INTERNATIONAL STANDARD

ISO 13779-3

Second edition
2018-12

Implants for surgery — Hydroxyapatite —

Part 3: Chemical analysis and characterization of crystallinity ratio and phase purity

Implants chirurgicaux — Hydroxyapatite —

Partie 3: Analyse chimique et caractérisation du rapport de cristallinité et de la puréte de phase

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Reference number
ISO 13779-3:2018(E)

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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 13779-3:2008), which has been technically revised.

A list of all parts in the ISO 13779 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

No known surgical implant material has ever been shown to cause absolutely no adverse reactions in the human body. However, long term clinical experience of the use of hydroxyapatite has shown that an applicable level of biological response can be expected, if the material is used in appropriate applications.

Biocompatibility and resorption rate of hydroxyapatite material for surgical application may depend of the presence of trace elements, foreign crystalline phases and crystallinity ratio. Amorphous calcium phosphate, tetracalcium phosphate, α-tricalcium phosphate and β-tricalcium phosphate have demonstrated to have a higher solubility and may resorb more rapidly than hydroxyapatite in the body. CaO and heavy metals may impair the biocompatibility of the material. As a consequence, it is important to assess the composition of the material.

In this field, the assessment of the different crystalline and amorphous phase components has been under continuing development (of both equipment and processing software). In this document a new method for measuring the crystallinity ratio of hydroxyapatite is introduced and the Rietveld method is introduced as an alternative method for measuring the foreign phase content.

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Implants for surgery — Hydroxyapatite —

Part 3: Chemical analysis and characterization of crystallinity ratio and phase purity

1 Scope

This document specifies methods of test for the chemical analysis, assessment of crystallinity ratio and phase composition of hydroxyapatite-based materials such as powders, coating or bulk products.

NOTE These tests are intended to describe properties of the material and to communicate these between organizations. These tests are not written with the objective of replacing a company's internal operational and assessment tests although they could be used as such.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
calibration curve
calculating plot translating the ratio of integrated intensity of foreign phases, measured on the X-ray diffraction pattern into the mass fraction of foreign phases compared to crystalline hydroxyapatite

3.2
detection limit
DL
lowest quantity of the foreign phase or trace element that can be distinguished from the absence of that foreign phase or trace element

Note 1 to entry: Requirements and procedure for estimating the detection limit of foreign phases is established in 5.6.3.

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3.3
quantification limit
QL
lowest quantity of the foreign phase or trace element that can be quantified

Note 1 to entry: Requirements and procedure for estimating the quantification limit of foreign phases is established in 5.6.3.

3.4
height
distance between the peak summit and the base line of the X-ray diffraction pattern from which the background has been subtracted

3.5
integrated intensity
area between the plot of the peak and the base line of the X-ray diffraction pattern from which the background has been subtracted

3.6
scraping
removal of the coating from the base material minimising contamination from of the base material itself

3.7
signal:noise ratio
height of a peak of the x-ray diffraction pattern divided by the maximum deviation of the base line oscillation

Note 1 to entry: The height of each peak of the X-ray diffraction pattern divided by the maximum deviation of the base line oscillation near to the peak location, in an area not likely to present a crystalline phase peak.

3.8
***α*-tricalcium phosphate**
***α*-TCP**
chemical compound with a crystallographic structure characterized by IC DD PDF 09-0348

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

3.9
***β*-tricalcium phosphate**
***β*-TCP**
chemical compound with a crystallographic structure characterized by IC DD PDF 09-0169

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

3.10
hydroxyapatite
HA
chemical compound with a crystallographic structure characterized by IC DD PDF 09-0432 or IC DD PDF 72-1243

Note 1 to entry: The chemical formula is $Ca_5(OH)(PO_4)_3$.

3.11
tetracalcium phosphate
TTCP
chemical compound with a crystallographic structure characterized by IC DD PDF 25-1137 or IC DD PDF 70-1379

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

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3.12
calcium oxide
CaO
chemical compound with a crystallographic structure characterized by ICDD PDF 4-0777 or IC DD PDF 82-1690

3.13
uncertainty
95 % confidence interval of the measurement, taking into account reproducibility of the measurement

3.14
crystallinity ratio
ratio between the sum of integrated intensities of a selection of peaks of HA in the sample and the sum of integrated intensities of the same peaks of HA after calcination of the sample at 1 000 °C for 15 h

Note 1 to entry: Sometimes crystallinity is defined as the ratio between the mass fraction of crystalline HA and the total mass fraction of HA (crystalline and amorphous). However, in this standard there is no method given to measure the total mass fraction of HA. For this reason, the term crystallinity ratio, as defined above, instead of crystallinity is used.

3.15
background
signal produced by the non-diffracted X-ray beam

Note 1 to entry: This is illustrated in Figure 1.

3.16
noise
variation of the signal intensity produced by the X-ray apparatus

Note 1 to entry: This is illustrated in Figure 1.

3.17
foreign crystalline phases
crystalline phases other than hydroxyapatite, being detectable by X-ray diffraction analysis and consisting of tetracalcium phosphate, α-tricalcium phosphate, β-tricalcium phosphate, and calcium oxide (CaO)

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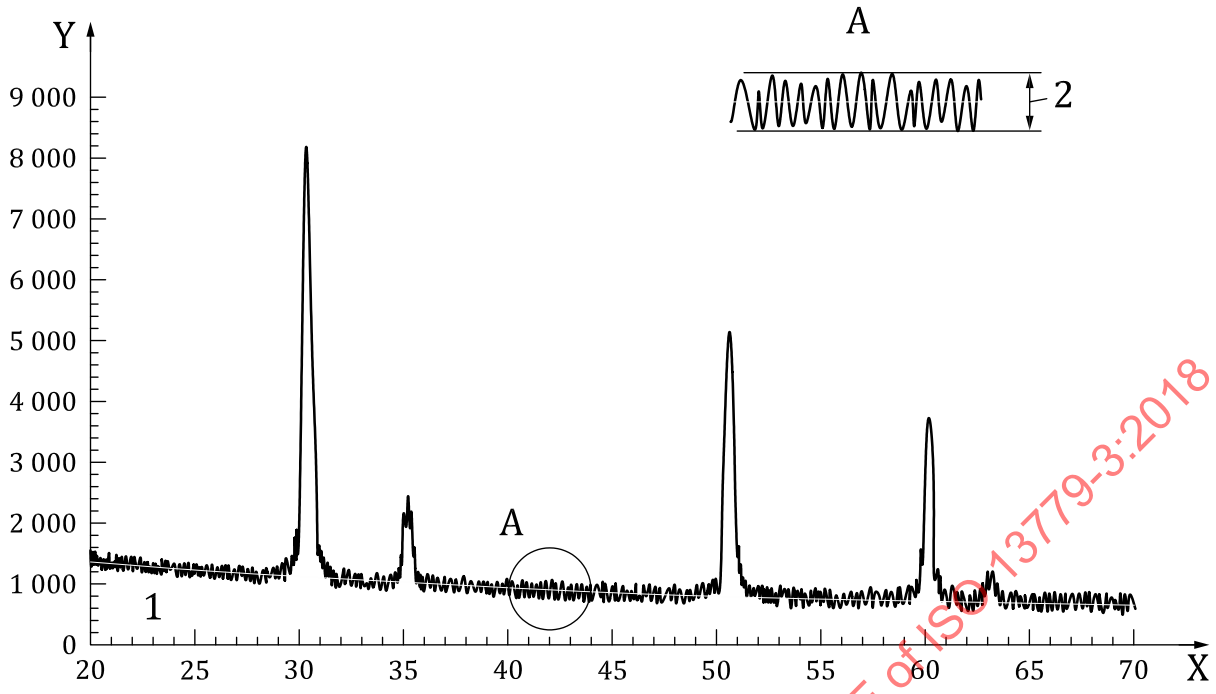

Key

1 background
2 noise
X = 2 theta
Y = counts

Figure 1 — Illustration of background and noise

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4 Chemical analysis

4.1 General

This clause specifies the methods for determining arsenic, cadmium, lead, mercury, and heavy metals content in hydroxyapatite-based materials for surgical implants.

