
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Methods for chemical analysis of
aluminium nitride powders**

*Céramiques techniques — Méthodes d'analyse chimique des poudres
de nitrure d'aluminium*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 21814:2019](https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-9e5e33a0db3e/iso-21814-2019)

<https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-9e5e33a0db3e/iso-21814-2019>



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 21814:2019

<https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-9e5e33a0db3e/iso-21814-2019>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

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
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

	Page
Foreword	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Analytes and ranges	2
5 Preparation of the test sample	2
5.1 General.....	2
5.2 Sampling.....	3
5.3 Drying.....	3
5.4 Weighing.....	3
6 Reporting the analytical values	3
6.1 Number of analyses.....	3
6.2 Blank test.....	3
6.3 Evaluation of the analytical values.....	3
6.4 Expression of the analytical values.....	3
7 Determination of the aluminium content	4
7.1 Classification of the determination methods.....	4
7.2 Acid decomposition-ICP-OES method.....	4
7.2.1 Principle.....	4
7.2.2 Reagents.....	4
7.2.3 Apparatus and instruments.....	4
7.2.4 Procedure.....	5
7.2.5 Blank test.....	5
7.2.6 Drawing of the calibration curve.....	5
7.2.7 Calculation.....	5
7.3 Acid pressure decomposition-CyDTA-zinc back titration method.....	5
7.3.1 Principle.....	5
7.3.2 Reagents.....	6
7.3.3 Apparatus and instruments.....	6
7.3.4 Procedure.....	8
7.3.5 Blank test.....	8
7.3.6 Calculation.....	8
8 Determination of the total nitrogen content	8
8.1 Classification of the determination methods.....	8
8.2 Acid pressure decomposition-distillation separation-acidimetric titration method.....	9
8.2.1 Principle.....	9
8.2.2 Reagents.....	9
8.2.3 Apparatus.....	10
8.2.4 Procedure.....	12
8.2.5 Recovery measurement.....	12
8.2.6 Calculation.....	13
8.3 Direct decomposition-distillation separation-acidimetric titration method.....	13
8.3.1 Principle.....	13
8.3.2 Reagents.....	13
8.3.3 Apparatus.....	13
8.3.4 Procedure.....	13
8.3.5 Recovery measurement.....	13
8.3.6 Calculation.....	13
8.4 Inert gas fusion-thermal conductivity method.....	13
8.4.1 Principle.....	13
8.4.2 Reagents.....	14
8.4.3 Apparatus.....	14

8.4.4	Instrument	14
8.4.5	Procedure	15
8.4.6	Blank test	16
8.4.7	Calculation of the calibration coefficient	16
8.4.8	Calculation	17
9	Determination of the sodium and potassium contents	17
9.1	Classification of the determination methods	17
9.2	Acid pressure decomposition-flame emission method	17
9.2.1	Principle	17
9.2.2	Reagents	17
9.2.3	Instruments	18
9.2.4	Procedure	18
9.2.5	Blank test	18
9.2.6	Drawing of the calibration curve	18
9.2.7	Calculation	18
9.3	Acid pressure decomposition-atomic absorption spectrometry method	19
9.3.1	Principle	19
9.3.2	Reagents	19
9.3.3	Instruments	19
9.3.4	Procedure	19
9.3.5	Blank test	19
9.3.6	Drawing of the calibration curve	19
9.3.7	Calculation	19
10	Determination of the trace element contents	19
10.1	Classification of the determination methods	19
10.2	Acid decomposition-ICP-OES method	20
10.2.1	Principle	20
10.2.2	Reagents	20
10.2.3	Apparatus and instruments	20
10.2.4	Procedure	21
10.2.5	Blank test	21
10.2.6	Drawing of the calibration curve	21
10.2.7	Calculation	21
10.3	Acid pressure decomposition-ICP-OES method	22
10.3.1	General	22
10.3.2	Reagents	22
10.3.3	Apparatus and instruments	22
10.3.4	Procedure	22
10.3.5	Blank test	22
10.3.6	Drawing of the calibration curve	22
10.3.7	Calculation	22
11	Determination of the oxygen content	22
11.1	Principle	22
11.2	Reagents	22
11.3	Apparatus	23
11.4	Instruments	23
11.5	Procedure	23
11.6	Blank test	23
11.7	Calculation of the calibration coefficient	23
11.8	Calculation	23
12	Determination of the carbon content	24
12.1	Classification of the determination methods	24
12.2	Combustion (resistance furnace)-IR absorption spectrometry	24
12.2.1	Principle	24
12.2.2	Reagents	24
12.2.3	Apparatus	24

