INTERNATIONAL STANDARD

ISO 23739

First edition
2021-08

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of zirconium oxide powders

Céramiques techniques — Méthodes pour l'analyse chimique des poudres d'oxyde de zirconium

Reference number
ISO 23739:2021(E)

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Published in Switzerland

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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).

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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 zirconium oxide powders

1 Scope

This document specifies methods for the chemical analysis of zirconium oxide powders used as the raw material for fine ceramics.

It stipulates the determination methods of the zirconium, aluminium, barium, calcium, cerium, cobalt, gadolinium, hafnium, iron, magnesium, potassium, silicon, sodium, strontium, titanium and yttrium contents in zirconium oxide powders for fine ceramics. The test sample is decomposed by acid pressure decomposition or alkali fusion. Contents of zirconium and yttrium are determined by using either a precipitation and gravimetric method or an inductively coupled plasma-optical emission spectrometry (ICP–OES) method. Contents of aluminium, barium, calcium, cerium, cobalt, gadolinium, hafnium, iron, magnesium, potassium, silicon, sodium, strontium and titanium are determined by using an ICP–OES method.

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 835, *Laboratory glassware — Graduated pipettes*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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 http://www.electropedia.org/

4 Analytes and ranges

— Zirconium (Zr), range of 60 % to 74 % (mass fraction).

— Aluminium (Al), range of 0,01 % to 0,5 % (mass fraction).

— Barium (Ba), range of 0,01 % to 0,5 % (mass fraction).

— Calcium (Ca), range of 0,01 % to 6 % (mass fraction).

— Cerium (Ce), range of 0,01 % to 0,5 % (mass fraction).

— Cobalt (Co), range of 0,01 % to 0,5 % (mass fraction).

— Gadolinium (Gd), range of 0,01 % to 0,5 % (mass fraction).

— Hafnium (Hf), range of 0,01 % to 2 % (mass fraction).

— Iron (Fe), range of 0,01 % to 0,5 % (mass fraction).

— Magnesium (Mg), range of 0,01 % to 6 % (mass fraction).

— Potassium (K), range of 0,01 % to 0,5 % (mass fraction).

— Sodium (Na), range of 0,01 % to 0,5 % (mass fraction).

— Silicon (Si), range of 0,01 % to 0,5 % (mass fraction).

— Strontium (Sr), range of 0,01 % to 0,5 % (mass fraction).

— Titanium (Ti), range of 0,01 % to 0,5 % (mass fraction).

— Yttrium (Y), range of 0,01 % to 15 % (mass fraction).

5 Preparation of the test sample

5.1 General

The sample preparation method shall be in accordance with ISO 8656-1, unless otherwise mutually agreed upon by the analyser and customer.

5.2 Sampling

The sample shall be collected in accordance with ISO 8656-1.

5.3 Drying

Place 10 g of the sample into a flat-type weighing bottle (60 mm × 30 mm) and spread it uniformly over the bottom of the bottle. Place the bottle in an air bath at 110 °C ± 5 °C for 2 h, uncovered, and cool in a desiccator, covered, for 1 h.

5.4 Weighing

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

6 Reporting the analytical values

6.1 Number of analyses

Analyse the test sample twice on different days.

6.2 Blank test

Upon analysis, perform a blank test to correct the measured values.

6.3 Evaluation of the analytical values

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

Table 1 — Tolerances for the analytical values

Units: % (mass fraction)

Component	Zr	Ca, Hf, Mg, Y	Al, Ba, Ce, Co, Gd, Fe, K, Na, Si, Sr, Ti
Tolerance	0,70	0,01 [a] 0,1 [b]	0,01
[a] Applicable to content of less than 0,1 %.			
[b] Applicable to content of not less than 0,1 %.			

6.4 Expression of analytical values

The analytical values shall be given in % (mass fraction) in dryness. The results shall be expressed to two decimal places (see Annex A).

7 Decomposition of the test sample

7.1 Classification of the sample decomposition methods

a) Acid pressure decomposition.

b) Alkali fusion, for the determination of the contents of major elements such as zirconium, calcium, hafnium, magnesium and yttrium, and also for the determination of silicon content.