Heavy metals content is considered as the sum of lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper and molybdenum.

NOTE This list of elements is contained in ISO 13779-6. Comparison of trace elements in an HA coating with the same trace elements in an HA powder indicates any detriment to the HA by coating process.

This method can also be used to measure calcium and phosphorous to calculate the Ca:P ratio as an alternative to the X-ray diffraction method detailed in 5.8. The equivalence of such results obtained from the chemical analysis with the results from the X-ray method should be demonstrated.

There can be a need to analyse other elements (see Annex A).

4.2 Analytical methods

The following methods can be used after validation and determination of detection limit, quantification limit and measurement tolerances. Each analytical method has its own DL and QL relevant to that method. An appropriate quantitative analysis apparatus, having a quantification limit which is not less than the required limit value in the analysed sample, shall be used.

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The list is not restrictive:

a) atomic absorption spectroscopy, hydride method;

b) atomic absorption spectroscopy with electro-thermal atomization using matrix modifiers;

EXAMPLE Palladium-magnesium nitrate.

c) flame atomic absorption spectroscopy after complexion and extraction;

d) inductively coupled plasma / mass spectroscopy (ICP-MS);

e) inductively coupled plasma / atomic emission spectroscopy (ICP-AES); or

f) atomic absorption spectroscopy (AAS).

Calibration can be external or based on the standard addition method. If external, the calibration solution shall contain HA with known content in elemental impurities to take into account the effect of the HA matrix. The HA concentration used for calibration shall be the same as the HA concentration in the tested solution.

4.3 Apparatus for chemical analysis

Vessels used shall be either of the following:

4.3.1 Class A glassware. or

4.3.2 PTFE flask (or similar).

The vessels used for dissolution of the sample shall not contaminate the solution.

NOTE For analysis of Mercury, PTFE vessels is suitable to avoid potential contamination of the solution.

Before use they shall be carefully washed with acid and then rinsed with grade 2 water (4.4.1).

4.4 Reagents for chemical analysis

All reagents shall be of analytical quality:

4.4.1 Grade 2 water, according to ISO 3696.

4.4.2 Analytical-grade nitric or hydrochloric acid.

4.4.3 Standard solutions of the elements to be determined, prepared either by weighing or from commercially available standard solutions.

4.5 Procedure

Bulk samples shall be crushed. In the case of coatings, the HA needs to be removed by scraping. Scraping of coatings and crushing of bulk samples shall be performed to minimize contamination of the sample.

The HA sample shall be dissolved in acid. The mass of the HA sample and acid type and concentration might need to be adjusted depending on the analytical method used as well as the quantity of the element to analyse, present in the HA matrix. The mass of the HA sample as well as the type, volume and concentration of the acid used shall be recorded.

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Place ground sample into PTFE or glass flask (4.3), carefully add the acid solution consisting of grade 2 water (4.4.1) and of nitric or hydrochloric acid [4.4.2].

NOTE As a starting point using (1,000 ± 0,001) g of ground sample, (30 ± 0,05) ml of grade 2 water and (1 ± 0,05) ml of 52,5 w% nitric or hydrochloric acid can be appropriate.

Dilute (e.g. to 50 ml volume) using grade 2 water (4.4.1), seal and shake thoroughly. Ensure that the sample is totally dissolved. A blank test shall be conducted at the same time.

The solution shall be analysis by a suitable analytical method (4.2).

4.6 Expression of results

Values of impurities which are less than the detection limit shall be expressed as follows:

$X < DL$,

where X is the analysed chemical element and DL the detection limit expressed in mg/kg.

Values of impurities which are between the detection limit and the quantitation limit shall be expressed as follows:

$DL < X < QL$, where X is the analysed chemical element, DL the detection limit expressed in mg/kg, and QL is the quantitation limit expressed in mg/kg.

For heavy metals content determination, the content of chemical elements below the detection limit shall be considered as equal to DL. If the value is between DL and QL the QL shall be used.

Values of heavy metal content and impurities which are greater than the detection limit shall be rounded to the nearest 0,1 mg/kg. The values for each heavy metal impurity shall be reported.

5 X-ray diffraction analysis

5.1 General

The X-ray diffraction (XRD) method described in Clause 5 is based on the comparison of the integrated intensities of the XRD pattern of a sample with reference materials.

An alternative method is the Rietveld method (see Annex G) for the calculation of the foreign phases content. The Rietveld method may be used instead of the method described in Clause 5 once correlated with the integrated intensity method. The QL, DL and accuracy of the Rietveld method shall be calculated and the difference of the results of the two methods quantified.

5.2 Apparatus

The apparatus shall consist of the following:

5.2.1 Mortar and pestle, in alumina, agate or other suitable material (minimising contamination of the sample).

5.2.2 38 or 40 micrometre sieve, complying with ISO 3310-1.

5.2.3 Oven, capable of maintaining a temperature of (1 000 ± 25) °C.

5.2.4 Desiccator.

5.2.5 Intensity reference material, to determine instrument intensity, suitable materials include bulk gold or alumina reference (not in the form of powder, for example NIST SRM 1976).

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5.2.6 X-ray diffraction apparatus having a resolution and reproducibility of at least 0,02° on a 2 θ angle scale and allowing the recording of the diffraction peak positions and intensities. A stabilized power supply is necessary. Software of the diffraction apparatus shall allow adjusting the peaks position, subtracting the background and measuring the integrated intensities of the peaks over a determined angular range.

5.3 Preparation of test samples

5.3.1 General

The test samples shall be crushed and sieved so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks. It is necessary to minimize any contamination or transformation in contact with humidity. Keep all the test samples in the desiccator.

5.3.2 Coatings

In the case of coatings, the HA needs to be removed by scraping. Scraping shall be performed to minimise contamination of the sample.

For thermally sprayed coatings, it is common that the layers near to the coating/substrate interface contain more amorphous material than those areas far from the interface. Therefore, the sample shall be taken from a mixture of the whole coating layer to obtain a representative sample of the coating.

5.3.3 Bulk sample

Bulk samples shall be reduced to powder form according to 5.3.1.

5.4 Calibration specimens

The calibration specimens listed below shall be used:

a) Pure β-tricalcium phosphate having an X-ray diffraction pattern as described in ICDD PDF 09-0169.

b) Pure α-tricalcium phosphate having an X-ray diffraction pattern as described in ICDD PDF 9-348.

c) Pure hydroxyapatite having an X-ray diffraction pattern as described in ICDD PDF 9-432 or ICDD PDF 72-1243.

d) Pure tetracalcium phosphate having an X-ray diffraction pattern as described in ICDD PDF 25-1137 or ICDD PDF 70-1379.

e) Pure calcium oxide having an X-ray diffraction pattern as described in ICDD PDF 4-0777 or ICDD PDF 82-1690.

Calibration specimens shall be prepared by the methods described in Annex E. They shall comply with the requirements described in Annex B.

5.5 X-ray diffraction pattern collection

5.5.1 General

The conditions of the X-ray diffraction pattern collection will allow the contribution of the apparatus to the full width half maximum, detection limit and tolerances to be minimized; these conditions shall be identical for the test sample and for the mixtures used to prepare the calibration curves. The signal: noise ratio shall be greater than 20 for peak 211 of HA. If an amorphous hump is visible in the pattern, it should be subtracted with the background before measuring the noise.

