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

iTeh STA *Céramiques techniques — Méthodes d'analyse chimique des poudres à
base de phosphate de calcium pour applications non biomédicales*
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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 document 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).

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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.

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 spectrometry (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 method. The phosphorus content is determined using a precipitation and gravimetric method or an ICP–OES method. 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 method.

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 spectrometry (AAS), inductively coupled plasma–mass spectrometry (ICP–MS) and flame atomic absorption spectrometry (FAAS).

2 Normative references

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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>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

calcium-phosphate-based powder

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.

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 $110\text{ °C} \pm 5\text{ °C}$, 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.

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 %	0,001
	Not less than 0,01 %, and less than 0,1 %	0,005
	Not less than 0,1 %	0,01

6.4 Expression of analytical results

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

- Calcium and phosphorus: express the results to four significant digits, as required.
- 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 (HNO₃), 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.

8 Determination of phosphorus and calcium contents

8.1 Classification of determination methods

Method A: Precipitation and gravimetric method (for phosphorus).

Method B: Titration method (for calcium).

Method C: ICP-OES method (for phosphorus and calcium).

Analytical results of interlaboratory study for chemical analysis of calcium-phosphate-based powder are described in [Annex B](#).

8.2 Precipitation and gravimetric method (for phosphorus)

8.2.1 Principle

Phosphorus (P) in the sample solution is precipitated as quinoline phosphomolybdate $[(C_9H_7NH)_3PO_4 \cdot 12MoO_3]$ by the addition of citromolybdate quinoline solution. After filtering, the mass of the precipitate is measured. The content of phosphorus is calculated from the mass of the quinoline phosphomolybdate.

8.2.2 Reagents

Use the reagents described in [7.1](#) together with the following.

8.2.2.1 Citromolybdate quinoline solution.

Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4 \cdot 2H_2O$) in 150 ml of water in a 250 ml beaker (solution A).

Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in 150 ml of water in a 250 ml beaker, then add 85 ml of nitric acid (solution B).

Pour solution A and solution B into a 1 000 ml beaker and mix well (solution C).

Add 35 ml of nitric acid and 5 ml of recently distilled quinoline (C_9H_7N) to 100 ml of water in a 250 ml beaker (solution D).

Pour solution C and solution D into a 1 000 ml beaker and mix well. Leave the solution undisturbed for at least 12 h. Filter the solution using a filter paper. Add 280 ml of acetone (C_3H_6O) to the filtrate and transfer it to a 1 000 ml volumetric flask. Dilute with water up to the mark and mix well.

Store the solution protected from light in a well-stoppered flask. Do not keep the solution for more than 1 week.

8.2.3 Apparatus

Use ordinary laboratory apparatus together with the following:

8.2.3.1 Sintered glass filter crucible, of porosity P10 (pore size between 4 μm and 10 μm).

Heat the filter crucible in an air bath controlled at $220 \text{ }^\circ\text{C} \pm 20 \text{ }^\circ\text{C}$ and leave for 15 min after the temperature stabilizes. Cool to room temperature in a desiccator containing silica gel.

8.2.3.2 Air bath, capable of heating at $250 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$.

8.2.4 Procedure

Transfer 25,0 ml of the sample solution (7.3.2) into a 250 ml beaker. Add 100 ml of citromolybdate quinoline solution (8.2.2.1) and heat the beaker on a hot plate at $75\text{ °C} \pm 5\text{ °C}$ for 1 h. Remove the beaker from the hot plate. Cool the solution to room temperature, stirring three or four times with a glass rod.

Decant the solution through the filter crucible (8.2.3.1). Store the filtrates and the washings for the measurement of calcium content. This solution is designated the sample test solution for the titration (8.3).

Wash the precipitate six times with approximately 30 ml of water each time. Place the filter crucible and the precipitate in the air bath (8.2.3.2) at $85\text{ °C} \pm 5\text{ °C}$ until completely dry. Cool to room temperature in a desiccator and weigh to the nearest 0,1 mg.

8.2.5 Blank test

Perform the procedure described in 8.2.4 with the blank solution (7.4).

8.2.6 Calculation

Calculate phosphorus content, W_p , expressed as a percentage mass fraction, using Formula (1).

$$W_p = \frac{(m_S - m_B)}{m} \times F \times \frac{250}{25} \times 100 \quad (1)$$

where

W_p is the phosphorus content, in per cent (mass fraction);

m is the mass of the test sample (7.3), in g;

m_S is the mass of the precipitate in the sample test solution (8.2.4), in g;

m_B is the mass of the precipitate in the blank test solution (8.2.5), in g;

F is the conversion factor from quinoline phosphomolybdate $[(C_9H_7NH)_3PO_4 \cdot 12MoO_3]$ to phosphorus (P) (= 0,014).

8.3 Titration method (for calcium)

8.3.1 Principle

Calcium (Ca) in the sample solution is precipitated as calcium oxalate (CaC_2O_4) by the addition of ammonium oxalate solution. After filtering, the solution is titrated with potassium permanganate. The content of calcium is calculated from the volume of the titrant at the endpoint.

8.3.2 Reagents

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

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

8.3.2.2 Ammonium chloride (NH_4Cl), 99,5 % min., as specified in ISO 6353-2 (R 5).

8.3.2.3 Ammonia solution (NH_4OH), 25 % min., as specified in ISO 6353-2 (R 3).

8.3.2.4 Ammonium oxalate ($C_2H_8N_2O_4$).