12.2.4	Instrument	24
12.2.5	Procedure	25
12.2.6	Blank test	26
12.2.7	Calculation of the calibration coefficient	26
12.2.8	Calculation	27
12.3	Combustion (radio frequency heating furnace)-thermal conductometry	27
12.3.1	Principle	27
12.3.2	Reagents	27
12.3.3	Apparatus	27
12.3.4	Instrument	27
12.3.5	Procedure	28
12.3.6	Blank test	29
12.3.7	Calculation of the calibration coefficient	29
12.3.8	Calculation	29
12.4	Combustion (radio frequency heating furnace)-IR absorption spectrometry	29
12.4.1	Principle	29
12.4.2	Reagents	29
12.4.3	Apparatus	29
12.4.4	Instrument	29
12.4.5	Procedure	30
12.4.6	Blank test	31
12.4.7	Calculation of the calibration coefficient	31
12.4.8	Calculation	31
13	Determination of the fluorine and chlorine contents	31
13.1	Principle	31
13.2	Reagents	31
13.3	Apparatus and instruments	32
13.4	Procedure	33
13.4.1	Extraction of fluorine and chlorine from the sample	33
13.4.2	Determination of the fluorine and chlorine contents	33
13.5	Blank test	33
13.6	Drawing of the calibration curve	34
13.7	Calculation	34
14	Test report	34
Annex A (informative) Analytical results obtained from the round robin test		35
Bibliography		36

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).

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).

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

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html. (standards.iteh.ai)

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 aluminium nitride powders

1 Scope

This document specifies methods for the chemical analysis of fine aluminium nitride powders used as the raw material for fine ceramics.

This document stipulates the determination methods of the aluminium, total nitrogen, boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium, zinc, zirconium, carbon, chlorine, fluorine, and oxygen contents in aluminium nitride powders. The aluminium content is determined by using either an acid pressure decomposition-CyDTA-zinc back titration method or an acid digestion-inductively coupled plasma-optical emission spectrometry (ICP-OES) method. The total nitrogen content is determined by using an acid pressure decomposition-distillation separation-acidimetric titration method, a direct decomposition-distillation separation-acidimetric titration method, or an inert gas fusion-thermal conductivity method. The boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium and zinc contents are determined by using an acid digestion-ICP-OES method or an acid pressure decomposition-ICP-OES method. The sodium and potassium contents are determined via an acid pressure decomposition-flame emission method or an acid pressure decomposition-atomic absorption spectrometry method. The oxygen content is determined by using an inert gas fusion-IR absorption spectrometry method, while that of carbon is determined via a combustion-IR absorption spectrometry method or a combustion-conductometry method. The chlorine and fluorine contents are determined by using a pyrohydrolysis method followed by ion chromatography or spectrophotometry.

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 2828, *Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method*

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*

ISO 21068-3:2008, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 3: Determination of nitrogen, oxygen and metallic and oxidic constituents*

ISO 21438-2, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 2: Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)*

ISO 21438-3, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 3: Hydrofluoric acid and particulate fluorides*

ISO 26845:2008, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

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) Aluminium (Al), range of 40 % to 70 % (mass fraction).
- b) Total nitrogen (T.N), range of 20 % to 40 % (mass fraction).
- c) Boron (B), range of 0,001 % to 0,03 % (mass fraction).
- d) Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction).
- e) Copper (Cu), range of 0,001 % to 0,03 % (mass fraction).
- f) Iron (Fe), range of 0,001 % to 0,03 % (mass fraction).
- g) Magnesium (Mg), range of 0,001 % to 0,03 % (mass fraction).
- h) Manganese (Mn), range of 0,001 % to 0,03 % (mass fraction).
- i) Molybdenum (Mo), range of 0,001 % to 0,03 % (mass fraction).
- j) Nickel (Ni), range of 0,001 % to 0,03 % (mass fraction).
- k) Potassium (K), range of 0,001 % to 0,03 % (mass fraction).
- l) Silicon (Si), range of 0,001 % to 0,03 % (mass fraction).
- m) Sodium (Na), range of 0,001 % to 0,03 % (mass fraction).
- n) Titanium (Ti), range of 0,001 % to 0,03 % (mass fraction).
- o) Tungsten (W), range of 0,001 % to 0,03 % (mass fraction).
- p) Vanadium (V), range of 0,001 % to 0,03 % (mass fraction).
- q) Zinc (Zn), range of 0,001 % to 0,03 % (mass fraction).
- r) Carbon (C), range of 0,01 % to 6 % (mass fraction).
- s) Chlorine (Cl), range of 0,001 % to 0,5 % (mass fraction).
- t) Fluorine (F), range of 0,001 % to 0,2 % (mass fraction).
- u) Oxygen (O), range of 0,05 % to 5 % (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 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 (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 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.