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The diffractometer settings shall allow a resolution of 0,02° on a 2 θ angle scale. Peak deconvolution should be avoided but may be required where overlap of peaks occurs.

The peak integrated intensities of all phases shall be determined to an accuracy greater than 5 %, using suitable software.

2 θ minimum and maximum limit for integration area of each peak shall be taken so that the full peak falls between the limits.

NOTE In some cases, it might be necessary to shift the axis in order to center the peak considered in the 2 θ range validated for the measurement of the peak integrated intensity.

The same 2 θ minimum and maximum limits shall be used for the construction of calibration curves according to 5.6 and for the analysis of samples according to 5.7 and 5.8.

5.5.2 Identification of the crystallized phases

The isolated crystalline phases shall be identified according to their characteristic lines given in the respective ICDD files[1]. The selected lines for the construction of calibration curves, foreign phases content and Ca:P ratio determination could be:

— the line 0.2.10 (d = 2,88 × 10^{-10} m) of the β-tricalcium phosphate;

— the line 4.4.1, 1.7.0 (d = 2,905 × 10^{-10} m) of the α-tricalcium phosphate;

— the line 0.4.0 (d = 2,995 × 10^{-10} m) of the tetracalcium phosphate;

— the line 2.0.0 (d = 2,405 × 10^{-10} m) of the calcium oxide;

— the line 2.1.0 (d = 3,08 × 10^{-10} m) of the hydroxyapatite.

Other lines may be chosen provided that they do not affect the sensitivity of the determination.

NOTE The quantification of the foreign phases is often difficult to carry out on account of spectral interference and of broadening of the lines of the foreign phases compared to the reference materials. The minor tetracalcium phosphate, α-tricalcium phosphate, β-tricalcium phosphate and calcium oxide contents are determined relative to the reference line of the hydroxyapatite phase which does not interfere with the main diffraction peaks of these phases.

Annex C shows some X-ray diffraction patterns of HA, TTCP, α-TCP, β-TCP and CaO.

5.6 Calibration curves, limits and uncertainties

5.6.1 General

To qualify the X-ray diffraction equipment and method used, calibration curves shall be plotted, detection limit, qualification limit and uncertainties of the test method shall be determined. This qualification is effective until the equipment or the method is changed.

The calibration curve represents the foreign phase mass fraction as a function of the respective foreign phase integrated intensity ratio (R1, R2, R3 and R4) as established in 5.6.2.

Use the calibration specimens described in 5.5 to prepare the samples for X-ray diffraction patterns required to plot the calibration curve.

At least three X-ray diffraction patterns, as described in 5.5, shall be collected for each calibration mix required in 5.6.2, for defining each point in calibration curves.

5.6.2 Plotting the calibration curves for the foreign phases

In order to plot the calibration curves of the hydroxyapatite associated with all every foreign phases:

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Produce, by weighing and crushing, pure calibration specimen mixtures (see 5.3) of hydroxyapatite and each foreign phases containing increasing quantities of foreign phases, with at least 5 mass fractions distributed to the range of expected mass fractions of the foreign phase in the test samples.

As the calibration curves are also used for determinations of the foreign phase detection limits (5.6.3), the minimum value should be sufficiently low to enable the detection and quantification limits to be assessed.

Collect the X-ray diffractogram for the 2 θ angle range corresponding to the peaks selected for hydroxyapatite and for the foreign phase in each sample (e.g. peak 2.1.0 of hydroxyapatite and the indicated peak for each foreign phase).

Determine the integrated intensity of the selected peaks for hydroxyapatite and foreign phase from each X-ray diffraction pattern for each sample.

Determine the average integrated intensities for the hydroxyapatite peak and the foreign phase.

For each mass fraction of foreign phase, calculate the foreign phase integrated intensity ratios, R_i, as follows:

The average integrated intensity ratio for HA/β-TCP:

$$R_i = \frac{\bar{I}_{\mathrm{HA}}}{\bar{I}_{\beta\text{-TCP}}} \tag{1}$$

The average integrated intensity ratio for HA/CaO:

$$R_i = \frac{\bar{I}_{\mathrm{HA}}}{\bar{I}_{\mathrm{CaO}}} \tag{2}$$

The average integrated intensity ratio for HA/α-TCP:

$$R_i = \frac{\bar{I}_{\mathrm{HA}}}{\bar{I}_{\alpha\text{-TCP}}} \tag{3}$$

The average integrated intensity ratio for HA/TTCP:

$$R_i = \frac{\bar{I}_{\mathrm{HA}}}{\bar{I}_{\mathrm{TTCP}}} \tag{4}$$

where

$\bar{I}_{\mathrm{HA}}$ is the average integrated intensity of the peak selected for HA;

$\bar{I}_{\beta\text{-TCP}}$ is the average integrated intensity of the peak selected for β-TCP;

$\bar{I}_{\mathrm{CaO}}$ is the average integrated intensity of the peak selected for CaO;

$\bar{I}_{\alpha\text{-TCP}}$ is the average integrated intensity of the peak selected for α-TCP;

$\bar{I}_{\mathrm{TTCP}}$ is the average integrated intensity of the peak selected for TTCP;

Using the R_i values for each material mixture, plot the calibration curves for each foreign phase.

5.6.3 Detection limit (DL) and quantification limit (QL) of foreign phases

Either Method A or Method B shall be used to determine the detection limit and the quantification limit.

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Method A

Produce 10 patterns (as described in 5.5) of pure HA (as described in 5.4). Use curve fitting to subtract the background.

After determining the minimum and maximum 2θ angles for measurement of the integrated intensity of foreign phases, the integrated intensity of the noise of the patterns shall be measured at the selected peak positions for TTCP, α-TCP, β-TCP and CaO.

— Corresponding content TTCP, α-TCP, β-TCP and CaO shall be determined using calibration curves.

— Calculate the average (X) and standard deviation (s) of the 10 measurements for TTCP, α-TCP, β-TCP and CaO.

— Calculate the DL as: DL = X + 3s for TTCP, α-TCP, β-TCP and CaO.

— Calculate the QL as: QL = X + 5s for TTCP, α-TCP, β-TCP and CaO.

Method B

A widespread procedure to determine the detection limit and the quantification limit is defined through the signal:noise approach. The signal/noise ratio is considered in terms of height of peaks.

Determination of the signal-to-noise ratio is performed:

— for the Detection limit: establishing the minimum concentration at which the analyte can be reliably detected. A typical signal:noise ratio, for the DL, is 3:1;

— for the Quantification limit: by establishing the minimum concentration at which the analyte can be reliably quantified. A typical signal:noise ratio, for the QL, is 5:1.

Produce at least 10 patterns (as described in 5.5), of pure HA (as described in 5.4). Use curve fitting to subtract the background.

After determining the minimum and maximum 2θ angles for measurement of the integrated intensity of foreign phases, the mean value of the height of the noise (N) of the patterns, in that range, shall be measured at the selected peak positions for TTCP, α-TCP, β-TCP and CaO.

For the determination of DL and QL, for each foreign phase, use the calibration curves:

for each acquisition, consider the % foreign phase amount [%fph] vs the signal:noise (S/N) ratio of that analysis and that foreign phase, plot a curve and calculate its formula,

like an example, $\frac{S}{N} = m\left[\%\text{fph}\right] + q$, where m and q are known,

— Calculate DL as: $\text{DL} = \frac{\frac{S}{N} - q}{m}$, where S/N=3 for TTCP, α-TCP, β-TCP and CaO.

