques and raw materials.

4.4 The maximum allowable limit of all heavy metals determined as lead will be 50 ppm as described in 2.4 or equivalent. Sample preparation will be identical to that for tribasic calcium phosphate as specified in the National Formulary (2-22.3) except that approximately 1 g of material will be dissolved in approximately 30 mL of 5 % HCl and boiled.

4.5 It is recommended that all metals or oxides not detected as lead present in concentrations equal to or greater than 0.1 % be listed on the package insert.

### 5. Test Specimen Fabrication

5.1 Prepare test specimens from the same batch of material and by the same processes as those employed in fabricating the ceramic implant device. Biocompatibility

<sup>7</sup> 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.

<sup>8</sup> Chemical Abstracts Service Registry Number [1306-06-5].

<sup>8</sup> Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, <http://www.asq.org>.

<sup>9</sup> The Joint Committee on Powdered Diffraction Standards 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 Crystallography and ASTM (American Society for Testing and Materials). Hydroxylapatite data can be found on file card number 9-432 and is available from the Joint Committee on Powder Diffraction Standards, 1600 Park Lane, Swarthmore, PA 19081.

<sup>9</sup> Chemical Abstracts Service Registry Number [1306-06-5].

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