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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of impurities in aluminium oxide powders using inductively coupled plasma-optical emission spectrometry

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Méthodes d'analyse chimique des impuretés contenues dans les poudres d'oxyde d'aluminium à l'aide de la spectrométrie d'émission optique par plasma à couplage inductif

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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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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 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 impurities in aluminium oxide powders using inductively coupled plasma-optical emission spectrometry

1 Scope

This document specifies methods for the chemical analysis of impurities present in aluminium oxide powders used as a raw material for fine ceramics.

Aluminium oxide powders are decomposed by acid pressure decomposition, acid decomposition or alkali fusion. The calcium, chromium, copper, iron, magnesium, manganese, potassium, silicon, sodium, titanium, zinc and zirconium contents in the test solution are determined by an inductively coupled plasma-optical emission spectrometer (ICP-OES).

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

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 https://www.electropedia.org/

4 Analytical range

Each element, range of 0,000 5 % to 0,5 % (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 analyser and the 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. Heat 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 0,01 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. Double repetition 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

Average value of two analytical results	Tolerance %
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 to four decimal places in % dryness (mass fraction).

7 Decomposition of test sample

7.1 Classification of decomposition methods

Aluminium oxide powders are decomposed by acid pressure decomposition, acid decomposition or alkali fusion.

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 Nitric acid (HNO_3), 65 % minimum, as specified in ISO 6353-2.

7.2.1.3 Sulfuric acid (H_2SO_4), 95 % minimum, as specified in ISO 6353-2.

7.2.1.4 Sulfuric acid solution (1 + 3). One volume of sulfuric acid (7.2.1.3) is mixed with three volumes of water.

7.2.1.5 Nitric acid solution (1 + 50). One volume of nitric acid (7.2.1.2) is mixed with 50 volumes of water.

7.2.1.6 Lanthanum standard solution (La 1 mg/ml). The SI traceable commercial standard solution is available. Other internal standard solutions (e.g. Sc, Y) can be used after validation.

7.2.1.7 Internal standard solution (La 0,025 mg/ml). Transfer 5,0 ml of lanthanum standard solution (7.2.1.6) to a 200 ml volumetric flask, dilute it with nitric acid solution (1 + 50) up to the mark and mix well.

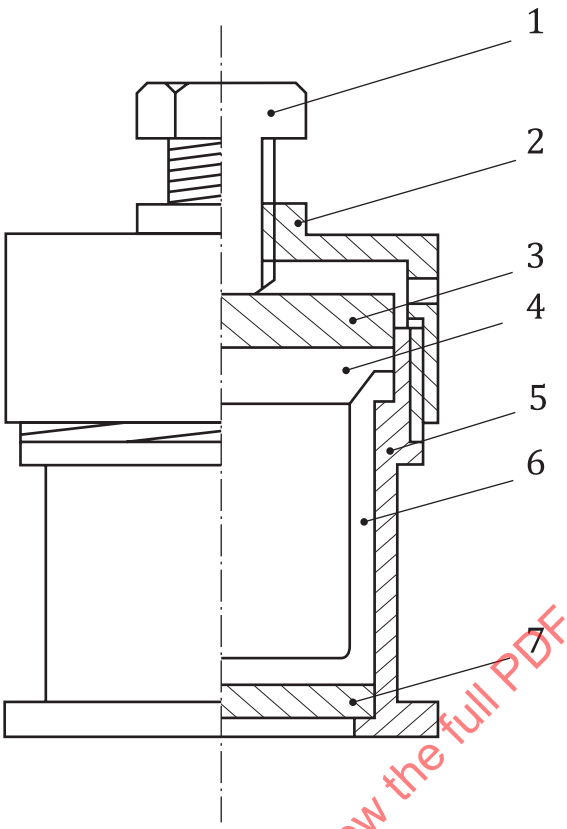
7.2.2 Apparatus and instruments

Use ordinary laboratory apparatus together with the following:

7.2.2.1 Polytetrafluoroethylene (PTFE) bottle with cap.

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

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 Sample decomposition procedure

7.2.3.1 Decomposition

Weigh 0,50 g of the sample in a PTFE bottle (7.2.2.1) and add 10 ml of sulfuric acid solution (1 + 3). Put the PTFE bottle into a pressure decomposition vessel (7.2.2.2) and close the vessel according to the manufacturer's instructions.

Place the vessel in an air bath and heat at 230 °C ± 5 °C for 16 h.

After cooling, disassemble the vessel and transfer the solution to a 250 ml plastic volumetric flask. Wash the PTFE bottle with warm water and collect the washings into the flask.