— Calculate DL as: $\text{QL} = \frac{\frac{S}{N} - q}{m}$, where S/N=5 for TTCP, α-TCP, β-TCP and CaO.

5.6.4 Uncertainty for determination of foreign phases content

The expanded uncertainties (U) (at 95 % of confidence) of the determination of the foreign phases content shall be determined by a suitable method. The JCGM 100[2] describes a suitable methodology for the determination of uncertainties.

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5.7 Qualitative and quantitative determination of the foreign phases

5.7.1 Procedure

Produce the X-ray diffraction pattern of the test sample (see 5.5). Measure the integrated intensities of the peaks selected as described in 5.4, calculate the R1 to R4 ratios and refer to the calibration curves in order to determine the corresponding content of the foreign phases (see 5.6).

Compare the mean value with DL and QL determined according to 5.6. If the mean value is below the DL, it shall be recorded on the report "<DL" or "no phase detected" and DL shall be recorded on the report. If the mean value is above DL but below the QL, it shall be recorded on the report "≥x and <y" where x is the DL and y the QL.

5.7.2 Expression of results

The results shall be expressed as a percentage of foreign phases in relation to the crystalline hydroxyapatite phase.

NOTE The percentage of each foreign phase as determined using the above methods is in relation to the crystalline HA phase. For samples with amorphous phase, their actual mass percentage in relation to total crystalline phases and amorphous phase should be lower.

5.8 Calcium to phosphorous (Ca:P) ratio determination

5.8.1 General

The method is based on the premise that, after homogenization and calcination at (1 000 ± 25) °C, the calcium phosphates having a Ca:P atomic ratio between 1,50 and 2,0 inclusive are expected to form at the most, two phases:

— α- and/or β-tricalcium phosphate and hydroxyapatite, if the Ca:P ratio is below 1,667;

— hydroxyapatite and calcium oxide, if the Ca:P ratio is above 1,667.

After quantifying the tricalcium phosphate or CaO content in the calcined sample, based on the chemical formula of HAP, TCP and CaO, it is possible to calculate the molar ratio between calcium and phosphorous in the sample.

Although X-ray diffraction techniques are the traditional methodology for determining Ca:P ratio, with the increased accuracy of modern analytical methods, those stated in Clause 4 may be used to determine Ca:P ratio as an alternative to the X-ray method detailed in this subclause. The equivalence of such results obtained from the chemical analysis with the results from the X-ray method should be demonstrated.

NOTE Contamination of the sample might result in the formation of other non-Ca:P phases and alter the mineralogy. If Ti contamination occurs, then $CaTiO_3$ might be formed.

5.8.2 Measurements on the sample

Calcine the sample in air at (1 000 ± 25) °C for at least 15 h in a platinum or alumina crucible. Withdraw the sample from the oven (still at 1 000 °C, or cooled to no lower than 100 °C) and put it immediately into a desiccator.

Collect an X-ray diffractogram of the sample according to 5.5. If more than one foreign phase is observed or if the presence of a foreign phase other than β– and/or α-tricalcium phosphate or calcium oxide is detected (tetracalcium phosphate, calcium pyrophosphate, etc.), recrush the sample and recalcine it.

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Calculate the foreign phase content according to 5.7 and calculate the Ca:P ratio by the following Formula (6):

$$Ca:P = \frac{n_{Ca}}{n_P} \tag{6}$$

where

n_{Ca} is the number of moles of calcium per g of sample;

n_p Is the number of moles of phosphorus per g of sample calculated as:

$$n_{Ca} = \frac{10}{M_{HA}} w_{HA} + \frac{3}{M_{TCP_{(\alpha;\beta)}}} \left(w_{TCP_\alpha} + w_{TCP_\beta} \right) + \frac{1}{M_{CaO}} w_{CaO} \tag{7}$$

and

$$n_P = \frac{6}{M_{HA}} w_{HA} + \frac{2}{M_{TCP_{(\alpha;\beta)}}} \left(w_{TCP_\alpha} + w_{TCP_\beta} \right) \tag{8}$$

where

M_{HA} = 1004,6 g

$M_{TCP(\alpha;\beta)}$ = 310,174 g/mol

M_{CaO} = 56,077 g/mol

and

w_{HA} is the mass fraction of crystalline HA;

$w_{\beta TCP}$ is the mass fraction of crystalline β-TCP;

$w_{\alpha TCP}$ is the mass fraction of crystalline α-TCP;

w_{CaO} is the mass fraction of crystalline CaO.

When $w_{\beta TCP}$, $w_{\alpha TCP}$ or w_{CaO} is less than the detection limit, the value should be counted as zero for the calculation of Ca:P.

5.8.3 Uncertainty of Ca:P measurement

The expanded uncertainty (U) (at 95 % of confidence) of the Ca:P measurement shall be determined by a suitable method. The JCGM 100[2] describes a suitable methodology for the determination of uncertainty.

The uncertainty of the Ca:P measurement can be derived from the uncertainties of the foreign phases content as described in Annex F.

5.8.4 Expression of results

The results shall be given by a dimensionless value representing the Ca:P atomic ratio expressed to the nearest two significant figures.

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5.9 Determination of the crystallinity ratio

5.9.1 Rationale on different methods of determination of the crystallinity ratio

In the previous version of this document, published in 2008, the determination of the crystallinity ratio was based on the measurement of the integrated intensity of ten lines, suitably chosen from the sample's X-ray diffraction pattern, and of the ten same lines of a fully crystalline HA powder. As no internationally recognized fully crystalline HA reference is available, each lab used a different fully crystallized HA reference. A round Robin test, conducted in 2014, demonstrated a poor inter-laboratory reproducibility of this method[3].

A new method is described in 5.9.2, 5.9.3 and 5.9.4, where the determination of the crystallinity ratio is based on the measurement of the integrated intensity of ten lines, suitably chosen from the sample's X-ray diffraction pattern, and of the same ten lines of the sample after calcination at (1 000 ± 25) °C for not less than 15 h (fully crystallized sample). The round Robin test conducted in 2014 demonstrated a better inter-laboratory reproducibility of this method, compared to the former method[3].

NOTE 1 Based on the results of a round robin[3] performed crystallinity ratio results are not expected to change with the new method on average as compared to the method of the 2008 version of this document. However, some laboratories reported significant differences in the results when comparing the two methods. Testing laboratories might have to analyse the impact of the new method on their crystallinity ratio results, compared to the former method. The manufacturer might need to analyse the impact of the crystallinity ratio results from the new method on the product.

The method of reference is the method described in 5.9.2, 5.9.3 and 5.9.4.

The method described in Annex H, may be used. As the two methods can give different results, the method used (the method described in 5.9.2, 5.9.3 and 5.9.4 or the method described in Annex H) shall be reported. For the method described in Annex H, the equivalency of the results with those of the method described in 5.9.2, 5.9.3 and 5.9.4 shall be verified for low and high crystallinity samples and for any batch of reference crystalline HA powder used.

NOTE 2 The Rietveld method is introduced in Annex G for the quantification of foreign phases. This method can allow to determine the mass fraction of the crystalline content (HA, β-TCP, α-TCP, TTCP, CaO) to the total mass of the sample. This method is not the recommended method to determine the crystallinity ratio as defined in 3.14.

5.9.2 General

The determination of the proportion of properly crystalline hydroxyapatite phase is based on the measurement of the integrated intensity of ten lines, suitably chosen from the sample's X-ray diffraction pattern, and of the same ten lines of the sample after calcination at (1 000 ± 25) °C for not less than 15 h (fully crystallized sample). The positions of the intensity lines are reported in Annex D, Table D.1.

