
**Chemical analysis of refractories
containing alumina, zirconia and silica —
Refractories containing 5 % to 45 %
of ZrO₂ (alternative to the X-ray
fluorescence method) —**

Part 1:

Apparatus, reagents and dissolution

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*Analyse chimique des matériaux réfractaires contenant de l'alumine,
de la zirconie et de la silice — Matériaux réfractaires contenant de 5 %
à 45 % de ZrO₂ (méthode alternative à la méthode par fluorescence de
rayons X)*

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ISO 21079-1:2008

Partie 1: Appareillage, réactifs et dissolution



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 21079-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21079 consists of the following parts, under the general title *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents and dissolution* [ISO 21079-1:2008](#)
- *Part 2: Wet chemical analysis* <https://standards.iteh.ai/catalog/standards/sist/2e472763-38ba-4b0d-9590-6dbacf37e433/iso-21079-1-2008>
- *Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method) —

Part 1: Apparatus, reagents and dissolution

1 Scope

This part of ISO 21079 specifies methods for the chemical analysis of AZS (alumina, zirconia, and silica) refractory products (containing 5 % to 45 % of ZrO₂) and raw materials, using traditional (“wet”) methods, inductively coupled plasma atomic emission (ICP-AE) spectrometry and flame atomic absorption (FAA) spectrometry. It covers apparatus, reagents and dissolution methods.

The range of determination is given in Table 1.

Table 1 — Range of determination (% by mass)

Component	Range	Component	Range
LOI	1 to 40	MgO	0,01 to 2
SiO ₂	0,1 to 45	Na ₂ O	0,01 to 3
Al ₂ O ₃	1 to 80	K ₂ O	0,01 to 1
Fe ₂ O ₃	0,01 to 2	Cr ₂ O ₃	0,01 to 3
TiO ₂	0,01 to 5	ZrO ₂	5 to 45
CaO	0,01 to 2	HfO ₂	0,01 to 2
NOTE The values other than LOI are on an ignited basis.			

This part of ISO 21079 is not applicable to MgO-based refractories.

NOTE This part of ISO 21079 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

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 26845, *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

For the purposes of this document, the terms and definitions given in ISO 26845 apply.

4 Apparatus

Ordinary laboratory apparatus and the apparatus given in ISO 26845.

5 Reagents

5.1 General

Prepare the following reagents, and those given in ISO 26845, as necessary.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Reagents should conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3, as appropriate.

5.2 Standard solutions specific to ISO 21079

Traceable standard solutions may be used in place of the following

5.2.1 Aluminium oxide standard solution, Al_2O_3 1 mg/ml

Wash the surface of a sufficient amount of aluminium metal (purity of more than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol and diethyl ether in succession, and dry in a desiccator. Weigh 0,529 2 g of aluminium and transfer into a 250 ml beaker. Cover with a watch glass, add 20 ml of hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml in a volumetric flask with water.

5.2.2 Calcium oxide standard solution, CaO 1 mg/ml.

Dissolve 1,785 g of pure calcium carbonate, previously dried at 150 °C, in a slight excess of dilute hydrochloric acid (1+4) in a 250 ml beaker, covered with a watch glass. Boil to expel carbon dioxide, cool and dilute to 1 000 ml in a volumetric flask.

5.2.3 Chromium(III) oxide standard solution, Cr_2O_3 1 mg/ml.

Dry about 2 g to 3 g of potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, at 110 °C. for at least 2 h. Weigh 1,935 g of this and dissolve in water, diluting to 1 000 ml in a volumetric flask.

5.2.4 Diluted chromium(III) oxide standard solution, Cr_2O_3 0,025 mg/ml.

Dilute 25 ml of the chromium(III) oxide standard solution (1 mg/ml), to 1 000 ml in a volumetric flask. Prepare this solution freshly when required.

5.2.5 CyDTA (trans-1,2-cyclohexanediamine-*N,N,N',N'*-tetracetic acid monohydrate) standard solution, 0,02 mol/l.

Add 16 ml of sodium hydroxide solution (100 g/l) and 150 ml of water to 7,30 g of CyDTA, and dissolve by heating. After cooling, dilute to 1 000 ml with water.

5.2.6 EDTA standard solution, 5 g/l.

Dissolve 5 g of EDTA Na_2 (ethylenediamine-tetraacetic acid disodium salt, dihydrate) in water and dilute to 1 000 ml in a volumetric flask. Store in a plastics bottle.

Standardize against calcium as follows.

Pipette 25 ml of standard calcium 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 EDTA solution (5 g/l), from a fluorescent green colour to pink.

Standardize against magnesium as follows.

Pipette 25 ml of standard magnesium 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 EDTA solution (5 g/l).

5.2.7 Hafnium oxide standard solution, HfO₂ 1 mg/ml.

Transfer 1,5 g of hafnium oxide (of a purity more than 99,9 % by mass) into a 20 ml platinum crucible, ignite at 1 050 °C \pm 25 °C for 1 h and cool in a desiccator. Weigh 0,500 0 g of the ignited residue into a 75 ml platinum crucible. After adding 3,0 g of anhydrous sodium carbonate, add 2,0 g of boric acid and mix. Then carry out melting at 1 050 \pm 25 °C in an electric muffle furnace. After cooling, add 55 ml of sulfuric acid (1+9) and heat until dissolved on a steam bath. After cooling, dilute to 500 ml in a flask with water.

5.2.8 Iron(III) oxide standard solution, Fe₂O₃ 1mg/ml.

Wash the surface of a sufficient amount of iron metal (purity greater than 99,9 %) with hydrochloric acid (1+4). Then dissolve the oxidized layer, and wash with water, ethanol and diethyl ether in succession. Then dry in a desiccator. Weigh 0,699 4 g of this, transfer to a beaker (200 ml), and cover with a watch glass. Add 30 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, dilute to 1 000 ml in a volumetric flask with water.