7.2.3.2 Internal standard injection and dilution

Add 10 ml of the internal standard solution (7.2.1.7) to the flask. Dilute it with water up to the mark and mix well. This solution is designated as the sample test solution.

7.2.4 Blank test

7.2.4.1 Making the blank dissolution

Perform the procedure in 7.2.3.1 without the sample. The final solution is designated as the blank dissolution.

7.2.4.2 Making the blank test solution

Perform the procedure 7.2.3.2 with the blank dissolution (7.2.4.1). This solution is designated as the blank test solution.

7.3 Acid decomposition

7.3.1 Reagents

Use the reagents described in 7.2.1 together with the following:

7.3.1.1 Phosphoric acid (H_3PO_4), 85 % minimum, as specified in ISO 6353-2.

7.3.1.2 Sulfuric acid-phosphoric acid mixture. Add 30 ml of sulfuric acid (7.2.1.3) to 70 ml of phosphoric acid (7.3.1.1) in a 200 ml plastic beaker and mix carefully.

7.3.2 Apparatus and instruments

Use ordinary laboratory apparatus together with the following:

7.3.2.1 Hot plate, capable of heating at 400 °C.

7.3.2.2 Platinum crucible (30 ml).

7.3.2.3 Platinum lid.

7.3.3 Sample decomposition procedure

7.3.3.1 Decomposition

Weigh 0,50 g of the test sample and transfer it to a platinum crucible (7.3.2.2). Carefully add 12 ml of sulfuric acid-phosphoric acid mixture (7.3.1.2). Cover the crucible with a platinum lid (7.3.2.3) and heat the crucible on a hot plate until the test sample has been completely dissolved.

After cooling, transfer the solution to a 250 ml plastic volumetric flask. Rinse the inner wall of the platinum crucible with a small quantity of water and pour the washings into the flask.

7.3.3.2 Internal standard injection and dilution

Add 10 ml of the internal standard solution (7.2.1.7) to the flask, dilute with water up to the mark and mix well. This solution is designated as the sample test solution.

7.3.4 Blank test

7.3.4.1 Making the blank dissolution

Perform the procedure in 7.3.3.1 without the sample. The final solution is designated as the blank dissolution.

7.3.4.2 Making the blank test solution

Perform the procedure in 7.3.3.2 with the blank dissolution (7.3.4.1). The solution is designated as the blank test solution.

7.4 Alkali fusion

7.4.1 Reagents

Use the reagents described in 7.2.1 together with the following:

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

Because some commercial products of lithium tetraborate contain certain contents of inorganic elements, such as calcium, potassium, silicon and sodium, check its conformity for the test before use.

7.4.2 Apparatus and instruments

Use ordinary laboratory apparatus, together with the following:

7.4.2.1 Platinum crucible (30 ml).

7.4.2.2 Platinum lid.

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

7.4.3 Sample decomposition

7.4.3.1 Decomposition

Mix 0,50 g of the test sample and 1,0 g of lithium tetraborate (7.4.1.1) in a platinum crucible (7.4.2.1). Cover the mixed sample with an additional 1,0 g of lithium tetraborate. Cover the crucible with a platinum lid (7.4.2.2). Place the crucible in an electric furnace (7.4.2.3). Raise the temperature of the furnace gradually and heat the crucible at 1 150 °C ± 50 °C until the contents are completely decomposed.

Remove the crucible from the furnace and cool it to room temperature. Wash the outer-bottom and outer-wall of the crucible with nitric acid solution (1 + 50) or water. Place the crucible into a 250 ml plastic beaker containing 10 ml of nitric acid (7.2.1.2) and 90 ml of water. Cover the beaker with a plastic cover and warm it in an ultrasonic bath until the melt is completely dissolved into the solution.

Remove the crucible carefully with a glass rod, rinse the crucible and cover with a minimum quantity of water. Transfer the washings to the beaker. After cooling, transfer the solution to a 250 ml plastic volumetric flask.

7.4.3.2 Internal standard injection and dilution

Add 10 ml of the internal standard solution (7.2.1.7) to the flask. Dilute it with water up to the mark and mix well. This is designated as the sample test solution.

7.4.4 Blank test

7.4.4.1 Making the blank dissolution

Perform the procedure in 7.4.3.1 without the sample. The final solution is designated as the blank dissolution.

7.4.4.2 Making the blank test solution

Perform the procedure in 7.4.3.2 with the blank dissolution (7.4.4.1). The solution is designated as the blank test solution.

8 Determination of impurity elements

8.1 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 the 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 an interlaboratory study for chemical analysis of impurity in aluminium oxide powder are described in Annex A.