Two procedures are proposed for the crystallinity ratio determination. Procedure A, 5.9.3, should be preferred when the quantity of sample is sufficient to divide the sample in two parts: one part non-calcined and another part for calcination. Procedure B, 5.9.4, should be used in other cases.

As in procedure B the intensity of the X-ray diffraction generator can vary between the first set of measurements and the second set after calcination, the integrated intensities of the 10 peaks shall be normalized by the integrated intensity of the intensity reference material (gold or alumina reference), except if it can be shown that the integrated intensity varies less than 1 % between the initial measurement and the measurement after calcination.

5.9.3 Procedure A

a) Divide the sample in two parts.

b) Calcine one part of the sample in air at (1 000 ± 25) °C for at least 15 h in a platinum or alumina crucible.

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c) Collect an X-ray diffraction pattern, according to 5.5, of the uncalcined test sample. Determine the integrated intensities of the hydroxyapatite phase ten peaks referenced in Table D.1. Calculate the sum (S1) of the integrated intensities of the 10 peaks.

d) Immediately before or after the measurement on the non-calcined sample, collect the X-ray diffraction pattern, according to 5.5, of the calcined test sample. Determine the integrated intensities of the hydroxyapatite phase ten peeks referenced in Table D.1. Calculate the sum (S2) of the integrated intensities of the 10 peaks.

e) Calculate the crystallinity ratio C, in %, as follows:

$$C = \frac{S1}{S2} \cdot 100 \quad (9)$$

5.9.4 Procedure B

a) Produce an X-ray diffraction pattern of the test sample as described in 5.5.

b) Determine the integrated intensities of the hydroxyapatite phase ten peaks referenced in Table D.1. Calculate the sum (S1) of the integrated intensities of the 10 peaks.

c) If required, produce an X-ray diffraction pattern of the intensity reference material (gold or alumina) immediately before or after this first set of measurements. Determine the integrated intensity (R1), of the main diffraction peak.

d) Calcine the samples in air at (1 000 ± 25) °C for at least 15 h in a platinum or alumina crucible. Produce the X-ray diffraction pattern of the calcined test sample as described in 5.5. Determine the integrated intensities of the hydroxyapatite phase ten peaks referenced in Table D.1. Calculate the sum (S2) of the surfaces of the 10 peaks.

e) If required, produce the X-ray diffraction pattern of the intensity reference material (gold or alumina) immediately before or after this second set of measurements. Determine the integrated intensity (R2) of the main diffraction peak.

f) Calculate the crystallinity ratios C, in %, as follows:

$$C = \frac{S1 \cdot R2}{S2 \cdot R1} \cdot 100 \quad (10)$$

5.9.5 Uncertainty of the crystallinity ratio

The expanded uncertainty (U) (at 95 % of confidence) of the crystallinity ratio shall be determined by a suitable method. The JCGM 100[2] describes a suitable methodology for the determination of uncertainty.

5.9.6 Expression of results

The results shall be expressed in percent expressed to two significant figures.

6 Test report

The test report shall contain the following information:

a) reference to this document (including its year of publication) ;

b) name, location and, if available, accreditation of the laboratory;

c) date of the test;

d) number and identification of specimens tested;

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e) nature of the apparatus used for chemical analysis;

f) nature of the apparatus used for XRD (generator, goniometer, etc.), the recording conditions, (wavelength, filters, apertures monochromators, assembly types, counting times);

g) X-ray diffraction patterns of the sample;

h) the average and the standard deviation for the following characteristics (or individual results if results are under the QL):

   1) the determined foreign phase content in %, the method used and, if the Rietveld method is used, the data to reconcile the Rietveld method and the method described in 5.7

   2) the determined Ca:P ratio;

   3) the determined concentration of As, Hg, Cd, Pb in mg/kg;

   4) the determined concentration of heavy metals in mg/kg;

i) the crystallinity ratio, the method used and, if the ISO 13779-3:2008 method is used, the data addressing the equivalence between the methods of ISO 13779-3:2008 and this document (ISO 13779-3:2018);

j) DL and QL values as well as the method used for their determination for trace elements and foreign phases content;

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Annex A
(informative)

Contamination of calcium phosphate

Due to manufacturing techniques currently in use, it is possible that undesirable chemicals, other than those cited in this document, contaminate the calcium phosphate. Manufacturers are therefore advised to carry out objective analysis of the risks of contamination due to the various manufacturing processes used within their company or by sub-contractors and, if necessary, to qualify, quantify and set the limits of acceptability for each chemical liable to be a contaminant.

Particular attention should be paid to the limits of the following metals: copper, iron, tungsten (arising during use of the plasma torch) and other types of selective or random contamination.

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Annex B
(normative)

Testing of the purity of the phases used in the production of the calibration curves

B.1 Hydroxyapatite

It is of the utmost importance that the reference hydroxyapatite used throughout the calibration setup is a highly crystalline stoichiometric phase. The hydroxyapatite shall be used in the calcined state [(1 000 ± 25) °C for 15 h].

The hydroxyapatite will be deemed to be acceptable if it meets all of the following conditions:

a) The starting hydroxyapatite will have a Ca:P ratio of 1,667 ± 0,005.

b) The starting powder shall not contain any significant impurities that might affect crystallinity (it is known that magnesium can suppress the formation of highly crystalline hydroxyapatite).

c) The absence of β- and α-tricalcium phosphate is confirmed. For testing, confirm absence of the line 0.2.10 at $d = 2{,}880 \times 10^{-10}$ m for the β-form and 4.4.1, 1.7.0 at $d = 2{,}905 \times 10^{-10}$ m for the α-form on an X-ray diffraction pattern having a resolution ≤0,02° and a signal/noise ratio of the hydroxyapatite (line 2.1.1) greater than or equal to 50.

d) The absence of CaO is tested using X-ray diffraction. The absence of the line 2.0.0 of the CaO at $d = 2{,}405 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal/noise ratio of the hydroxyapatite (line 2.1.1) greater than or equal to 50, indicates the absence of CaO.

e) The absence of TTCP is tested using X-ray diffraction. The absence of the line 0.4.0 of the TTCP at $d = 3{,}00 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal/noise ratio of the hydroxyapatite (line 2.1.1) greater than or equal to 50, indicates the absence of TTCP.

B.2 β-tricalcium phosphate

The β-tricalcium phosphate shall be considered pure if it meets the following conditions.

a) The absence of hydroxyapatite is tested using X-ray diffraction. The absence of the line 2.1.1 of the apatite at $d = 2{,}81 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 0.2.10 of the tricalcium phosphate)/noise ratio ≥30 indicates the absence of hydroxyapatite.

b) The absence of α-TCP is tested using X-ray diffraction. The absence of the line 1.7.0 of the α-TCP at $d = 2{,}905 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 0.2.10 of the tricalcium phosphate)/noise ratio ≥30 indicates the absence of α-TCP.

c) The absence of calcium pyrophosphate is tested, using infrared spectrometry. Record the infrared spectrum; the absence of bands at 757 cm^{-1} and 434 cm^{-1} indicates the absence of the α-form of calcium pyrophosphate and the absence of bands at 1 210 cm^{-1}, 1 185 cm^{-1}, 723 cm^{-1} and 454 cm^{-1} indicates the absence of the β-form of calcium pyrophosphate.

d) The β-tricalcium phosphate is well crystalline as characterised by the peak width of the main peak. The full width half maximum of the $d = 2{,}881 \times 10^{-10}$ m peak (217) shall be not more than 0,20°.

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B.3 Calcium oxide

Calcium oxide shall be considered pure if the pattern conforms to ICDD PDF 4-0777 or IC DD PDF 82-1690 with no foreign peak.

