ISO/FDIS 3180:2023(E)

ISO TC 206/WG 3

Date: 2023-~~01~~02-20

Secretariat: JISC

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of calcium-phosphate-based powders for non-biomedical applications

Style Definition: Heading 1: Indent: Left: 0 pt, First line: 0 pt, Tab stops: Not at 21.6 pt

Style Definition: Heading 2: Font: Bold, Tab stops: Not at 18 pt

Style Definition: Heading 3: Font: Bold

Style Definition: Heading 4: Font: Bold

Style Definition: Heading 5: Font: Bold

Style Definition: Heading 6: Font: Bold

Style Definition: ANNEX

Style Definition: ハッシュタグ1

Style Definition: メンション1

Style Definition: スマート ハイパーリンク1

Style Definition: 未解決のメンション1

Style Definition: AMEND Terms Heading: Font: Bold

Style Definition: AMEND Heading 1 Unnumbered: Font: Bold

Style Definition: IneraTableMultiPar: Font color: Auto, French (Switzerland), Adjust space between Latin and Asian text, Adjust space between Asian text and numbers

Formatted: French (Switzerland)

Formatted: French (Switzerland)

Formatted: Footer

Formatted Table

Formatted: Font: 11 pt

Contents

Formatted: Font: 11 pt

Formatted: Footer

Formatted: Footer
Formatted Table
Formatted: Font: 11 pt

Formatted: Font: 11 pt
Formatted: Footer

Formatted: Footer
Formatted Table
Formatted: Font: 11 pt

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.

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~~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~~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 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~~www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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~~www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of calcium-phosphate-based powders for non-biomedical applications

1 Scope

This document specifies wet chemical and inductively coupled plasma–optical emission spectrometer (ICP–OES)-based methods for the chemical analysis of calcium-phosphate-based powders for non-biomedical applications, such as those in the chemical industry, the treatment of air, water and soil contamination.

It stipulates the methods used for the determination of major elements of calcium-phosphate-based powders and their impurities. Calcium-phosphate-based powders are decomposed by acid decomposition. The calcium content is determined using a titration method or an ICP–OES. The phosphorus content is determined using a precipitation and gravimetric method or an ICP–OES. Certain impurities, such as aluminium, barium, chromium, copper, iron, magnesium, manganese, nickel, potassium, selenium, silicon, sodium, strontium, titanium and zinc contents, are determined by an ICP–OES.

This document does not include calcium-phosphate-based powders for biomedical applications. The ISO 13779 series characterizes hydroxyapatite powders for biomedical applications using various methods, such as atomic absorption spectroscopy (AAS), inductively coupled plasma–mass spectroscopy (ICP–MS) and flame atomic absorption spectroscopy (FAAS).

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

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

3 Terms and definitions

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

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

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

— IEC Electropedia: available at ~~https://www.electropedia.org/~~https://www.electropedia.org/

3.1
calcium-phosphate-based powder

Formatted: Font: 11.5 pt
Formatted: Don't adjust space between Latin and Asian text, Don't adjust space between Asian text and numbers
Formatted: Don't adjust space between Latin and Asian text, Don't adjust space between Asian text and numbers
Formatted: English (United Kingdom)
Formatted: Font: Cambria, 11 pt, English (United Kingdom)
Formatted: No underline, Font color: Auto, English (United Kingdom)
Formatted: Don't adjust space between Latin and Asian text, Don't adjust space between Asian text and numbers, Tab stops: 19.85 pt, Left + 39.7 pt, Left + 59.55 pt, Left + 79.4 pt, Left + 99.25 pt, Left + 119.05 pt, Left + 138.9 pt, Left + 158.75 pt, Left + 178.6 pt, Left + 198.45 pt, Left
Formatted: English (United Kingdom)
Formatted: Default Paragraph Font, English (United Kingdom)
Formatted: English (United Kingdom)
Formatted: Font: 11 pt
Formatted: Footer
Formatted Table

calcium phosphate powder with a small amount (not more than 1,0 % mass fraction) of each inorganic element or impurity except calcium, phosphorous, oxygen and hydrogen

Note 1 to entry: Examples of calcium phosphate powders include tricalcium phosphate, octacalcium phosphate and hydroxyapatite.

