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

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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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

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 °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.

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.

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.

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 °C ± 20 °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 °C ± 5 °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 °C ± 5 °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 °C ± 5 °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).

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

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 ($C_2H_8N_2O_4$).

8.3.2.5 Ammonium oxalate solution. Dissolve 50 g of ammonium oxalate (8.3.2.4) in 500 ml water in a 1 000 ml volumetric flask. Dilute with water (8.3.2.1) up to the mark and mix well.

8.3.2.6 Sodium oxalate ($Na_2C_2O_4$).

8.3.2.7 Sodium oxalate solution (0,025 mol/l). Dissolve 3,35 g of sodium oxalate (8.3.2.6) in 200 ml water in a 1 000 ml volumetric flask. Dilute it with water (8.3.2.1) up to the mark and mix well.

8.3.2.8 Potassium permanganate ($KMnO_4$).

8.3.2.9 Potassium permanganate solution. Dissolve 1,58 g of potassium permanganate (8.3.2.8) in 1 000 ml of water (8.3.2.1) and leave undisturbed for at least 24 h.

Filter the solution using a filter paper and store, protected from light, in a well-stoppered flask. The solution should be standardized by titration against sodium oxalate solution before use. The standardization procedure is as follows:

Place 25 ml of sodium oxalate solution (8.3.2.7) and 100 ml of hot water in a 500 ml beaker. After adding 50 ml of sulfuric acid solution (8.3.2.11), place the beaker on a magnetic stirrer and keep the solution at 80 °C ± 5 °C. While stirring, titrate the solution by adding the prepared potassium permanganate solution until the colour of solution turns pale pink. Titrate at least three times and calculate the average volume at the endpoints. Calculate the concentration of the potassium permanganate solution according to Formula (2).

$$C = 0{,}074\ 627 \times \frac{m_{\mathrm{so}}}{V} \qquad (2)$$

where

C is the concentration of the potassium permanganate solution, in mol/l;

m_{so} is the mass of the sodium oxalate used for the sodium oxalate solution (8.3.2.7), in g;

V is the average volume of the potassium permanganate solution at the endpoints, in ml.

8.3.2.10 Sulfuric acid (H_2SO_4), 95 % min. as specified in ISO 6353-2 (R 37).

8.3.2.11 Sulfuric acid solution (1 + 4). One volume of sulfuric acid (8.3.2.10) is mixed with four volumes of water.

8.3.3 Apparatus

Use ordinary laboratory apparatus together with the following.

8.3.3.1 Filter paper, pore size between 4 µm and 10 µm.

8.3.3.2 Magnetic stirrer, with a hot plate.

8.3.3.3 Burette.

8.3.4 Procedure

Transfer sample test solution, obtained in 8.2.4, into a 500 ml beaker and add 1 g of ammonium chloride (8.3.2.2) and 50 ml of the ammonium oxalate solution (8.3.2.5). Adjust pH to between 10 and 11 using the ammonia solution (8.3.2.3). Heat the beaker on a hot plate at 180 °C ± 5 °C for 30 min. Remove the beaker from the hot plate and leave it undisturbed until the supernatant becomes clear.

Filter the solution using a filter paper (8.3.3.1) and then wash the precipitate three times with approximately 30 ml of hot water each time. Carefully attach the filter paper to the inner wall of a 500 ml beaker and then wash the precipitate down with 150 ml of hot water.

After adding 50 ml of sulfuric acid solution (8.3.2.11), place the beaker on a magnetic stirrer(8.3.3.2) and keep the solution at 80 °C ± 5 °C. While stirring, titrate the solution by adding potassium permanganate solution (8.3.2.9) until the colour of solution turns pale pink. After dropping the filter paper attached on the inner wall of the beaker to the bottom, check the colour of the solution. If the solution returns colourless, continue to titrate until the solution turns pale pink, persisting for 30 s. Record the final volume of the titrant as the endpoint.

8.3.5 Blank test

Perform the procedure described in 8.3.4 with the blank test solution (8.2.5).

8.3.6 Calculation

Calculate calcium content, W_{Ca}, expressed as a percentage mass fraction, using Formula (3).

$$W_{\mathrm{Ca}} = 0{,}100\ 195 \times C \times \frac{(V_{\mathrm{S}} - V_{\mathrm{B}})}{m} \times \frac{250}{25} \times 100 \tag{3}$$

where

W_{Ca} is the calcium content, in % (mass fraction);

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

V_{S} is the volume of the titrant at the endpoint in the sample test solution (8.3.4), in ml;

V_{B} is the volume of the titrant at the endpoint in the blank test solution (8.3.5), in ml;

C is the concentration of the potassium permanganate solution, in mol/l (8.3.2.9).