B.4 α-tricalcium phosphate

The α-tricalcium phosphate shall be considered pure if it meets the following two conditions.

a) The absence of hydroxyapatite is tested using X-ray diffraction. The absence of the line 2.1.1 of the apatite at $d = 2{,}881 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 1.7.0 of the α-tricalcium phosphate)/noise ratio ≥30 indicates the absence of hydroxyapatite.

b) The absence of β-TCP is tested using X-ray diffraction. The absence of the line 2.1.4 of the β-TCP at $d = 3{,}21 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 1.7.0 of the α-tricalcium phosphate)/noise ratio ≥ 30 indicates the absence of β-TCP.

B.5 Tetracalcium phosphate

The tetracalcium phosphate shall be considered pure if it meets the following two conditions.

a) The absence of hydroxyapatite is tested using X-ray diffraction. The absence of the line 2.1.1 of the apatite at $d = 2{,}881 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 0.4.0 of the tetracalcium phosphate)/noise ratio ≥30 indicates the absence of hydroxyapatite.

b) The absence of β and α-tricalcium phosphate is confirmed. For testing, confirm absence of the line 0.2.10 at $d = 2{,}88 \times 10^{-10}$ m for the β-form and 1.7.0 at $d = 2{,}905 \times 10^{-10}$ m for the α-form on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 1.7.0 of the tetracalcium phosphate)/noise ratio ≥30 indicates the absence of β-TCP and α-TCP.

c) The absence of CaO is tested using X-ray diffraction. The absence of the line 2.0.0 of the CaO at $d = 2{,}405 \times 10^{-10}$ m on an X-ray diffraction pattern having a resolution ≤0,02° and a signal (line 1.7.0 of the tetracalcium phosphate)/noise ratio ≥30 indicates the absence of CaO.

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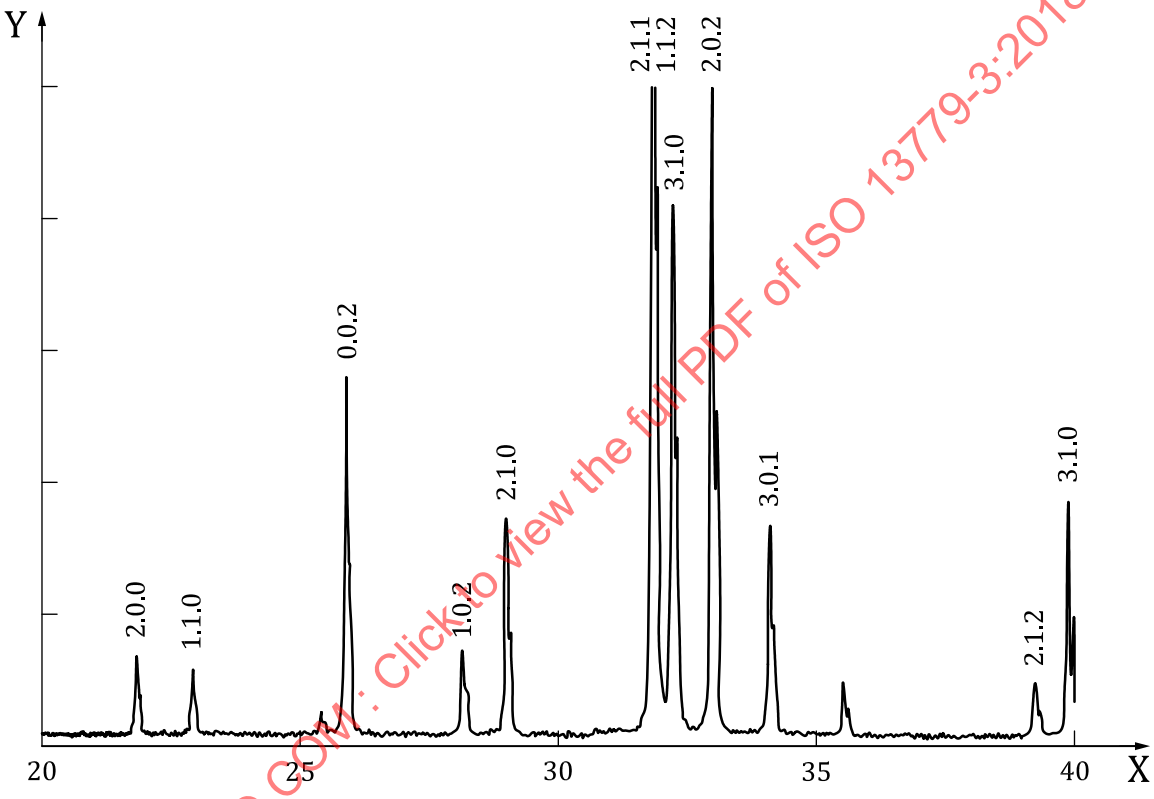
Annex C
(informative)

Examples of X-ray diffraction patterns collected from various mixtures used to plot the calibration curves

NOTE The curves in the Figures C.1 to C.5 are before background subtraction.



Key

X 2θ (deg) for Cu Kα radiation

Y intensity

Figure C.1 — Pure HA

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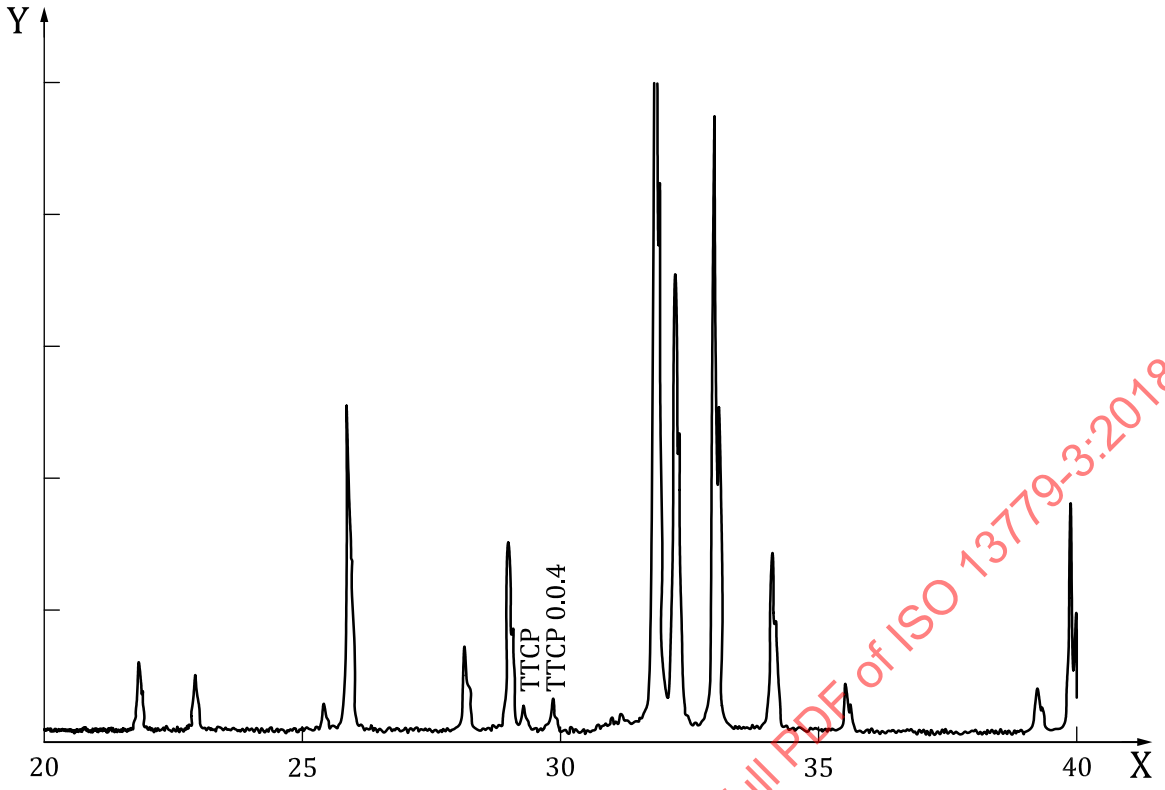