6.4 Expression of the analytical values

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

- Aluminium, total nitrogen, and oxygen: express the results in four figures to two decimal places.
- Others: express the results to three decimal places.

Table 1 — Tolerances for the analytical values

Units: % (mass fraction)

Component	Al	Total N	Si, Ti, Fe, Ca, Mg, V, Mo, W, Cu, Ni, Zn, Mn, B, Na, K, F, Cl	O	C
Tolerance	0,20 ^a 0,30 ^b	0,20 ^c 0,40 ^d	0,001 ^e 0,005 ^f	0,05	0,005
^a Acid pressure decomposition-CyDTA-zinc back titration method. ^b Acid decomposition-ICP-OES method. ^c Acid pressure decomposition (or direct decomposition)-distillation separation-acidimetric titration method. ^d Inert gas fusion-thermal conductivity method. ^e Applicable to content of less than 0,01 % (mass fraction). ^f Applicable to content of not less than 0,01 % (mass fraction).					

7 Determination of the aluminium content

7.1 Classification of the determination methods

Method A, acid decomposition-ICP-OES method.

Method B, acid pressure decomposition-CyDTA-zinc back titration method.

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

7.2 Acid decomposition-ICP-OES method

7.2.1 Principle

A portion of the sample is decomposed in water, sulfuric acid, and hydrogen peroxide. After making up to the required volume, the emission intensity of the aluminium present in the test solution is measured by ICP-OES at one or more of the wavelengths: 396,15 nm, 309,28 nm and 394,40 nm.

7.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

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

7.2.2.2 **Sulfuric acid (1+9, 1+180).** (standards.iteh.ai)

7.2.2.3 **Hydrochloric acid (1+1, 1+3).** [ISO 21814:2019](https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-0e5e3370113e/iso-21814-2019)
<https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-0e5e3370113e/iso-21814-2019>

7.2.2.4 **Aluminium stock solution (Al 10 mg/ml).** <https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-0e5e3370113e/iso-21814-2019>

Wash the surface of the aluminium (more than 99,999 % purity by mass fraction) with a hydrochloric acid solution (1+3). Wash the oxidised layer with water, ethanol (99,5 %), and diethyl ether. Dry the aluminium in a desiccator (desiccant: magnesium perchlorate). Weigh 5 g aluminium in a PTFE beaker and cover with a PTFE watch glass. Add 50 ml hydrochloric acid solution (1+1) and heat to dissolve on a steam bath. After cooling, transfer the solution to a 500 ml volumetric flask, dilute with water to the mark and mix well.

NOTE The SI traceable commercial standard solution is also available.

7.2.2.5 **Aluminium standard solution (Al 1 mg/ml).**

Place 10 ml aluminium stock solution ([7.2.2.4](#)) in a 100 ml plastic volumetric flask. Dilute with sulfuric acid (1+180) to the mark and mix well.

7.2.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for the chemical analyses in accordance with ISO 26845:2008, Clause 4.

7.2.3.1 **ICP-OES.**

7.2.4 Procedure

7.2.4.1 Sample decomposition

Weigh 0,30 g test sample and transfer it into a 250 ml beaker. Add 15 ml water, 20 ml sulfuric acid, and 10 ml hydrogen peroxide. Covering the beaker with a watch glass and heat at 280 °C until the test sample has been completely dissolved. After cooling, transfer the solution to a 500 ml volumetric flask, dilute with water to the mark and mix well.

NOTE If the test sample is not completely dissolved, add a little more hydrogen peroxide and continue heating to dissolve the sample. Alternatively, you may apply the acid pressure decomposition method (7.3).

7.2.4.2 Measurement

Transfer a 10 ml aliquot of the stock solution to a 100 ml volumetric flask, dilute with water to the mark and mix well. Spray a portion of the test solution into the flame of the ICP-OES and measure the emission intensity for aluminium at one or more of the wavelengths: 396,15 nm, 309,28 nm and 394,40 nm.

