

Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 percent to 45 percent of ZrO_2 (alternative to the X-ray fluorescence method)

**Part 2: Wet chemical analysis (ISO
21079-2:2008)**

ICS 81.080

National foreword

This British Standard is the UK implementation of EN ISO 21079-2:2008.

The UK participation in its preparation was entrusted to Technical Committee RPI/1, Refractory products and materials.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Analyse chimique des matériaux réfractaires contenant de
l'alumine, de la zircone et de la silice - Matériaux
réfractaires contenant de 5 % à 45 % de ZrO₂ (méthode
alternative à la méthode par fluorescence de rayons X) -
Partie 2: Méthodes d'analyse chimique par voie humide
(ISO 21079-2:2008)

Chemische Analyse von aluminiumoxid-, zirkoniumoxid-
und silicium(IV)-oxidhaltigen feuerfesten Erzeugnissen -
Feuerfeste Erzeugnisse mit einem Massenanteil an ZrO₂
von 5 % bis 45 % (Alternative zum
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Management Centre: rue de Stassart, 36 B-1050 Brussels

Foreword

This document (EN ISO 21079-2:2008) has been prepared by Technical Committee ISO/TC 33 "Refractories" in collaboration with Technical Committee CEN/TC 187 "Refractory products and materials" the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2008, and conflicting national standards shall be withdrawn at the latest by October 2008.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 21079-2:2008 has been approved by CEN as a EN ISO 21079-2:2008 without any modification.

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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.

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ISO 21079-2 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*
- *Part 2: Wet chemical analysis*
- *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 ZrO_2 (alternative to the X-ray fluorescence method) —

Part 2: Wet chemical analysis

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 ZrO_2) and raw materials, using traditional (“wet”) methods.

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

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 10058-2:—¹⁾, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 21079-1:2008, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO_2 (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents and dissolution*

ISO 21079-3:2008, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO_2 (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 21587-2:2007, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

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*

1) To be published.

3 Determination of silicon(IV) oxide

3.1 General

The determination of silicon(IV) oxide is carried out using one of the following methods.

- a) Combined use of the dehydration or the coagulation and molybdenum blue methods. This method is applied to samples consisting of more than 4 % by mass of silicon(IV) oxide.
- b) Molybdenum blue method. This method is applied to samples consisting of less than 8 % by mass of silicon(IV) oxide.

3.2 Combined use of dehydration or coagulation and molybdenum blue methods

3.2.1 Principle

An aliquot portion of stock solution (S1) or (S'1) is treated with ammonium molybdate and the silicomolybdate is reduced to yield molybdenum blue, the absorbance of which is measured.

The sum of this residual silicon(IV) oxide in solution plus the mass of silicon(IV) oxide ($m_1 - m_2$) derived in accordance with 9.2.2.3 or 9.2.3.3 of ISO 21079-1:2008 gives the total silicon(IV) oxide content.

3.2.2 Procedure

This determination should be commenced with little delay after the stock solution (S1) or (S'1) is prepared, as prolonged standing could allow polymerization of silica to occur leading to low results.

Transfer a 10,0 ml aliquot portion of stock solution (S1) or (S'1) (see Annex A of ISO 21079-1:2008) to a 100 ml plastic beaker, add 2 ml of hydrofluoric acid (1+9), mix with a plastic rod and allow to stand for 10 min. Add 50 ml of boric acid solution, 2 ml of ammonium molybdate solution while swirling at a temperature of about 25 °C, and allow to stand for 10 min. Add 5 ml of L (+)-tartaric acid solution while swirling and after 1 min add 2 ml of L (+)-ascorbic acid solution while shaking. Transfer a solution to a 100 ml plastics volumetric flask, dilute to the mark with water and allow to stand for 60 min.

Measure the absorbance of the solution in a 10 mm cell at a wavelength of 650 nm using water as reference.

3.2.3 Plotting the calibration graph

Transfer 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml aliquot portions of diluted silicon(IV) oxide solution (0 mg to 0,4 mg as silicon(IV) oxide) into six 100 ml plastic beakers and, to each, add 10 ml of blank solution (B1) or (B'1) (see Annex A of ISO 21079-1:2008). Treat these solutions and measure the absorbance as described in 3.2.2, and plot the absorbances against the amounts of silicon(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

3.2.4 Calculation

Calculate the mass fraction of silicon(IV) oxide, $w(\text{SiO}_2)$, expressed as a percentage, using Equation (1) with the absorbances obtained in 3.2.2 and the calibration graph.

$$w(\text{SiO}_2) = \frac{(m_1 - m_2) + (m_s - m_b) \times \frac{500}{10}}{m} \times 100 \quad (1)$$

where

m_1 is the mass from 9.2.2.3 or 9.2.3.3 of ISO 21079-1:2008, in grams;