8.4 ICP–OES method (for calcium and phosphorus)

8.4.1 Principle

A portion of the sample solution with an internal standard is sprayed into the plasma of an ICP–OES and each emission intensity of calcium and phosphorus is measured at the selected wavelength. The contents of calcium and phosphorus in the sample solution are calculated by the concentration ratios of the analyte elements and the spiked internal standard element in comparison with those in the standard solutions.

8.4.2 Reagents

It shall be ascertained that the reagents are of sufficiently high purity to permit their use without compromising the accuracy of the determination. The reagents should be preserved in plastic bottles.

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

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

8.4.2.3 Nitric acid solution (1 + 50). One volume of nitric acid (8.4.2.2) is mixed with 50 volumes of water.

8.4.2.4 Calcium standard solution (Ca 1 mg/ml). The SI traceable commercial standard solution is available.

8.4.2.5 Phosphorus standard solution (P 1 mg/ml). The SI traceable commercial standard solution is available.

8.4.2.6 Lanthanum standard solution (La 1 mg/ml). The SI traceable commercial standard solution is available. Other element standard solution (e.g. yttrium standard solution) should be used after validation.

8.4.2.7 Internal standard solution (La 0,02 mg/ml). Transfer 5,0 ml of lanthanum standard solution (8.4.2.6) to a 250 ml volumetric flask, dilute with nitric acid solution (8.4.2.3) up to the mark and mix well.

8.4.3 Apparatus

Use ordinary laboratory apparatus, along with an ICP–OES.

ICP–OES is used to determine elements in solution. The solution is dispersed by a suitable nebulizer and the resulting aerosol is transported into the plasma. In a radio-frequency inductively coupled plasma the solvent is evaporated and the dried salts are then vaporized, dissociated, atomized and ionized.

The atoms or ions are excited thermally and the number of photons emitted during transition to a lower energy level is measured with optical emission spectrometry. The spectrum is dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photosensitive devices. The identification of the element takes place by means of the wavelength of the radiation (energy of photons), while the concentration of the element is proportional to the intensity of the radiation (number of photons).

The ICP–OES method can be used to perform multi-element determinations using an optical system. The ICP–OES system shall be capable of simultaneously measuring the analyte emission wavelengths and the emission wavelength of the internal standard with a minimum optical resolution of 0,02 nm.

8.4.4 Procedure

8.4.4.1 Preparation of sample test solution

Transfer 5,0 ml of the sample solution (7.3.2) to a 200 ml volumetric flask and add 20 ml of internal standard solution (8.4.2.7). Dilute with nitric acid solution (8.4.2.3) up to the mark and mix well. This solution is designated the sample test solution.

8.4.4.2 Preparation of calibration standard solutions

Transfer an appropriate portion of calcium standard solution (8.4.2.4) and phosphorus standard solution (8.4.2.5) into 100 ml volumetric flasks and add 10 ml of internal standard solution (8.4.2.7).

Dilute each flask with nitric acid solution (8.4.2.3) up to the mark and mix well. These solutions are designated the calibration standard solutions.

Prepare the solutions freshly before every use.

An example of the calibration standard solution set is shown in Table 2.

Table 2 — Example of the calibration standard solutions for major elements

Standard solution	Solution 1 ml	Solution 2 ml	Solution 3 ml	Solution 4 ml
Calcium standard solution (Ca 1 mg/ml)	0	1	3	6
Phosphorus standard solution (P 1 mg/ml)	0	1	2	3
Internal standard solution (La 0,02 mg/ml)	10	10	10	10
Nitric acid solution (8.4.2.3)	90	88	85	81

8.4.4.3 Preparation of blank test solution

Perform the procedure described in 8.4.4.1 with the blank solution (7.4). This solution is designated the blank test solution.

8.4.5 Measurement

8.4.5.1 Set up of the instruments

Set up the ICP–OES in accordance with the manufacturer's instructions and choose appropriate background correction positions. A clean torch, spray chamber and sample uptake tubes shall be used and the plasma shall be stabilized before use, following the recommendations of the instruments' manufacturer. The data-processing unit of the ICP–OES is used to establish a measuring programme in which the intensities of the analyte emission wavelengths and the internal standard emission wavelengths can be measured simultaneously.

8.4.5.2 Measurement of sample test solution and calibration standard solutions

Measure the emission intensity of each element in the sample test solution and the calibration standard solutions at an appropriate wavelength (Table 3). Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

Each solution shall be measured at least five times.

Table 3 — Examples of the analytical wavelength for calcium and phosphorus

Element	Wavelength 1 nm	Wavelength 2 nm
Ca	317,933	315,887
P	213,617	177,434
La (internal standard)	379,749	408,672

8.4.5.3 Measurement of blank test solution

Perform the procedure described in 8.4.5.2 with the blank test solution (8.4.4.3).

