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

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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)~~www.iso.org~~

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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~~document 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)).

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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](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).

## Introduction

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

## 1 Scope

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

It stipulates the methods for the determination of metal impurity elements in silicon dioxide powders that are decomposed by acid decomposition. The aluminium, cadmium, calcium, copper, iron, lead, lithium, magnesium, manganese, nickel, potassium, sodium, titanium, zinc and zirconium contents in the test solution are determined by inductively coupled plasma-optical emission spectrometry (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 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 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 Analytes and ranges

- a) Aluminium (Al), range of 1,0 mg/kg to 100 mg/kg.
- b) Cadmium (Cd), range of 1,0 mg/kg to 100 mg/kg.

- c) Calcium (Ca), range of 1,0 mg/kg to 100 mg/kg.
- d) Copper (Cu), range of 1,0 mg/kg to 100 mg/kg.
- e) Iron (Fe), range of 1,0 mg/kg to 100 mg/kg.
- f) Lead (Pb), range of 1,0 mg/kg to 100 mg/kg.
- g) Lithium (Li), range of 1,0 mg/kg to 100 mg/kg.
- h) Magnesium (Mg), range of 1,0 mg/kg to 100 mg/kg.
- i) Manganese (Mn), range of 1,0 mg/kg to 100 mg/kg.
- j) Nickel (Ni), range of 1,0 mg/kg to 100 mg/kg.
- k) Potassium (K), range of 1,0 mg/kg to 100 mg/kg.
- l) Sodium (Na), range of 1,0 mg/kg to 100 mg/kg.
- m) Titanium (Ti), range of 1,0 mg/kg to 100 mg/kg.
- n) Zinc (Zn), range of 1,0 mg/kg to 100 mg/kg.
- o) Zirconium (Zr), range of 1,0 mg/kg to 100 mg/kg.

## 5 Preparation of test sample

### 5.1 General <https://standards.iteh.ai/catalog/standards/sist/7cea704d-cb1a-4585-9bd9-a6252e205b33/iso-5189-2023>

Prepare the sample in accordance with ISO 8656-1, unless otherwise mutually agreed upon by the analyser and 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 (60 mm × 30 mm) and spread it uniformly at the bottom of the bottle. Place the bottle in an air bath at 110 °C ± 5 °C for 2 h, then cover the mouth of the bottle and cool in a desiccator for 1 h.

### 5.4 Weighing

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



## 6 Reporting analytical values

### 6.1 Blank test

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

### 6.2 Evaluation of analytical values

If the difference between the maximum and minimum analytical values does not exceed the tolerance value (Table 1), report the average value. However, if the difference between the two values exceeds the tolerance value, perform two additional analyses. If the difference between the values of these further two analyses does not exceed the tolerance value, report the average value thereof. However, if the difference again exceeds the tolerance value, report the median of the four analytical values.

The results of the interlaboratory test are given in Annex A.

**Table 1 — Tolerances for analytical values**

Unit: mg/kg

Item	Value	Criteria
Tolerance	5,0	Applicable to amounts of less than 50 mg/kg
	25	Applicable to amounts of not less than 50 mg/kg

### 6.3 Expression of analytical values

Present the analytical values to two significant figures in mg/kg, in dryness.

## 7 Decomposition of test sample

Silicon dioxide powders are decomposed by acid decomposition.

### 7.1 Reagents

Use reagents of analytical grade.

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

**7.1.2 Hydrofluoric acid (HF)**, (ISO 6353-3, R 67), 40,0 % ~~to~~ 42,0 % (mass fraction).

**7.1.3 Nitric acid (HNO<sub>3</sub>)**, (ISO 6353-2, R19), 65 % (mass fraction).

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

**7.1.5 Hydrochloric acid solution (1+3)**. Mix one part hydrochloric acid and three parts water (7.1.1).

**7.1.6 Nitric acid solution (1+3)**. Mix one part nitric acid and three parts water (7.1.1).

### 7.2 Apparatus and instruments

Ordinary laboratory apparatus together with the following shall be used:

**7.2.1 Hot plate**, capable of heating at 400 °C.

**7.2.2 PTFE (Polytetrafluoroethylene) beakers (25 ml) and covers.**

**7.2.3 Platinum dishes (20 ml or 30 ml) and covers.**

**7.2.4 PP (Polypropylene) volumetric flask (10 ml).**

### **7.3 Sample decomposition**

Weigh 1,0 g of the test sample and transfer it to a PTFE beaker (7.2.2) or platinum dishes (7.2.3). Add 15 ml of hydrofluoric acid (7.1.2) and 3 ml of nitric acid (7.1.3). Cover the beaker with a PTFE cover and heat gently on a hot plate. After heating for 1\_h, open the PTFE cover and continue to heat gently on a hot plate to evaporate to dryness. Cool to room temperature.

For determination of Al, Fe, Ti and Mn, add 8 ml of hydrochloric acid solution (7.1.5) and heat on a hot plate to dissolve the solid. For determination of Cd, Ca, Cu, Pb, Li, Mg, Ni, K, Na, Zn and Zr, add 8 ml of nitric acid solution (7.1.6) and heat on a hot plate to dissolve the solid.

After cooling, transfer the solution to a 10 ml PP volumetric flask (7.2.4). Rinse the inner wall of the PTFE beaker with a small amount of water and pour the washings into the flask. Dilute with water up to the mark and mix well. This solution is designated as the sample solution.

### **7.4 Blank test**

To obtain the blank test value, perform the procedure described in 7.3 without taking the sample.

## **8 Determination of impurity elements**

### **8.1 Reagents**

Use reagents of analytical grade.

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

**8.1.2 Hydrochloric acid solution (1+4)**. Mix one part hydrochloric acid with four parts water (7.1.1).

**8.1.3 Nitric acid solution (1+4)**. Mix one part hydrochloric acid with four parts water (7.1.1).

#### **8.1.4 Elemental standard solutions.**

- a) Aluminium standard solution (Al 1 mg/ml).
- b) Cadmium standard solution (Cd 1 mg/ml).
- c) Calcium standard solution (Ca 1 mg/ml).
- d) Copper standard solution (Cu 1 mg/ml).
- e) Iron standard solution (Fe 1 mg/ml).
- f) Lead standard solution (Pb 1 mg/ml).
- g) Lithium standard solution (Li 1 mg/ml).