
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Methods for chemical analysis of high
purity barium titanate powders**

*Céramiques techniques — Méthodes d'analyse chimique des poudres
de titanate de baryum à haute pureté*

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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.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of high purity barium titanate powders

1 Scope

This document specifies methods for the chemical analysis of fine high purity barium titanate powders used as the raw material for fine ceramics.

This document stipulates the determination methods of the barium, titanium, aluminium, cadmium, calcium, cobalt, dysprosium, iron, lead, magnesium, manganese, nickel, niobium, potassium, silicon, sodium, strontium, vanadium, zirconium, carbon, oxygen and nitrogen contents in high purity barium titanate powders. The barium and titanium contents, the major elements, are determined by using an acid decomposition-gravimetric method or an acid decomposition-inductively coupled plasma-optical emission spectrometry (ICP-OES) method. The aluminium, cadmium, calcium, chromium, cobalt, dysprosium, iron, lead, magnesium, manganese, nickel, niobium, potassium, silicon, strontium, vanadium and zirconium contents are simultaneously determined via an acid digestion-ICP-OES method. The nitrogen content is determined by using an inert gas fusion-thermal conductivity method, while that of oxygen is determined via an inert gas fusion-IR absorption spectrometry method. Finally, the carbon content is determined using a combustion-IR absorption spectrometry method or a combustion-conductometry 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second 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 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

- a) Barium (Ba), range of 40 % to 60 % (mass fraction).

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- b) Titanium (Ti), range of 10 % to 30 % (mass fraction).
- c) Aluminium (Al), range of 0,001 % to 0,03 % (mass fraction).
- d) Cadmium (Cd), range of 0,001 % to 0,03 % (mass fraction).
- e) Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction).
- f) Cobalt (Co), range of 0,001 % to 0,03 % (mass fraction).
- g) Dysprosium (Dy), range of 0,001 % to 0,03 % (mass fraction).
- h) Iron (Fe), range of 0,001 % to 0,03 % (mass fraction).
- i) Lead (Pb), range of 0,001 % to 0,03 % (mass fraction).
- j) Magnesium (Mg), range of 0,001 % to 0,03 % (mass fraction).
- k) Manganese (Mn), range of 0,001 % to 0,03 % (mass fraction).
- l) Nickel (Ni), range of 0,001 % to 0,03 % (mass fraction).
- m) Niobium (Nb), range of 0,001 % to 0,03 % (mass fraction).
- n) Potassium (K), range of 0,001 % to 0,03 % (mass fraction).
- o) Silicon (Si), range of 0,001 % to 0,03 % (mass fraction).
- p) Sodium (Na), range of 0,001 % to 0,03 % (mass fraction).
- q) Strontium (Sr), range of 0,001 % to 0,03 % (mass fraction).
- r) Vanadium (V), range of 0,001 % to 0,03 % (mass fraction).
- s) Zirconium (Zr), range of 0,001 % to 0,03 % (mass fraction).
- t) Total nitrogen (T.N), range of 0,01 % to 5 % (mass fraction).
- u) Oxygen (O), range of 10 % to 30 % (mass fraction).
- v) Carbon (C), range of 0,01 % to 5 % (mass fraction).

5 Preparation of 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 a 10 g 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 $110\text{ °C} \pm 5\text{ °C}$ for 2 h, uncovered, and cool in a desiccator (desiccant: magnesium perchlorate), covered, for 1 h.

5.4 Weighing

Weigh the 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 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 in 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.

6.4 Expression of the analytical values

The analytical values shall be given in % (mass fraction), in dryness.

- Barium, titanium, oxygen, and nitrogen: express the results to two decimal places.
- Others: express the results to three decimal places.

Table 1 — Tolerances for the analytical values

<https://standards.iteh.ai/catalog/standards/iso/b12a341d-29e9-4e28-8c7d-f719d560e7c> Units: % (mass fraction)

Element	Ba	Ti	Al, Cd, Ca, Co, Dy, Fe, Pb, Mg, Mn, Ni, Nb, K, Si, Na, Sr, V, Zr,	Total N	O	C
Tolerance	0,30 ^a 0,40 ^b	0,20 ^a 0,30 ^b	0,001 ^c 0,005 ^d	0,01 ^e 0,05 ^f	0,50	0,005

^a Acid decomposition-gravimetric method.
^b Acid decomposition-ICP-OES method.
^c Applicable to content of less than 0,01 % (mass fraction).
^d Applicable to content of not less than 0,01 % (mass fraction).
^e Applicable to content of less than 1,0 % (mass fraction).
^f Applicable to content of not less than 1,0 % (mass fraction).

7 Determination of the barium and titanium contents

7.1 Classification of the determination methods

The barium and titanium contents shall be determined by either of the following methods:

- Method A, acid decomposition-gravimetric method;
- Method B, acid decomposition-ICP-OES method.

If analytical results with four significant figures are required, use method A; if two or three significant figures are required, method B can be used.

