
**Chemical analysis of aluminosilicate
refractory products (alternative to the
X-ray fluorescence method) —**

**Part 1:
Apparatus, reagents, dissolution and
gravimetric silica**

*Analyse chimique des produits réfractaires d'aluminosilicates (méthode
alternative à la méthode par fluorescence de rayons X) —*

*Partie 1: Appareillage, réactifs, dissolution et teneur en silice par
gravimétrie*



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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 21587-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21587 consists of the following parts, under the general title *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents, dissolution and gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Inductively coupled plasma and atomic absorption spectrometry methods*

Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) —

Part 1:

Apparatus, reagents, dissolution and gravimetric silica

1 Scope

This part of ISO 21587 specifies reagents, dissolution and gravimetric silica analysis for the chemical analysis of aluminosilicate refractory products and raw materials.

This part of ISO 21587 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677:2003, *Chemical analysis of refractory products by XRF — Fused cast bead method*.

This part of ISO 21587 should be used in conjunction with ISO 21587-2 and ISO 21587-3, which give the analytical procedures for the determination of the following:

- silicon(IV) oxide (SiO_2)
- aluminium oxide (Al_2O_3)
- iron(III) oxide (total iron oxide calculated as Fe_2O_3)
- titanium(IV) oxide (TiO_2)
- manganese(II) oxide (MnO)
- calcium oxide (CaO)
- magnesium oxide (MgO)
- sodium oxide (Na_2O)
- potassium oxide (K_2O)
- chromium(III) oxide (Cr_2O_3)
- zirconium oxide (ZrO_2)
- phosphorous(V) oxide (P_2O_5)

2 Normative references

The following referenced documents are indispensable for the application 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 21587-2, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 21587-3, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 3: Inductively coupled plasma and atomic absorption spectrometry methods*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry and inductively coupled plasma methods*

3 Reagents

Standard solutions specified in ISO 26845 and the following reagents.

3.1 Standard volumetric solutions

3.1.1 Standard volumetric CyDTA solution, $c(\text{CyDTA}) = 0,05 \text{ mol/l}$.

Dissolve 18 g of 1,2 cyclohexanediamine-N,N,N',N'-tetraacetic acid monohydrate (CyDTA) in 500 ml of water by the progressive addition of the minimum amount of potassium hydroxide solution.

NOTE Approximately 25 ml is required. Determine the exact strength of this solution by titration against the standard volumetric zinc solution, $c(\text{Zn}) = 0,05 \text{ mol/l}$.

3.1.2 Standard volumetric CyDTA solution, $c(\text{CyDTA}) = 0,02 \text{ mol/l}$.

Add 16 ml of sodium hydroxide solution (100 g/l) and approximately 150 ml of water to 7,30 g of 1,2-cyclohexanediamine-N,N,N',N'-tetraacetic acid monohydrate (CyDTA), and dissolve by heating. After cooling, dilute to 1 000 ml with water.

NOTE Approximately 25 ml is required. Determine the exact strength of this solution by titration against the standard volumetric zinc solution, $c(\text{Zn}) = 0,02 \text{ mol/l}$.

3.1.3 Standard volumetric EDTA solution, $c(\text{EDTA}) = 0,012 5 \text{ mol/l}$.

Dissolve 5 g of EDTANa_2 (ethylenediamine-tetraacetic acid disodium salt, dihydrate) in water and dilute to 1 000 ml in a volumetric flask. Store in a plastic bottle.

Standardize against calcium as follows.

Pipette 25 ml of standard calcium oxide solution (1 mg/ml), into a 500 ml conical flask, add 10 ml of potassium hydroxide solution, and dilute to about 200 ml. Add about 0,015 g of screened Calcein indicator, and titrate with the standard volumetric EDTA solution, from a fluorescent green colour to pink.

Standardize against magnesium as follows.

Pipette 25 ml of standard magnesium oxide solution (1 mg/ml), into a 500 ml conical flask, add 20 drops of hydrochloric acid (concentrated) and 20 ml of ammonia solution (concentrated), and dilute to about 200 ml. Add about 0,04 g of methylthymol blue complexone indicator, and titrate with the standard volumetric EDTA solution.