

---

---

**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*

[ISO 3169:2023](#)

<https://standards.iteh.ai/catalog/standards/sist/d936493e-6bf9-4ceb-8c4e-7d6fd53abfc/iso-3169-2023>



# iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO 3169:2023

<https://standards.iteh.ai/catalog/standards/sist/d936493e-6bf9-4ceb-8c4e-7d6fd53abfcf/iso-3169-2023>



## **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

Foreword.....	iv
<b>1 Scope.....</b>	<b>1</b>
<b>2 Normative references.....</b>	<b>1</b>
<b>3 Terms and definitions.....</b>	<b>1</b>
<b>4 Analytical range.....</b>	<b>1</b>
<b>5 Preparation of test sample.....</b>	<b>1</b>
5.1 General.....	1
5.2 Sampling.....	2
5.3 Drying.....	2
5.4 Weighing.....	2
<b>6 Reporting analytical values.....</b>	<b>2</b>
6.1 Number of analyses.....	2
6.2 Blank test.....	2
6.3 Evaluation of analytical results.....	2
6.4 Expression of analytical results.....	2
<b>7 Decomposition of test sample.....</b>	<b>2</b>
7.1 Classification of decomposition methods.....	2
7.2 Acid pressure decomposition.....	3
7.2.1 Reagents.....	3
7.2.2 Apparatus and instruments.....	3
7.2.3 Sample decomposition procedure.....	4
7.2.4 Blank test.....	5
7.3 Acid decomposition.....	5
7.3.1 Reagents.....	5
7.3.2 Apparatus and instruments.....	5
7.3.3 Sample decomposition procedure.....	5
7.3.4 Blank test.....	5
7.4 Alkali fusion.....	6
7.4.1 Reagents.....	6
7.4.2 Apparatus and instruments.....	6
7.4.3 Sample decomposition.....	6
7.4.4 Blank test.....	6
<b>8 Determination of impurity elements.....</b>	<b>7</b>
8.1 Principle.....	7
8.2 Reagents.....	7
8.2.1 Aluminium standard solution (Al 10 mg/ml).....	7
8.2.2 Elemental standard solutions.....	7
8.2.3 Mixed standard solution (each element 50 mg/l).....	7
8.3 Apparatus and instruments.....	8
8.3.1 ICP-OES.....	8
8.4 Preparation of calibration standard solutions.....	8
8.5 Measurement.....	8
8.5.1 Set-up of the instruments.....	8
8.5.2 Measurement of sample test solution and calibration standard solutions.....	9
8.5.3 Measurement of blank test solution.....	9
8.6 Drawing of calibration curve.....	9
8.7 Calculation.....	10
<b>9 Test report.....</b>	<b>10</b>
<b>Annex A (informative) Interlaboratory chemical analysis of impurity in alumina powder.....</b>	<b>12</b>

## 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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://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](http://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  $110\text{ °C} \pm 5\text{ °C}$ , 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<sub>3</sub>)**, 65 % minimum, as specified in ISO 6353-2.

**7.2.1.3 Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)**, 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.

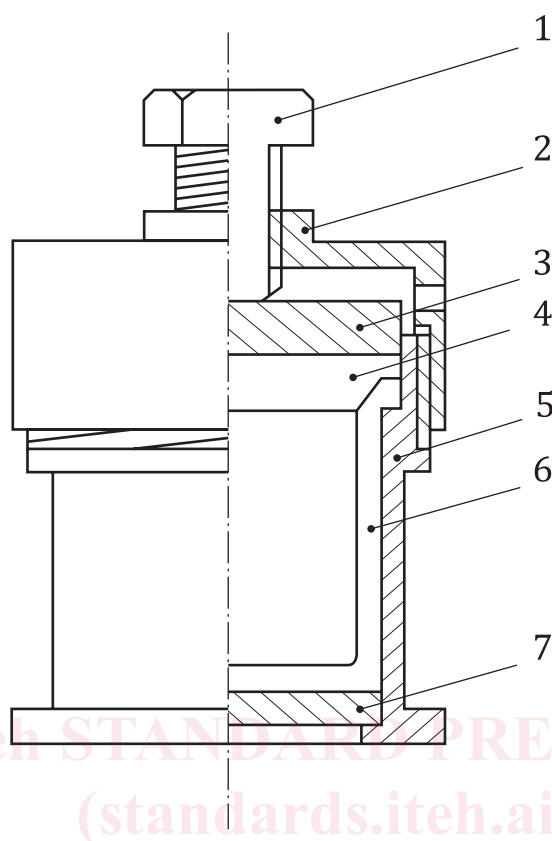
### 7.2.2 Apparatus and instruments

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  $230\text{ °C} \pm 5\text{ °C}$ .



**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  $230\text{ °C} \pm 5\text{ °C}$  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 ( $\text{H}_3\text{PO}_4$ )**, 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 ( $\text{Li}_2\text{B}_4\text{O}_7$ ), 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  $1\ 150\text{ °C} \pm 50\text{ °C}$  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.