7.2 Acid pressure decomposition

7.2.1 Reagents

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

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

7.2.1.2 Sulfuric acid (1+1).

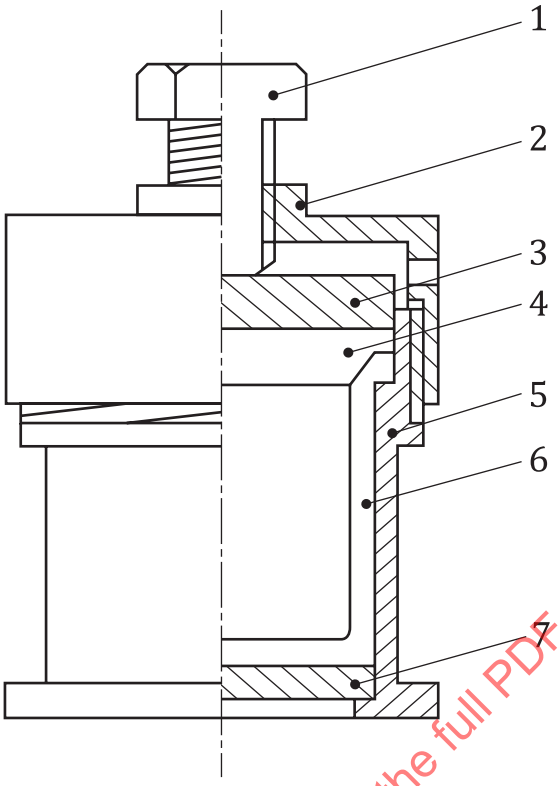
7.2.2 Apparatus and instruments

Use ordinary laboratory apparatus and instruments together with the following:

7.2.2.1 Pressure decomposition vessel. A pressure decomposition vessel is shown in Figure 1. Use the vessel exclusively for this analysis to avoid cross-contamination.

7.2.2.2 Polytetrafluoroethylene (PTFE) bottle, with cap.

7.2.2.3 Air bath, capable of heating at 230 °C ± 5 °C.



Key

1 centre screw
2 screw cap
3 top plate
4 PTFE cap
5 cylinder
6 PTFE bottle
7 bottom plate

Figure 1 — Example of a pressure decomposition vessel

7.2.3 Procedure

Weigh 0,2 g of the test sample in a polytetrafluoroethylene bottle (7.2.2.2) and add 10 ml of sulfuric acid (1+1). Put the PTFE bottle into a pressure decomposition vessel (7.2.2.1) and close the vessel according to the manufacturer's instructions. Place the vessel in an air bath and heat it at 230 °C ± 5 °C for 16 h.

After cooling, disassemble the vessel and transfer the dissolved solution to a 150-ml beaker. Wash the bottle six times with approximately 10 ml of warm water each time and collect the washings into the beaker. Transfer the solution into a 200-ml volumetric flask, dilute it with water up to the mark and mix well. This solution is designated as the sample solution.

7.2.4 Blank test

Perform the operation described in 7.2.3 without taking a sample to obtain the blank test value. The resulting solution is designated as blank test solution.

7.3 Alkali fusion

7.3.1 Reagents

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

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

7.3.1.2 Lithium tetraborate ($Li_2B_4O_7$), powdery, more than 99,995 % purity by trace metal basis.

Some commercial products of lithium tetraborate can contain certain impurities such as calcium, potassium, silicon and sodium. Check their conformity with the test before use.

7.3.1.3 Hydrochloric acid (35 %, mass fraction).

7.3.2 Apparatus and instruments

Use ordinary laboratory apparatus and instruments together with the following:

7.3.2.1 Platinum crucible (30 ml), heated at 1 200 °C ± 50 °C for 15 min, and then cooled down to room temperature in a desiccator.

7.3.2.2 Platinum lid.

7.3.2.3 Electric furnace, capable of being operated at 1 300 °C.

7.3.2.4 Ultrasonic bath.

7.3.3 Procedure

Mix 0,2 g of the test sample and 1,0 g of lithium tetraborate (7.3.1.2) in a platinum crucible (7.3.2.1). Add 1,0 g of lithium tetraborate to the mixed sample. After covering the crucible with a platinum lid (7.3.2.2), place the crucible in an electric furnace (7.3.2.3).

Raise the temperature of the furnace gradually and heat the crucible at 1 200 °C ± 50 °C until the contents are completely decomposed.

Remove the crucible from the furnace and cool it to room temperature.

Put the crucible and the platinum lid into a 150-ml beaker containing 20 ml of hydrochloric acid (7.3.1.3) and 100 ml of water (7.3.1.1). Covering the beaker with a watch glass, warm the beaker in an ultrasonic bath (7.3.2.4) until the melt is completely dissolved into the solution.