7.2 Acid decomposition-gravimetric method

7.2.1 Principle

A portion of the sample is decomposed using hydrogen peroxide and hydrochloric acid. The barium present in the test solution is analysed by gravimetric analysis using sulfuric acid. The titanium in the test solution is analysed by gravimetric analysis using ammonia solution.

7.2.2 Reagents

During the analysis, unless otherwise stated, only reagents of recognized analytical grade and only distilled water or water of equivalent purity shall be used.

Reagents shall conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3 as appropriate. Specific requirements for the reagents are given in the appropriate clause.

7.2.2.1 **Ammonia water (NH₃)**, (ISO 6353-2, R 3), 25 % (mass fraction).

7.2.2.2 **Hydrogen peroxide (H₂O₂)**, (ISO 6353-2, R 14), 30 % (mass fraction).

7.2.2.3 **Hydrochloric acid (HCl)**, (ISO 6353-2, R 13), 35 % (mass fraction).

7.2.2.4 **Sulfuric acid (H₂SO₄)**, (ISO 6353-2, R 37), 95 % (mass fraction).

7.2.2.5 **Hydrogen peroxide (1+10)**.

7.2.2.6 **Hydrochloric acid (1+10)**.

7.2.2.7 **Sulfuric acid (1+1)**.

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

7.2.3 Apparatus

Ordinary laboratory apparatus together with the following:

7.2.3.1 **PTFE beaker**, with a range of the appropriate volume (250 ml).

7.2.3.2 **Burette**, with a 0,1 ml scale and a maximum volume of 50 ml.

7.2.3.3 **PTFE pipette**, suitable for the transfer of each sample or standard solution.

7.2.3.4 **Desiccator**, containing dried silica gel as the drying agent.

7.2.3.5 **Balance**, capable of weighing to $\pm 0,1$ mg.

7.2.3.6 **Electric furnace**, for operation at $(1\ 000 \pm 50)$ °C.

7.2.3.7 **Platinum crucible** (30 ml).

7.2.3.8 PTFE beaker cover.**7.2.3.9 Volumetric flask** (100 ml, 500 ml).**7.2.3.10 Hot plate**, with magnetic stirrer.**7.2.4 Procedure**

7.2.4.1 Weigh 0,30 g of the test sample and transfer it into a 250 ml PTFE beaker (7.2.3.1). Place the magnetic bar containing the test sample and carefully add 20 ml water, 10 ml hydrogen peroxide (7.2.2.2) and 20 ml hydrochloric acid (7.2.2.3) to the beaker. Cover the beaker with a PTFE beaker cover (7.2.3.8) and heat the contents at $(85 \pm 5) ^\circ\text{C}$ until the test sample is completely dissolved. After cooling, transfer the solution to a 100 ml volumetric flask, dilute with water to the mark and mix well.

The highly pure fine barium titanate powder sample completely decomposes in hydrochloric acid and hydrogen peroxide. However, the presence of impurities or coarse grain in the sample may hinder the decomposition process. If the sample is not completely decomposed by the acid decomposition method, it is recommended that other decomposition methods are applied. These include the acid pressure decomposition, fusion or acid microwave dissolution methods.

7.2.4.2 Transfer a 50 ml aliquot of the test solution (7.2.4.1) to a 250 ml PTFE beaker (7.2.3.1) and add 10 ml sulfuric acid (1+1) (7.2.2.7). After covering the beaker with a PTFE beaker cover (7.2.3.8), heat the contents at $200 ^\circ\text{C}$ for 1 h.

7.2.4.3 Filter the solution with ashless filter paper and wash the precipitate several times with hot water. Keep the filtrate and washings in the beaker covered with the watch glass for the determination of the titanium content.

7.2.4.4 Transfer the precipitate and the filter paper to a 30 ml platinum crucible. Heat the crucible in an electric furnace at low temperature until the filter paper has been completely burned to ashes. Heat the crucible and its contents in an electric furnace $(1\ 000 \pm 50) ^\circ\text{C}$ for 1 h. After cooling in a desiccator, weigh the barium sulfate.

7.2.4.5 Add 50 ml ammonia water (7.2.2.1) to the filtrate (7.2.4.3). Filter the solution with ashless filter paper and wash over the precipitate several times with hot water. Transfer the precipitate with filter paper to a 30 ml platinum crucible. Heat the sample in an electric furnace at low temperature until ashing of the filter paper is complete. Next, heat the crucible and its contents in an electric furnace at $(1\ 000 \pm 50) ^\circ\text{C}$ for 1 h. After cooling in a desiccator, weigh the titanium oxide.

7.2.5 Blank test

Carry out the procedure described in 7.2.4 without the sample. Designate the final solution as the blank solution.

7.2.6 Calculation

Calculate the barium and titanium contents according to [Formula \(1\)](#).

$$W = \left[(W_2 - W_1) / m \right] \times V / a \times F_1 \text{ or } F_2 \times 100 \quad (1)$$