Formatted: Don't adjust space between Latin and Asian text, Don't adjust space between Asian text and numbers, Tab stops: 19.85 pt, Left + 39.7 pt, Left + 59.55 pt, Left + 79.4 pt, Left + 99.25 pt, Left + 119.05 pt, Left + 138.9 pt, Left + 158.75 pt, Left + 178.6 pt, Left + 198.45 pt, Left

4 Analytical ranges

— Calcium (Ca), range of 30 % to 40 % (mass fraction).

— Phosphorus (P), range of 10 % to 20 % (mass fraction).

— Other analytes, range of 0,000 5 % to 1,0 % (mass fraction).

5 Preparation of test sample

5.1 General

Prepare the sample in accordance with ISO 8656-1, unless otherwise mutually agreed upon by the analyst and customer.

5.2 Sampling

Collect the sample in accordance with ISO 8656-1.

5.3 Drying

Place 10 g of the sample into a flat-type weighing bottle and spread it uniformly at the bottom of the bottle. Place the bottle for 2 h at 110 °C ± 5 °C, then cover the mouth of the bottle and cool it in a desiccator for 1 h.

5.4 Weighing

Weigh the sample to the nearest 0,1 mg of the required quantity using a balance.

6 Reporting analytical values

6.1 Number of analyses

Prepare each sample twice and analyse them at intervals of time.

6.2 Blank test

Upon analysis, perform a blank test to correct the measured values. A double blank digestion is highly recommended for the blank value determination.

6.3 Evaluation of analytical results

When the absolute difference between the two analytical results does not exceed the tolerance (Table 1), the average value shall be reported. When the absolute difference between the two analytical results exceeds the tolerance, perform two additional analyses. When the absolute difference of these further two analyses does not exceed the tolerance, the average value thereof shall be reported. If the difference also exceeds the tolerance, the median of four analytical results shall be reported.

Formatted: Footer

Formatted: Font: 11 pt

Table 1 — Tolerances for two analytical results

Unit: % (mass fraction)

Analyte	Range of results	Tolerance
Ca, P	–	0,1
Al, Ba, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Se, Si, Sr, Ti, Zn	Less than 0,01 % Not less than 0,01 %, and less than 0,1 % Not less than 0,1 %	0,001 0,005 0,01

6.4 Expression of analytical results

Express the analytical results in % (mass fraction), in dryness.

a) Calcium and phosphorus: express the results to four significant digits, as required.

b) Others: express the results to four decimal places.

7 Decomposition of test sample

7.1 Reagents

It shall be ascertained that the reagents are of sufficiently high purity to permit their use without compromising the accuracy of the determination.

7.1.1 Water, grade 1 or superior, as specified in ISO 3696.

7.1.2 Nitric acid (HNO_3), 65 % min., as specified in ISO 6353-2 (R 19).

7.2 Apparatus

Use ordinary laboratory apparatus.

7.3 Procedure

7.3.1 Decomposition

Weigh 1,0 g of the test sample and transfer it to a 250 ml beaker. Add 10 ml of nitric acid (7.1.2) to the beaker. Cover the beaker with a watch-glass and heat it at 180 °C ± 5 °C on a hot plate until the test sample dissolves completely. Remove the beaker from the hot plate and cool it to room temperature.

If the precipitate falls out of the solution after the decomposition procedure, an additional process (e.g. alkali fusion method) is necessary for decomposing the insoluble salt (see Annex A).

7.3.2 Dilution

After cooling, transfer the solution to a 250 ml volumetric flask. Rinse the inner wall of the beaker and the watch-glass with a small quantity of water and put the washings into the flask. Dilute with water up to the mark and mix well. This solution is designated the sample solution.

7.4 Blank test

Perform the operation described in 7.3 without sample. The resulting solution is designated as blank solution.

Formatted: std_publisher
Formatted: std_docNumber
Formatted: Font: 11 pt
Formatted: Footer