Remove the beaker from the ultrasonic bath. Wash the watch glass, the crucible and the platinum lid several times with approximately 50 ml of water and collect the washings into the beaker. After cooling, transfer the solution into a 200-ml volumetric flask, dilute it with water up to the mark and mix well. This is designated as the sample solution.

7.3.4 Blank test

Perform the operation described in 7.3.3 without taking a sample to obtain the blank test value. The resulting solution is designated as blank test solution.

8 Determination of the zirconium content

8.1 Precipitation and gravimetric method

8.1.1 Principle

Zirconium with hafnium in the sample solution is precipitated by adding DL-mandelic acid solution. After igniting the precipitate, the mass of the zirconium oxide (ZrO_2) and hafnium oxide (HfO_2) is measured. The zirconium content is calculated from the mass of zirconium oxide as well as the hafnium oxide content.

8.1.2 Reagents

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

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

8.1.2.2 Hydrochloric acid (35 %, mass fraction).

8.1.2.3 DL-mandelic acid.

8.1.2.4 DL-mandelic acid solution (1 M), dissolve 152,15 g DL-mandelic acid (8.1.2.3) in 1 000 ml of warm water.

8.1.2.5 Washing solution, dissolve 10 g DL-mandelic acid (8.1.2.3) in 200 ml of warm water and 4 ml of hydrochloric acid (8.1.2.2).

8.1.3 Apparatus and instruments

Use the apparatus and instruments described in 7.3.2 together with the following:

8.1.3.1 Glass volumetric pipette, 50 ml, Class A, according to ISO 835.

8.1.3.2 Filter paper (ashless), diameter 110 mm, pore size index 8 µm.

8.1.3.3 Hot plate, with magnetic stirrer, capable of being operated at 450 °C.

8.1.4 Procedure

Transfer 50 ml of the sample solution (7.2.3 or 7.3.3) using a volumetric pipette (8.1.3.1) into a 500-ml beaker. After adding 25 ml of hydrochloric acid (8.1.2.2) to the beaker, heat the solution to 90 °C on a hot plate. While stirring the solution, add 50 ml of DL-mandelic acid solution (8.1.2.4). Covering the beaker with a watch glass, heat the beaker at 90 °C for 1h. Wash the watch glass with approximately 30 ml of warm water and add the washings into the beaker. Filter the solution using a filter paper (8.1.3.2) and wash the precipitate several times with hot washing solution (8.1.2.5).

Weigh the mass of a platinum crucible (7.3.2.1) to the nearest 0,1 mg. Put the used filter paper and the precipitate into the crucible and place the crucible in an electric furnace (7.3.2.3). Raise the temperature of the furnace gradually and ignite the contents at (1 050 ± 50) °C for 30 min.

Remove the crucible from the furnace and cool it in a desiccator. After weighing the mass of the crucible and the contents to the nearest 0,1 mg, calculate the mass of the precipitate.

8.1.5 Blank test

Perform the operation described in 8.1.4 with the blank test solution (7.2.4 or 7.3.4).

8.1.6 Calculation

Calculate the zirconium content, w_{Zr}, from Formula (1).

$$w_{Zr} = [(m_i - m_0)/m_s \times 200/50 \times 100 - w_{Hf} \times F_1] \times F_2 \quad (1)$$

where

w_{Zr} is the zirconium content, in % (mass fraction);

m_i is the mass of the precipitate in the sample solution (7.2.3 or 7.3.3), in g;

m_0 is the mass of the precipitate in the blank test solution (7.2.4 or 7.3.4), in g;

m_s is the mass of the test sample (7.2.3 or 7.3.3), in g;

w_{Hf} is the hafnium content calculated from Formula (4) or Formula (5), in % (mass fraction);

F_1 is the conversion factor from hafnium to hafnium oxide (HfO_2) (= 1,179 2);

F_2 is the conversion factor from zirconium oxide (ZrO_2) to zirconium (= 0,740 3).

8.2 ICP–OES method

8.2.1 Principle

Zirconium in a portion of the sample solution (7.2.3 or 7.3.3) is analysed using an ICP–OES at the selected wavelength.