Key

X 2θ (deg) for Cu Kα radiation

Y intensity

Figure C.2 — Mixture of hydroxyapatite and 5 w% TTCP

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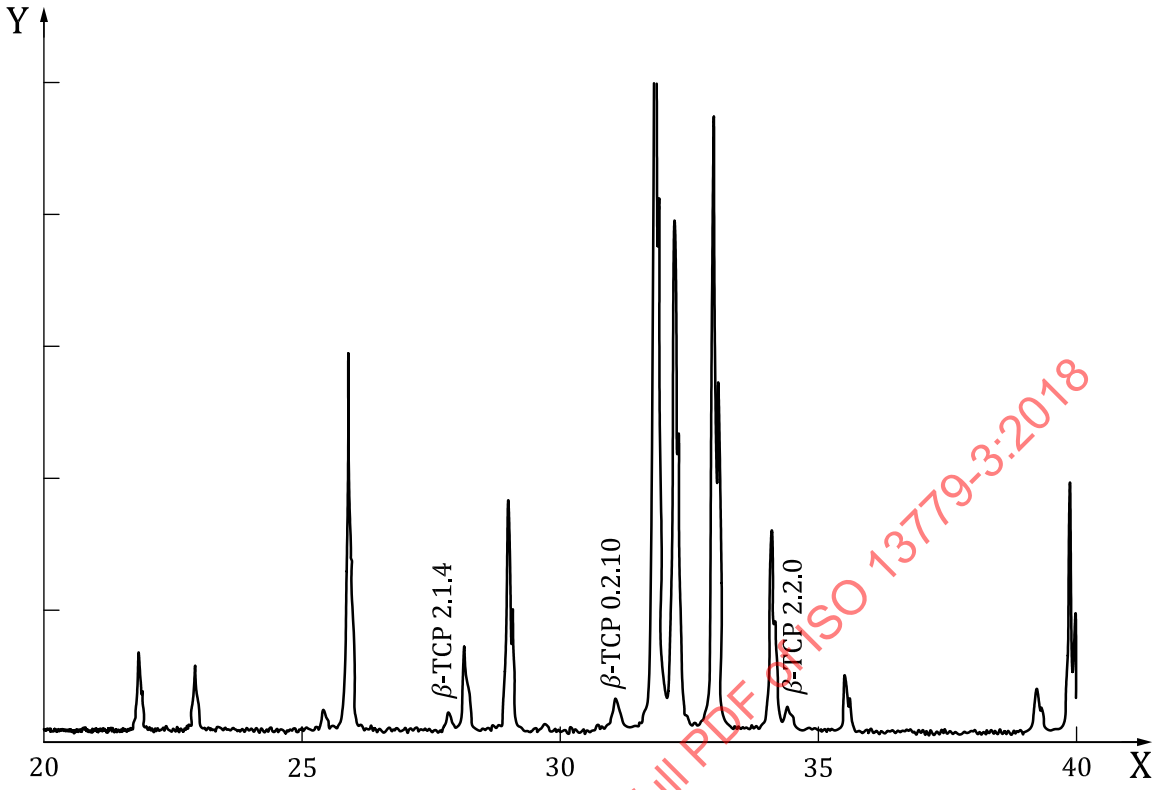

Key

X 2θ (deg) for Cu Kα radiation

Y intensity

Figure C.3 — Mixture of hydroxyapatite and 5 w% β-TCP

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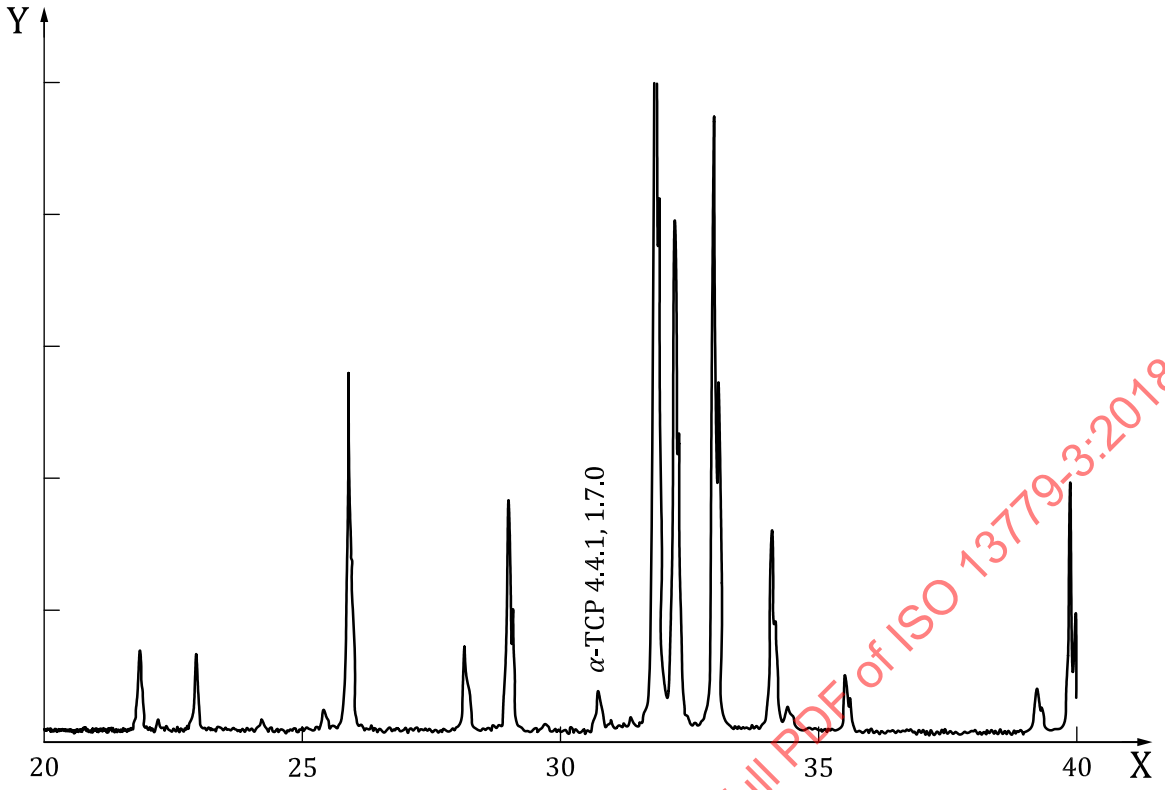

Key

X 2θ (deg) for Cu Kα radiation

Y intensity

Figure C.4 — Mixture of hydroxyapatite and α-tricalcium phosphate (α-TCP) at 5 w%

