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

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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).

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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 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 terminological 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).

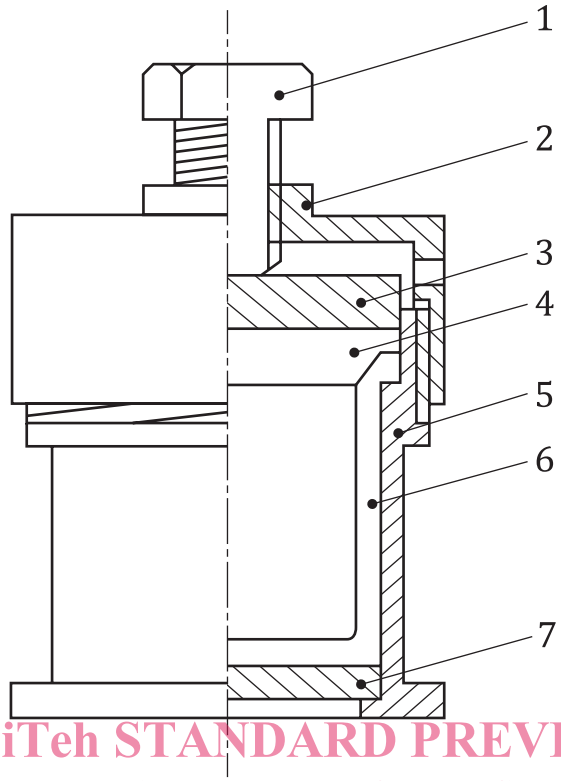
7.2.2 Apparatus and instruments

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.