8.2.2 Reagents

Use the reagents described in 7.2.1 together with the following:

8.2.2.1 Hydrochloric acid (35 %, mass fraction).

8.2.2.2 Hydrochloric acid (1+50).

8.2.2.3 Zirconium standard solution (Zr 1 mg/ml).

NOTE SI traceable commercial standard solutions are available.

8.2.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments together with an ICP–OES.

8.2.4 Procedure

Transfer 10 ml aliquot of the sample solution (7.2.3 or 7.3.3) into a 500-ml volumetric flask, dilute it with hydrochloric acid (1+50) up to the mark and mix well. This solution is designated as the test solution.

Spray a portion of the test solution into the flame of an ICP–OES. Measure the emission intensity at appropriate wavelength for zirconium (e.g. 343,823 nm or 339,197 nm).

8.2.5 Blank test

Perform the operation described in 8.2.4 with the blank test solution (7.2.4 or 7.3.4).

8.2.6 Drawing of calibration curve

Transfer appropriate aliquot portions (e.g. 0 ml, 1 ml, 2 ml, and 3 ml) of zirconium standard solution (8.2.2.3) into a 100-ml volumetric flask, separately. Dilute each flask with hydrochloric acid (1+50) up to the mark and mix well. Prepare the solutions freshly before every use.

Spray a portion of each solution into the flame of an ICP–OES and measure the emission intensity at the selected wavelength for zirconium.

8.2.7 Calculation

Determine zirconium concentrations in the test solution (8.2.4) and the blank test solution (7.2.4 or 7.3.4) from the calibration curve. Calculate zirconium content, w_{Zr}, from Formula (2):

$$w_{Zr} = [(A_i - A_0)/m_s] \times 500/10 \times 100 \quad (2)$$

where

w_{Zr} is zirconium content, in % (mass fraction);

A_i is the amount of zirconium in the test solution (8.2.4), in g;

A_0 is the amount of zirconium in the blank test solution (7.2.4 or 7.3.4), in g;

m_s is the mass of the test sample (7.2.3 or 7.3.3), in g.

9 Determination of the major elements contents

9.1 Precipitation and gravimetric method (for yttrium)

9.1.1 Principle

Yttrium in the test sample solution is precipitated as yttrium oxide (Y_2O_3) by the addition of oxalic acid. After igniting the precipitate, the mass of the yttrium oxide (Y_2O_3) is measured. The content of yttrium is calculated from the mass of yttrium oxide (Y_2O_3).

9.1.2 Reagents

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

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

9.1.2.2 Ammonia solution (25 %, mass fraction).

9.1.2.3 Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$).

9.1.2.4 Washing solution, dissolve 10,5 g oxalic acid dihydrate (9.1.2.3) in 250 ml water.

9.1.3 Apparatus and instruments

Use the apparatus and instruments described in 8.1.3 together with the following:

9.1.3.1 pH meter.

9.1.3.2 Glass volumetric pipette, 100 ml, Class A, according to ISO 835.

9.1.4 Procedure

Transfer 100 ml of the sample solution (7.2.3 or 7.3.3) using a volumetric pipette (9.1.3.2) into a 500-ml beaker. While stirring the solution, add 7 g of oxalic acid dihydrate (9.1.2.3) little by little. After attaining pH in the range 1 to 2 (measured with a pH meter) with ammonia solution (9.1.2.2), place the beaker on a hot plate at 100 °C for 1 h. Remove the beaker from the hot plate and cool it to room temperature. Allow it to stand overnight.

Filter the precipitate using a filter paper (8.1.3.3) and wash the precipitate on the filter paper several times with washing solution (9.1.2.4).

Weigh the mass of a platinum crucible (7.3.2.1) to the nearest 0,1 mg. Put the used filter paper and the precipitate into the crucible and place the crucible in an electric furnace (7.3.2.3). Raise the temperature of the furnace gradually and ignite the contents at (1 050 ± 50) °C for 30 min.

Remove the crucible from the furnace and cool it in a desiccator. After weighing the mass of the crucible and the contents to the nearest 0,1 mg, calculate the mass of the precipitate.

9.1.5 Blank test

Perform the operation described in 9.1.4 with the blank test solution (7.2.4 or 7.3.4).