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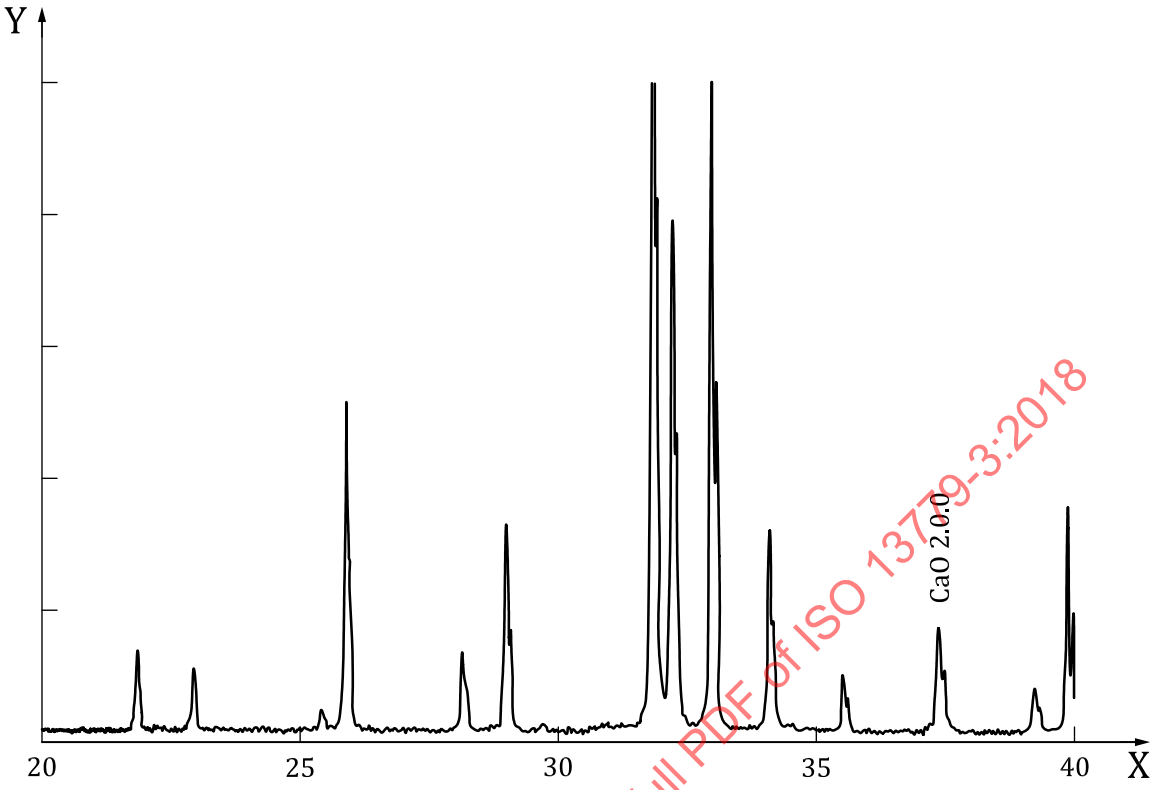

Key

X 2θ (deg) for Cu Kα radiation

Y intensity

Figure C.5 — Mixture of hydroxyapatite and 5 w% CaO

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Annex D
(normative)

Positions of hydroxyapatite lines used to measure the crystallinity ratio

Table D.1 — Position of the 10 lines used to measure the crystallinity ratio

Lines
$d_1 = 3{,}44 \times 10^{-10}$ m
$d_2 = 3{,}17 \times 10^{-10}$ m
$d_3 = 3{,}08 \times 10^{-10}$ m
$d_4 = 2{,}81 \times 10^{-10}$ m
$d_5 = 2{,}78 \times 10^{-10}$ m
$d_6 = 2{,}72 \times 10^{-10}$ m
$d_7 = 2{,}63 \times 10^{-10}$ m
$d_8 = 2{,}26 \times 10^{-10}$ m
$d_9 = 1{,}94 \times 10^{-10}$ m
$d_{10} = 1{,}84 \times 10^{-10}$ m

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Annex E
(normative)

Methods for the preparation of reference materials

E.1 Method of preparation for hydroxyapatite (HA)

Pure and highly crystalline hydroxyapatite can be purchased as commercial powder or prepared by different techniques.

It shall be crushed and sieved so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks.

After crushing and sieving, the powder shall be calcined at (1 000 ± 25) °C for at least 15 h. After cooling, as the calcination might cause agglomeration, the powder shall be re-sieved at 40 µm and any retained powder shall be re-crushed to pass the 40 µm sieve. After this procedure the powder shall be stored away from humidity.

E.2 Method of preparation for the beta-tricalcium phosphate (β-TCP)

Pure β-TCP can be purchased as commercial powder or prepared by different techniques.

It shall be crushed and sieved so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks.

The following procedure shall be carried out unless the β-TCP is already in accordance with B.2 d). After crushing and sieving, the powder shall be calcined at (1 000 ± 25) °C for at least 15 h. After cooling, as the calcination might cause agglomeration, the powder shall be re-sieved at 40 µm and any retained powder shall be re-crushed to pass the 40 µm sieve. After this procedure the powder shall be stored away from humidity.

E.3 Preparation for the tetra-calcium phosphate (TTCP)

E.3.1 Available methods

One of the methods described in E.3.2 to E.3.4 can be used to prepare TTCP reference material.

E.3.2 TTCP purchased as pure commercial powder

Pure TTCP can be purchased as commercial powder.

It shall be crushed and sieved so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks.

The following procedure shall be carried out unless the purchased powder is already in accordance with B.5. After crushing and sieving, the powder shall be calcined at 1 400 ± 25 °C for at least 6 h and immediately quenched, preferably in liquid nitrogen. After quenching, as the calcination might cause agglomeration, the powder shall be re-sieved at 40 µm and any retained powder shall be re-crushed to pass the 40 µm sieve. After this procedure the powder shall be stored away from humidity.

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E.3.3 Example of method for the preparation of TTCP

The following reagents shall be used:

— dicalcium phosphate, (dihydrated: DCPD or anhydrous: DCPA), analytical grade, and

— calcium carbonate, analytical grade,

— liquid nitrogen (optional).

The following apparatus shall be used:

— wide platinum crucible,

— shaker,

— oven, capable of operating at (1 400 ± 25) °C.

The solid state reaction is:

$$2\ CaCO_3 + 2\ CaHPO_4 \rightarrow Ca_4(PO_4)_2O + 2\ CO_2 + H_2O$$

Thoroughly mix equal molar quantities of calcium carbonate and DCPD (or DCPA) powders in the shaker. Place the mixture in the crucible, then place the crucible in the oven. Heat the mixture to (1 400 ± 25) °C and maintain it at that temperature for at least 6 h, preferably under nitrogen atmosphere. Take the crucible out of the oven and quench it in a non-reactive medium, preferably in liquid nitrogen

Crushed and sieve the powder so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks.

After sieving, the powder shall be stored away from humidity.

E.3.4 Other methods for the preparation of TTCP

Pure TTCP can be obtained by other methods. Whatever the method, it shall be crushed and sieved so that the particle size does not exceed 40 µm. Care should be taken not to crush the samples too much as the particle size does have an influence on the width of the diffraction peaks.

The following procedure shall be carried out unless the prepared powder is already in accordance with B.5. After crushing and sieving, the powder shall be calcined at (1 400 ± 25) °C for at least 6 h and immediately quenched, preferably in liquid nitrogen. After quenching, as the calcination might cause agglomeration, the powder shall be re-sieved at 40 µm and any retained powder shall be re-crushed to pass the 40 µm sieve. After this procedure the powder shall be stored away from humidity. [GB110]

E.4 Preparation for the α-tricalcium phosphate (α-TCP)

The following starting powder shall be used:

— β-tricalcium phosphate, (β-TCP) with no detectable Mg2+, Fe2+ and any other bivalent ions impurities susceptible to stabilize the β-TCP,

— liquid nitrogen (optional).

The following apparatus shall be used:

— wide platinum crucible,

— oven, capable of operating at (1 250 ± 25) °C.

The reaction is a phase transition occurring at 1 125 °C.

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