8.4.6 Drawing of calibration curve

The method of internal standardization is based on the linear relation between the intensity ratios of the analyte element and internal standard element, and the concentration ratios of the analyte element and internal standard element.

The data-processing unit provides the quotients from the simultaneously registered single measurements of the intensities of the analyte element and internal standard element. Calculate the intensity ratio of the analyte element and internal standard element of each calibration standard solution using Formula (4).

$$R_\mathrm{I} = \frac{I_\mathrm{A}}{I_\mathrm{IS}} \tag{4}$$

where

R_I is the intensity ratio of the analyte element and internal standard element;

I_A is the net emission intensity of the analyte element;

I_IS is the net emission intensity of the internal standard element.

The relative standard deviation (RSD) of the intensity ratio values (R_I) of the analyte element and internal standard element during repeat measurement shall not be larger than 0,3 %.

The mean intensity ratio of the analyte element and internal standard element during repeat measurement of each solution is calculated using Formula (5).

$$\bar{R}_\mathrm{I} = \frac{1}{n}\sum_{i=1}^{n} R_{\mathrm{I,\,i}} \tag{5}$$

where

$\bar{R}_\mathrm{I}$ is the mean intensity ratio of the analyte element and internal standard element;

n is the number of measurements.

The calibration curve for each element is constructed using linear regression with the mean intensity ratios of the analyte element and internal standard element, and their corresponding concentration ratios of the analyte element and internal standard element in the calibration standard solutions.

8.4.7 Calculation

Using the linear regression curve (8.4.6), calculate the concentration ratio (R_C) of each analyte element and internal standard element in the sample test solution and the blank test solution.

From the obtained concentration ratio (R_C), calculate the concentration of each analyte element in the sample test solution (C_A) and the blank test solution (C_B) using Formula (6).

$$C_{\mathrm{A\ or\ B}} = R_\mathrm{C} \times C_\mathrm{IS} \tag{6}$$

where

R_C is the concentration ratio of the analyte element and internal standard element;

C_IS is the net concentration of the internal standard element.

Calculate the content of each analyte element of the sample using Formula (7). This will be done by using the appropriate software functions of the ICP spectrometer.

$$W_\mathrm{Ei} = \frac{(C_\mathrm{A} - C_\mathrm{B})}{m} \tag{7}$$

where

W_{Ei} is each element content, in per cent (mass fraction);

C_{A} is the concentration of analyte element in the sample test solution (8.4.4.1), in mg/l;

C_{B} is the concentration of analyte element in the blank test solution (8.4.4.3), in mg/l;

m is the mass of the test sample (7.3.1), in g.

9 Determination of impurity elements

9.1 Principle

A portion of the sample solution mixed with an internal standard is sprayed into the plasma of an ICP–OES and the emission intensity of each analyte element is measured at the selected wavelength. The contents of analyte elements in the sample solution are calculated by the concentration ratios of the analyte elements and the spiked internal standard element in comparison with those in the standard solutions.

Analytical results of interlaboratory study for chemical analysis of calcium-phosphate-based powder are described in Annex B.

9.2 Reagents

It shall be ascertained that the reagents are of sufficiently high purity to permit their use without compromising the accuracy of the determination. The reagents should be preserved in plastic bottles.

Use the reagents described in 8.4.2 as well as the following.

9.2.1 Calcium standard solution (Ca 10 mg/ml). The SI traceable commercial standard solution is available.

9.2.2 Phosphorus standard solution (P 10 mg/ml). The SI traceable commercial standard solution is available.

9.2.3 Elemental standard solutions. The SI traceable commercial standard solution is available.

- Aluminium standard solution (Al 1 mg/ml).
- Barium standard solution (Ba 1 mg/ml).
- Chromium standard solution (Cr 1 mg/ml).
- Copper standard solution (Cu 1 mg/ml).
- Iron standard solution (Fe 1 mg/ml).
- Magnesium standard solution (Mg 1 mg/ml).
- Manganese standard solution (Mn 1 mg/ml).
- Nickel standard solution (Ni 1 mg/ml).
- Potassium standard solution (K 1 mg/ml).
- Selenium standard solution (Se 1 mg/ml).
- Silicon standard solution (Si 1 mg/ml).

— Sodium standard solution (Na 1 mg/ml).

— Strontium standard solution (Sr 1 mg/ml).

— Titanium standard solution (Ti 1 mg/ml).

— Zinc standard solution (Zn 1 mg/ml).

9.2.4 Mixed standard solution (each element 50 mg/l). Add 5,0 ml each of the solutions (9.2.3) in a 100 ml plastic volumetric flask. Dilute with nitric acid solution (8.4.2.3) up to the mark and mix well. Pay attention to ensure that no precipitation occurs during mixing, because some elements can be precipitated depending on pH. If the precipitate falls out after adding an elemental standard solution, dilute it separately.