9.1.6 Calculation

Calculate the yttrium content, w_Y, from Formula (3).

$$w_Y = (m_i - m_0)/m_s \times 200 \times F \quad (3)$$

where

w_Y is the yttrium content, in % (mass fraction);

m_i is the mass of the precipitate in the sample solution (7.2.3 or 7.3.3), in g;

m_0 is the mass of the precipitate in the blank test solution (7.2.4 or 7.3.4), in g;

m_s is the mass of the test sample (7.2.3 or 7.3.3), in g;

F is the conversion factor from yttrium oxide (Y_2O_3) to yttrium (= 0,787 4).

9.2 ICP–OES method (for calcium, hafnium, magnesium and yttrium)

9.2.1 Principle

A portion of the sample solution is sprayed into the flame of an ICP–OES, and each emission intensity of calcium, hafnium, magnesium and yttrium is measured at the selected wavelength.

9.2.2 Reagents

Use the reagents described in 8.2.2 together with the following:

9.2.2.1 Elemental standard solution.

— Calcium standard solution (Ca 1 mg/ml).

— Hafnium standard solution (Hf 1 mg/ml).

— Magnesium standard solution (Mg 1 mg/ml).

— Yttrium standard solution (Y 1 mg/ml).

NOTE SI traceable commercial standard solutions are available.

9.2.3 Apparatus and instruments

Use the apparatus and instruments described in 8.1.3 together with the following:

9.2.3.1 Glass volumetric pipette, 25 ml, Class A, according to ISO 835.

9.2.3.2 ICP-OES.

9.2.4 Procedure

Transfer 25 ml of the sample solution (7.2.3 or 7.3.3) using a volumetric pipette (9.2.3.1) into a platinum crucible (7.3.2.1). Heat the crucible on a hot plate at 250 °C until the solution has been completely dried, and then raise the temperature of the hot plate to 350 °C for 30 min.

After cooling, add 2 ml of hydrochloric acid (8.2.2.1) and 20 ml of water, and heat the crucible again on a hot plate at 250 °C until the solution becomes clear.

Remove the crucible from the hot plate and cool it to room temperature.

Wash the crucible with 20 ml of water and collect the washings into a 50-ml volumetric flask. Dilute it with water up to the mark and mix well.

Spray a portion of the solution into the flame of an ICP–OES and measure the emission intensity at appropriate wavelength (Table 2). Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

Table 2 — Examples of the analytical wavelength for Ca, Hf, Mg and Y

Element	Wavelength 1 nm	Wavelength 2 nm
Ca	396,847	317,933 [a]
Hf	277,336	264,141
Mg	279,553	280,271
Y	371,029 [a]	–

[a] Some laboratories reported that the 317,933 nm wavelength for calcium can be partly convoluted with the 371,029 nm wavelength for yttrium.

9.2.5 Blank test

Perform the operation described in 9.2.4 with the blank test solution (7.2.4 or 7.3.4).

9.2.6 Drawing of calibration curve

Considering the zirconium content in the test sample (Clause 8), transfer a certain aliquot portion of the zirconium standard solution (8.2.2.3) into a 100-ml plastic volumetric flask. Add appropriate aliquot portions of the elemental standard solution (9.2.2.1) into the flask, separately (Table 3). Dilute each flask with hydrochloric acid (1 + 50) up to the mark and mix well. Prepare the solutions freshly before every use.

Table 3 — Examples of the calibration solutions matching for 60 % of zirconium content

Element	Solution 1 ml	Solution 2 ml	Solution 3 ml	Solution 4 ml
Zr	3	3	3	3
Ca	0	1	2	3
Hf	0	0,5	1	1,5
Mg	0	1	2	3
Y	0	1	4	8

Spray a portion of each solution into the flame of an ICP–OES and measure the emission intensity at the selected wavelength.

9.2.7 Calculation

Determine the concentration of each element in the sample solution (7.2.3 or 7.3.3) and the blank test solution (7.2.4 or 7.3.4) from the calibration curve. Calculate the content of each element, w_{Ei}, from Formula (4).

$$w_{Ei} = (A_i - A_0)/m_s \times 100 \quad (4)$$

where

w_{Ei} is the content of each element in the sample, in % (mass fraction);

A_i is the amount of each element in the sample solution (7.2.3 or 7.3.3), in g;

A_0 is the amount of each element in the blank test solution (7.2.4 or 7.3.4), in g;

m_s is the mass of the test sample (7.2.3 or 7.3.3), in g.

10 Determination of the trace elements contents

10.1 Principle

A portion of the sample solution is sprayed into the flame of an ICP–OES, and each emission intensity of aluminium, barium, calcium, cerium, cobalt, gadolinium, hafnium, iron, magnesium, potassium, silicon, sodium, strontium, titanium and yttrium is measured at the selected wavelength.