8.2 Reagents

Use the reagents described in 7.2.1, 7.3.1 or 7.4.1 together with the following:

8.2.1 Aluminium standard solution (Al 10 mg/ml).

The SI traceable commercial standard solutions are available.

8.2.2 Elemental standard solutions.

The SI traceable commercial standard solutions are available.

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

b) Chromium standard solution (Cr 1 mg/ml).

c) Copper standard solution (Cu 1 mg/ml).

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

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

f) Manganese standard solution (Mn 1 mg/ml).

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

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

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

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

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

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

8.2.3 Mixed standard solution (each element 50 mg/l).

Pour 5,0 ml each of standard solution (described in 8.2.2) into a 100 ml plastic volumetric flask. Dilute with nitric acid solution (1 + 50) up to the mark and mix well. Pay attention to ensure that no precipitation occurs during mixing.

Prepare the solution afresh before every use.

8.3 Apparatus and instruments

Use ordinary laboratory apparatus together with the following:

8.3.1 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 should 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 Preparation of calibration standard solutions

Refer to 7.2.4.1, 7.3.4.1 or 7.4.4.1 and prepare five blank dissolutions (four dissolutions for the calibration standard solutions and one dissolution for a blank test solution) in 250 ml plastic volumetric flasks.

Add an appropriate aliquot of the aluminium standard solution (8.2.1) and mixed standard solution (8.2.3) into each blank dissolution, then add 10 ml of the internal standard solution (7.2.1.7). Dilute with water (7.2.1.1) up to the mark and mix well.

Prepare the solutions afresh before every use.

An example is shown in Table 2.

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

Elements	Solution 1 ml	Solution 2 ml	Solution 3 ml	Solution 4 ml
Aluminium solution	25	25	25	25
Mixed standard solution	0	1,0	10	50
La (internal standard)	10	10	10	10

8.5 Measurement

8.5.1 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, a spray chamber and sample uptake tubes shall be used. The plasma shall be stabilized before use following the recommendations of the instrument's 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.

8.5.2 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 each element

Element	Wavelength 1 nm	Wavelength 2 nm
Ca	393,366	396,847
Cr	283,563	284,325 or 267,716
Cu	327,393	324,752
Fe	238,204	259,939
Mg	279,553	280,271
Mn	257,610	260,568
K	766,490	–
Si	251,611	288,158
Na	589,562	–
Ti	334,940	336,121
Zn	213,857	206,200
Zr	343,823	339,197
La (internal standard)	379,749	408,672

8.5.3 Measurement of blank test solution

Perform the procedure described in 8.5.1 with the blank test solution (7.2.4.2, 7.3.4.2 or 7.4.4.2).

8.6 Drawing of calibration curve

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

The data-processing unit provides the quotients from the simultaneously registered single measurements of the intensities of analyte element and the internal standard element. Calculate the intensity ratio of the analyte element and the internal standard element of each calibration standard solution using Formula (1).

$$R_{\mathrm{I}} = \frac{I_{\mathrm{A}}}{I_{\mathrm{IS}}} \tag{1}$$

where

R_{I} is the intensity ratio of the analyte element and the 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 of the analyte element and the internal standard element (R_{I}) during repeat measurement shall not be larger than 0,3 %.

The average intensity ratio of the analyte element and the internal standard element during repeat measurement of each solution is calculated using Formula (2).

$$\bar{R}_{\mathrm{I}} = \frac{1}{n}\sum_{i=1}^{n} R_{\mathrm{I,i}} \tag{2}$$

where

$\bar{R}_{\mathrm{I}}$ is the average intensity ratio of 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 average intensity ratios of the analyte element and the internal standard element, and the corresponding concentration ratios of the analyte element and the internal standard element in the calibration standard solutions.

8.7 Calculation

Using the linear regression curve (8.6), calculate the concentration ratio (R_{C}) of each analyte element and the 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 (C) in the sample test solution (C_{A}) and the blank test solution (C_{B}), using Formula (3).

$$C = R_{\mathrm{C}} \times C_{\mathrm{IS}} \tag{3}$$

where

R_{C} is the concentration ratio of the analyte element and the 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 (4).

$$W_{\mathrm{Ei}} = \frac{(C_{\mathrm{A}} - C_{\mathrm{B}})}{m} \times 250 \times 10^{-4} \tag{4}$$

where

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

C_{A} is the concentration of each element in the sample test solution, in mg/l;

C_{B} is the concentration of each element in the blank test solution, in mg/l;

m is the mass of the test sample, in g.

9 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 3169:2023;

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

d) any deviations from the specified procedure;