Prepare fresh solution before every use.

9.3 Apparatus

Use the apparatus described in 8.4.3.

9.4 Procedure

9.4.1 Preparation of sample test solutions

Transfer 50,0 ml of the sample solution (7.3.2) to a 100 ml volumetric flask and add 10 ml of internal standard solution (8.4.2.7). Dilute with nitric acid solution (8.4.2.3) up to the mark and mix well. This solution is designated the sample test solution.

9.4.2 Preparation of calibration standard solutions

Considering the calcium and phosphorus contents in the sample solution (clause 8), transfer appropriate aliquot of the calcium standard solution (9.2.1), phosphorus standard solution (9.2.2) and mixed standard solution (9.2.4) into three 100 ml plastic volumetric flasks. Add 5,0 ml of internal standard solution (8.4.2.7) to each of the flasks.

Dilute each solution with nitric acid solution (8.4.2.3) up to the mark and mix well.

Prepare fresh solutions before every use.

An example is shown in Table 4.

Table 4 — Example of the calibration solutions for impurity elements

Standard solution	Solution 1 ml	Solution 2 ml	Solution 3 ml
Calcium standard solution (Ca 10 mg/ml)	8	8	8
Phosphorus standard solution (P 10 mg/ml)	4	4	4
Mixed standard solution (Each element 50 mg/l)	0	5	50
Internal standard solution (La 0,02 mg/ml)	5	5	5
Nitric acid solution (8.4.2.3)	83	78	33

9.4.3 Preparation of blank test solution

Perform the procedure described in 9.4.1 with the blank solution (7.4). This solution is designated the blank test solution.

9.5 Measurement

9.5.1 Set up of the instrument

Set up the ICP–OES in accordance with the manufacturer's instructions and choose appropriate background correction positions. A clean torch, spray chamber and sample uptake tubes shall be used and the plasma shall be stabilized before use, following the recommendations of the instrument manufacturer. The data-processing unit of the ICP–OES is used to establish a measuring programme in which the intensities of the analyte emission wavelengths and the internal standard emission wavelengths can be measured simultaneously.

9.5.2 Measurement of sample test solution and calibration standard solutions

Measure the emission intensity of each element in the sample test solution and the calibration standard solutions at an appropriate wavelength (see Table 5). Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

Each solution shall be measured at least five times.

Table 5 — Examples of the analytical wavelength for each element

Element	Wavelength 1 nm	Wavelength 2 nm
Al	396,153	308,215
Ba	233,527	230,425
Cu	327,393	224,700
Cr	267,716	205,560
Fe	259,939	234,830
K	766,490	–
Mg	285,213	279,077
Mn	259,372	260,568
Na	589,592	–
Ni	231,604	232,003
Se	196,026	–
Si	251,611	288,158
Sr	460,733	232,235
Ti	336,121	337,279
Zn	206,200	202,548
La (internal standard)	379,749	408,672

9.5.3 Measurement of blank test solution

Perform the procedure described in 9.5.2 with the blank test solution (9.4.3).

9.6 Drawing of the calibration curve

According to 8.4.6, the calibration curve for each analyte element is constructed using linear regression with concentration of the analyte element in the calibration standard solutions and the corresponding concentration ratio (R_C) of analyte element and internal standard element.

9.7 Calculation

Using the linear regression curve (9.6), calculate the concentration ratio (R_C) of each analyte element and internal standard element in the sample test solution and the blank test solution.

From the obtained concentration ratio (R_C), calculate the concentration of each analyte element in the sample test solution (C_A) and blank test solution (C_B) using Formula (6).

Calculate the content of each analyte element of the sample using Formula (8). This can be done using the appropriate software functions of the ICP spectrometer.

$$W_{Ei} = \frac{\left(C_A - C_B\right)}{m} \times 250 \times \frac{100}{50} \times 10^{-4} \qquad (8)$$

where

W_{Ei} is each element content, in per cent (mass fraction);

C_A is the concentration of each element in the sample test solution (9.4.1), in mg/l;

C_B is the concentration of each element in the blank test solution (9.4.3), in mg/l;

m is the mass of the test sample (7.3.1), in g.

10 Test report

The test report shall contain, as a minimum, the following information:

a) all information necessary for the identification of the sample, laboratory and date of analyses;

b) the method used, by reference to this document, i.e. ISO 3180:2023;

c) the results and the form in which they are expressed;

d) any deviations from the specified procedure;

e) any unusual features noted during the determination;

f) any procedure not specified in this document or any optional procedure that could possibly impact the results.