INTERNATIONAL STANDARD

ISO 5189

First edition

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

PROOF/ÉPREUVE

Reference number
ISO 5189:2023(E)

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

Published in Switzerland

Contents

Page

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 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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.

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 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 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

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_3), (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).

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

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

j) Nickel standard solution (Ni 1 mg/ml).

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

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

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

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

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

NOTE The SI-traceable commercial standard solutions are available.

8.1.5 Mixed standard solution (each element 5 mg/L).

Transfer 500 µl of each standard solution (described in 8.1.4) into a 100 ml volumetric flask. Dilute with water up to the mark and mix well. Pay attention to ensure that no precipitation occurs during mixing. Prepare the solution before every use. Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

8.2 Apparatus and instruments

Ordinary laboratory apparatus together with the following shall be used:

8.2.1 ICP-OES.

8.3 Measurement

Spray a portion of the mixed standard solution (8.1.4) into the plasma of ICP-OES and measure the emission intensity at an appropriate wavelength (Table 2). Interferences can be encountered. Therefore, carefully choose the optimum wavelength such that it is free from overlapping peaks.

Table 2 — Examples of the analytical wavelength for each element

Element	Wavelength 1 nm	Wavelength 2 nm
Al	396,153	308,215
Cd	228,802	214,440
Ca	317,933	396,847
Cu	327,393	324,752
Fe	238,204	239,562
Pb	220,353	217,000
Li	670,784	610,362
Mg	285,213	279,077
Mn	257,610	259,372
Ni	231,604	232,003
K	766,490	—
Na	589,592	—
Ti	337,279	336,121
Zn	206,200	213,857
Zr	343,823	339,197

8.4 Drawing of the calibration curve

Transfer 1 ml, 5 ml, 10 ml, 15 ml and 20 ml of the mixed standard solution (8.1.5) stepwise to 100 ml volumetric flasks. For determination of Al, Fe, Ti and Mn, dilute the contents of each flask with hydrochloric acid solution (8.1.2) up to the mark and mix well. For determination of Cd, Ca, Cu, Pb, Li, Mg, Ni, K, Na, Zn and Zr, dilute the contents of each flask with nitric acid solution (8.1.3) up to the mark and mix well. Spray a portion of each solution into the plasma of ICP-OES and measure the emission intensity at an appropriate wavelength.

8.5 Calculation

Determine the concentration of each element in the test solution and in the blank solution from the calibration curve. Calculate the element content using Formula (1).

$$W_i = (m_i - m_0) \times 10 / m \quad (1)$$

where

W_i is each element content, in mg/kg;

m_i is the concentration of each element in the test solution, in mg/L;

m_0 is the concentration of each element in the blank solution, in mg/L;

m is the mass of the test portion, 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;

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

d) any deviations from the specified procedure;

e) any unusual features noted during the determination;

f) any procedures not specified in this document or any optional procedure that could have impacted the results.

