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Implants for surgery — Hydroxyapatite —

Part 6: Powders

Implants chirurgicaux — Hydroxyapatite —

Partie 6: Poudres

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials.*

ISO 13779 consists of the following parts, under the general title *Implants for surgery — Hydroxyapatite*:

— *Part 1: Ceramic hydroxyapatite*

— *Part 2: Coatings of hydroxyapatite*

— *Part 3: Chemical analysis and characterization of crystallinity and phase purity*

— *Part 4: Determination of coating adhesion strength*

— *Part 6: Powders*

This corrected version of ISO 13779-6:2016 incorporates the following change:

— In A.8, the last line of formula for the value ka_1 has been corrected and added.

Introduction

No known surgical implant material has ever been shown to be completely free of adverse reactions in the human body. However, long term clinical experience of the use of the material referred to in this part of ISO 13779 has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.

This part of ISO 13779 describes specifications for hydroxyapatite raw material powders used to obtain high-quality medical devices. However, the quality of the final device depends on the manufacturing process and it is recognized that a separate performance standard can be necessary for each end-use product.

Implants for surgery — Hydroxyapatite —

Part 6:
Powders

1 Scope

This part of ISO 13779 specifies requirements for hydroxyapatite powders used as a raw material for the manufacturing of surgical implants or coating of surgical implants.

This part of ISO 13779 does not apply to hydroxyapatite coatings, ceramic hydroxyapatite, glass ceramics, α- and β-tricalcium phosphate, or other forms of calcium phosphate.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate*

ISO 13779-3, *Implants for surgery — Hydroxyapatite — Part 3: Chemical analysis and characterization of crystallinity and phase purity*

ISO 14971, *Medical devices — Application of risk management to medical devices*

ISO 24235, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of particle size distribution of ceramic powders by laser diffraction method*

European Pharmacopoeia 5.0: Tribasic calcium phosphate

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
atomisation
<spray drying> process for producing more or less spherical agglomerates of powder particles (atomized powders) by spraying a suspension of particles followed by immediate drying

3.2
α tricalcium phosphate
α-TCP
chemical compound with a crystallographic structure characterized by ICDD PDF 09-0348 (see Bibliography)

Note 1 to entry: The chemical formula is $Ca_3(PO_4)_2$.

Note 2 to entry: International Centre for Diffraction Data Powder Diffraction File (ICDD PDF).

3.3
β tricalcium phosphate
β-TCP
chemical compound with a crystallographic structure characterized by ICDD PDF 09-0169 (see Bibliography)

Note 1 to entry: The chemical formula is $Ca_3(PO_4)_2$.

Note 2 to entry: International Centre for Diffraction Data Powder Diffraction File (ICDD PDF).

3.4
calcination
thermal treatment of the powder in order to remove volatile impurities or to change the density or specific surface area of the powder

3.5
calcium oxide
CaO
chemical compound with a crystallographic structure characterized by ICDD PDF 4-0777 or 82-1690 (see Bibliography)

Note 1 to entry: International Centre for Diffraction Data Powder Diffraction File (ICDD PDF).

3.6
crystallinity ratio
ratio between the mass fraction of crystalline hydroxyapatite and the total mass fraction of hydroxyapatite (crystalline and amorphous)

3.7
D_{50}
particle diameter corresponding to 50 % of the cumulative undersize volume distribution

Note 1 to entry: On a volumetric basis size distribution, 50 % of the particles is smaller than D_{50}.

3.8
D_{10}
particle diameter corresponding to 10 % of the cumulative undersize volume distribution

Note 1 to entry: On a volumetric basis size distribution, 10 % of the particles is smaller than D_{10}.

3.9
D_{90}
particle diameter corresponding to 90 % of the cumulative undersize volume distribution

Note 1 to entry: On a volumetric basis size distribution, 90 % of the particles is smaller than D_{90}.

3.10
foreign phase
crystalline phase other than hydroxyapatite

3.11
grinding
process for reducing the size of the raw powder particles

3.12
hydroxyapatite
HA
chemical compound with a crystallographic structure characterized by ICDD PDF 09-0432 or 72-1243 (see Bibliography)

Note 1 to entry: The chemical formula is $Ca_{10}\,(PO_4)_6(OH)_2$.

Note 2 to entry: International Centre for Diffraction Data Powder Diffraction File (ICDD PDF).

3.13
hydroxyapatite ceramic
hydroxyapatite which has been sintered into a body of high crystallinity

3.14
hydroxyapatite coating
hydroxyapatite which has been deposited onto the surface of a metallic or non-metallic substrate

Note 1 to entry: Material deposition can be obtained either by means of a thermal spray process which produces a ceramic-type coating or by means of a solution-based technique which may deposit hydroxyapatite directly or may require thermal or other treatment to convert it into a crystalline form.

3.15
pressing
process for producing green (before sintering) ceramics under pressure causing the consolidation of powders to the shape of the die used

3.16
sintering
process for production of ceramics in which the application of heat promotes the coalescence of ceramic grains and causes a significant reduction of particle surface area and bulk volume to achieve densification and consequent increase in mechanical properties

3.17
specific surface area
total surface area of the powder particles per unit of mass, $m^2 g^{-1}$

3.18
tetracalcium phosphate
TTCP
chemical compound with a crystallographic structure characterized by ICDD PDF 25-1137 or 70-1379 (see Bibliography)

Note 1 to entry: The chemical formula is $Ca_4(PO_4)_2O$.

Note 2 to entry: International Centre for Diffraction Data Powder Diffraction File (ICDD PDF).

4 Requirements

4.1 General

The minimum requirements for the hydroxyapatite powder are established in 4.2 to 4.7.

According to the use of the hydroxyapatite powder, other characterization tests can be useful and should be conducted (see Annex A).

4.2 Calcium to phosphorus molar ratio (Ca/P)

The content of calcium and phosphorus of the hydroxyapatite powder shall be determined in accordance with ISO 13779-3.

A calcium to phosphorus molar ratio, Ca/P, of $1,66 \leq Ca/P \leq 1,71$ is suitable to fit the requirements of ISO 13175-3, ISO 13779-1, and ISO 13779-2.

4.3 Trace elements

The limits of specific trace elements for hydroxyapatite powders are given in Table 1.