7.2.5 Blank test

Perform the operation described in 7.2.4 without taking a sample to obtain the blank test value.

7.2.6 Drawing of the calibration curve

Transfer 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml aliquots of the aluminium standard solution (7.2.2.5) separately to six 100 ml volumetric flasks. To each flask add 10 ml sulfuric acid (1+10), dilute with water to the mark and mix well.

Spray a portion of each solution into the flame of the ICP-OES and measure the emission intensity for aluminium at one or more of wavelengths: 396,15 nm, 309,28 nm and 394,40 nm. Interferences may be encountered. Carefully choose the optimum wavelength that is free from concomitants.

7.2.7 Calculation

Determine the concentration of the aluminium in the test solution and blank from the calibration curve. Calculate the aluminium content, Al , expressed as a percent mass fraction, from Formula (1).

$$Al = \left[(A - A_0) / m \right] \times 500 / 10 \times 100 \quad (1)$$

where

Al is the aluminium content, in % (mass fraction);

A is the mass of aluminium in the test solution, in g;

A_0 is the mass of aluminium in the blank solution, in g;

m is the mass of the sample, in g.

7.3 Acid pressure decomposition-CyDTA-zinc back titration method

7.3.1 Principle

A sample is pressure-decomposed with sulfuric acid in a decomposition vessel, to which excess CyDTA solution is added. After adjusting the pH with hexamethylenetetramine, the solution is titrated with a zinc standard solution using xylenol orange as an indicator.

7.3.2 Reagents

Reagents of analytical grade shall be used. The reagent solutions shall be preserved in plastic bottles. Use the reagents described in 7.2.2 together with the following:

7.3.2.1 Hexamethylenetetramine solution (200 g/l).

7.3.2.2 CyDTA solution (0,02 mol/l).

Add 16 ml sodium hydroxide solution (100 g/l) and 300 ml water to 7,3 g cyclohexanediaminetetraacetic acid (hydrate) and heat until dissolved. After cooling, transfer the solution to a 1 000 ml volumetric flask, dilute with water to the mark and mix well.

7.3.2.3 Zinc standard solution (0,02 mol/l).

7.3.2.4 Xylenol orange solution (1 g/l).

7.3.2.5 Sulfuric acid (1+2).

7.3.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for the chemical analyses in accordance with ISO 26845:2008, Clause 4.

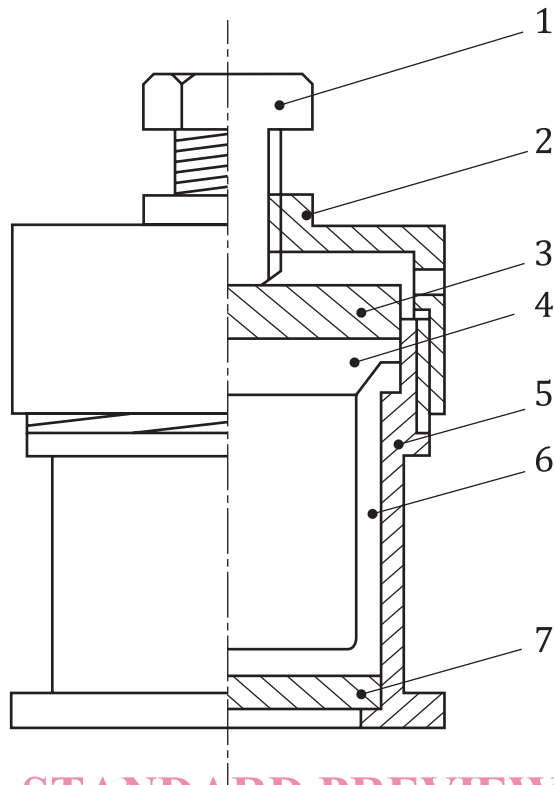
iTeh STANDARD PREVIEW

7.3.3.1 Platinum crucible (20 ml). (standards.iteh.ai)

7.3.3.2 **Pressure decomposition vessel**, in general a commercial pressure decomposition vessel may be used.

ISO 21814:2019
<https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-0e533a04b3e1/iso-21814-2019>

An example is presented in [Figure 1](#). Use the vessels exclusively for this analysis to avoid cross-contamination.



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 21814:2019

<https://standards.iteh.ai/catalog/standards/sist/bfb7be56-4b60-4e68-b67c-9e5e33a0db3e/iso-21814-2019>

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 sealed decomposition vessel