5.2.9 Diluted iron(III) oxide standard solution, Fe₂O₃ 0,04 mg/ml.

Transfer 40 ml of the iron(III) oxide (1 mg/ml) standard solution to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly as required.

5.2.10 Magnesium oxide standard solution, MgO 1 mg/ml.

Wash the surface of a sufficient amount of magnesium metal (purity, more than 99,9 % by mass) with hydrochloric acid (1+1) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession and dry in a desiccator. Weigh 0,301 5 g of the washed magnesium, transfer to a 200 ml beaker and cover with a watch glass. Add 10 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.11 Potassium oxide standard solution, K₂O 1 mg/ml.

Transfer 1 g to 1,5 g of potassium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C \pm 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,791 4 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.12 Silicon(IV) oxide standard solution, SiO₂ 1 mg/ml.

Weigh 0,7 g to 1,0 g of silicon(IV) oxide (purity, greater than 99,9 % by mass) in a platinum crucible (e.g. 30 ml) and heat for 30 min at 1 150 °C ± 50 °C. Cool in a desiccator and then weigh 0,500 0 g of this silicon(IV) oxide into a platinum crucible (e.g. 30 ml). Fuse the silicon(IV) oxide with 3,0 g of anhydrous sodium carbonate. Cool and wipe the outside of the crucible, and dissolve in warm water (150 ml) in a plastic 200 ml beaker while stirring using a plastic rod. Cool and dilute without heating to 500 ml in a volumetric flask. Transfer this solution to a plastic bottle immediately.

5.2.13 Diluted silicon(IV) oxide standard solution, SiO₂ 0,04 mg/ml.

Dilute 10 ml of silicon(IV) oxide standard solution (1,0 mg/ml) to 250 ml with water.

5.2.14 Sodium oxide standard solution, Na₂O 1 mg/ml.

Transfer 1 to 1,5 g of sodium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C ± 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,942 9 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.15 Titanium(IV) oxide standard solution, TiO₂ 1,0 mg /ml.

Weigh 0,599 4 g of titanium (of a purity more than 99,9 % by mass) on a 100 ml platinum dish and cover with a watch glass made of polyethylene tetrafluoride resin. Add 20 ml of hydrofluoric acid, 15 ml of sulfuric acid (1+1) and 0,5 ml of nitric acid. Heat until dissolved on a steam bath. Remove the watch glass and rinse the watch glass with water. Heat the solution on a sand bath until sulfuric acid fumes can be detected. After cooling, rinse the inner wall of the platinum with a small amount of water and heat again until fumes are detectable. After cooling, add water and dilute to 1 000 ml in a volumetric flask with water.

5.2.16 Diluted titanium(IV) oxide standard solution, TiO₂ 0,01 mg/ml.

Dilute 10 ml of titanium(IV) oxide standard solution (1,0 mg/ml) to 1 000 ml with water.

5.2.17 Zinc standard solution, Zn 0,02 mol/l.

Wash the surface of the zinc (of a purity more than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer. Wash with water, ethanol and diethyl ether in succession, and dry in a desiccator. Weigh (1,307 4 ± 0,1) mg of zinc, cover with a watch glass and transfer it to a 300 ml beaker. Add 20 ml of water, carefully add 10 ml of nitric acid and heat until dissolved on a steam bath. After cooling, dilute to 1 000 ml in a volumetric flask with water.

Calculate the factor, *F*, of the 0,02 mol/l zinc solution using Equation (1).

$$F = \frac{m}{0,6537} \times \frac{A}{100} \tag{1}$$

where

m is the mass of the weighed zinc, in grams;

A is the purity of the zinc, as a percentage by mass.

6 Sampling

Prepare the sample as described in ISO 26845.

7 General procedures

Carry out the chemical analysis and a blank test as specified in ISO 26845.

8 Determination of loss on ignition

Determine the loss on ignition as specified in ISO 26845.

9 Decomposition of the sample and preparation of solutions used in the analysis

9.1 General decomposition techniques

The techniques used in this standard method are traditional “wet” methods, flame photometry, atomic absorption spectrometry and ICP-AE spectrometry. To meet the needs of these procedures five separate decompositions are required.

Carry out one or more of the decompositions given in the following list, depending on the requirements of the particular technique(s) chosen:

- a) decomposition by fusion followed by removal of the bulk of the silica present by dehydration, as described in 9.2.2;
- b) decomposition by fusion followed by removal of the bulk of the silica present by coagulation, as described in 9.2.3;
- c) decomposition by fusion and dissolution in sulfuric acid, as described in 9.2.4;
- d) decomposition and removal of the silica by hydrofluoric acid for flame photometry, as described in 9.3.2;
- e) decomposition and removal of the silica by hydrofluoric acid for atomic absorption spectroscopy, as described in 9.3.3;
- f) decomposition and removal of the silica by hydrofluoric acid for ICP-AE spectrometry, as described in 9.3.4.

9.2 Dissolution methods by fusion

9.2.1 General

Prepare a solution for the determination of silicon(IV) oxide in solution, aluminium oxide, ferric oxide, titanium oxide, calcium oxide, magnesium oxide, chromium(III) oxide, zirconium oxide, and hafnium oxide by one of the following methods:

- a) fusion with alkali carbonate and boric acid, dissolution of the melt in hydrochloric acid and removal of the bulk of the silicon(IV) oxide by dehydration;
- b) fusion with alkali carbonate and boric acid, dissolution of the melt in hydrochloric acid and removal of the bulk of the silicon(IV) oxide by coagulation;
- c) fusion with alkali carbonate and boric acid and dissolution of the melt in sulfuric acid.