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ISO/DIS 13779-6

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Implants for surgery — Hydroxyapatite —

Part 6:
Powders

Implants chirurgicaux — Hydroxyapatite —

Partie 6: Poudres

ICS: 11.040.40

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 13779-6 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials.*

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

Part 1: Ceramic hydroxyapatite

Part 2: Coatings of hydroxyapatite

Part 3: Chemical analysis and characterization of cristallinity and phase purity

Part 4: Determination of coating adhesion strength

Part 6: Powders

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 Standard has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.

This standard 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 characterised by 09-0348 of the ICDD.PDF (see bibliography)

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

3.3
β tricalcium phosphate
(β-TCP)
chemical compound with a crystallographic structure characterised by 09-0169 of the ICDD.PDF (see bibliography)

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

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 4-0777 or 82-1690 of the ICDD.PDF (see bibliography)

3.6
$\boldsymbol{D_{50}}$
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}.

3.7
$\boldsymbol{D_{10}}$
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}.

3.8
$\boldsymbol{D_{90}}$
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}

3.9
grinding
process for reducing the size of the raw powder particles

3.10
hydroxyapatite
(HA)
chemical compound with a crystallographic structure characterised by 09-0432 or 72-1243 of the ICDD.PDF (see bibliography)

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

3.11
hydroxyapatite ceramic
hydroxyapatite which has been sintered into a body of high crystallinity

3.12
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.

3.13
pressing
process for producing green (before sintering) ceramics under pressure causing the consolidation of powders to the shape of the die used

3.14
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

3.15
specific surface area
total surface area of the powder particles per unit of mass, $m^2 g^{-1}$

3.16
tetracalcium phosphate
(TTCP)
Chemical compound with a crystallographic structure characterised by 25-1137 or 70-1379 of the ICDD PDF(see bibliography)

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

3.17
foreign phase
crystalline phase other than hydroxyapatite

4 Requirements

4.1 General

4.2 to 4.8 define the minimum requirements for the hydroxyapatite powder.

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 13779-1, ISO 13779-2 and ISO 13179-3.