10.2 Reagents

Use the reagents described in 8.2.2 together with the following:

10.2.1 Elemental standard solution

— Aluminium standard solution (Al 1 mg/ml).

— Barium standard solution (Ba 1 mg/ml).

— Calcium standard solution (Ca 1 mg/ml).

— Cerium standard solution (Ce 1 mg/ml).

— Cobalt standard solution (Co 1 mg/ml).

— Gadolinium standard solution (Gd 1 mg/ml).

— Hafnium standard solution (Hf 1 mg/ml).

— Iron standard solution (Fe 1 mg/ml).

— Magnesium standard solution (Mg 1 mg/ml).

— Potassium standard solution (K 1 mg/ml).

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

— Silicon standard solution (Si 1 mg/ml).

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

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

— Yttrium standard solution (Y 1 mg/ml).

NOTE SI traceable commercial standard solutions are available.

10.2.2 Mixed standard solution (each element 50 mg/l)

Put 5 ml of each elemental standard solution (10.2.1) into a 100-ml plastic volumetric flask. Dilute it with hydrochloric acid (1 + 50) up to the mark and mix well. Prepare the mixed standard solution freshly before use.

10.3 Apparatus and instruments

Use the apparatus and instruments described in 9.2.3.

10.4 Procedure

1) Preparation of the test sample solution except for silicon.

   Transfer 25 ml of the sample solution (7.2.3) using a volumetric pipette (9.2.3.1) into a platinum crucible (7.3.2.1). Heat the crucible on a hot plate at 250 °C until the solution has been completely dried, then raise the temperature of the hot plate to 350 °C for 30 min.

   After cooling, add 2 ml of hydrochloric acid (8.2.2.1) and 20 ml of water, and heat the crucible again until the solution becomes clear. Wash the crucible with 20 ml of water and collect the washings into a 50-ml volumetric flask. Dilute it with water up to the mark and mix well.

2) Preparation of the test sample solution for silicon.

   Transfer 25 ml of the sample solution (7.3.3) using a volumetric pipette (9.2.3.1) into a 50-ml volumetric flask. Dilute it with water up to the mark and mix well.

3) Measurement of trace elements.

   Spray a portion of the test sample solutions into the flame of an ICP–OES and measure the emission intensity at an appropriate wavelength (Table 4). Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

Table 4 — Examples of the analytical wavelength for trace elements

Element	Wavelength 1 nm	Wavelength 2 nm
Al	394,401	309,271
Ba	233,527	230,425
Ca	396,847	317,933 [a]
Ce	413,764	–
Co	228,616	230,786
Gd	417,206	–
Hf	277,336	264,141
Fe	259,939	239,562
K	766,490	–
Mg	279,553	280,271
Na	589,592	588,595
Si	251,611	212,412
Sr	407,771	421,552
Ti	336,121	334,940
Y	371,029 [a]	–

[a] Some laboratories reported that the 317,933 nm wavelength for calcium can be partly convoluted with the 371,029 nm wavelength for yttrium.

10.5 Blank test

Perform the operation described in 10.4 with the blank test solution (7.2.4 or 7.3.4).

10.6 Drawing of the calibration curve

Considering the zirconium content in the test sample (Clause 8), transfer a certain aliquot portion of the zirconium standard solution (8.2.2.3) into a 100-ml plastic volumetric flask. Add appropriate aliquot portions of mixed standard solution (10.2.2) into the flask, separately (Table 5). Dilute each flask with hydrochloric acid (1 + 50) up to the mark and mix well. Prepare the solutions freshly before every use.

Table 5 — Examples of the calibration solutions matching for 60 % of zirconium content

Elements	Solution 1 ml	Solution 2 ml	Solution 3 ml	Solution 4 ml
Zr	3	3	3	3
Mixed standard solution	0	1	3	5

Spray a portion of each solution into the flame of an ICP–OES and measure the emission intensity at the selected wavelength.

10.7 Calculation

Determine the concentration of each element in the test sample solution (10.4) and the blank test solution (7.2.4 or 7.3.4) from the calibration curve. Calculate the content of each element, w_{Ei}, from Formula (5).

$$w_{Ei} = [(A_i - A_0)/m_s] \times 50/25 \times 100 \quad (5)$$

where