



FINAL DRAFT International Standard

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Phase quantitative analysis of residual quartz in silica bricks — X-ray diffraction method

*Analyse quantitative de la phase de quartz résiduel dans les
briques de silice — Méthode par diffraction des rayons X*

ISO/TC 33

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 33, *Refractories*.

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.

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Phase quantitative analysis of residual quartz in silica bricks — X-ray diffraction method

1 Scope

This document specifies a method for the quantitative analysis of residual quartz (i.e. alpha-quartz) in silica bricks within the mass fraction range of 0,3 % to 5,0 %, by X-ray diffraction (XRD) using a Bragg-Brentano diffractometer. This document includes details of sample preparation and of preliminary establishment of a working curve using external standards.

This document does not address the safety issues associated with its use. The ground silica brick powders and reference materials may cause damage to lungs through prolonged or repeat inhalation during tests. It is responsibility of the users of this standard to establish appropriate safety and health practices.

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 836, *Terminology for refractories*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

ISO 23071:2021, *Refractory products — Determination of reduced species in carbon containing refractories by XRD*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 836 and the following apply.

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/>

3.1 sample

representative collection of items that can be obtained by sampling according to a sampling plan agreed upon by the interested parties

Note 1 to entry: An example sampling plan is given in ISO 5022.

3.2 silica brick

refractory brick containing SiO₂ greater than or equal to 93 % by mass.

3.3 item

refractory *silica brick* (3.2).

3.4

test portion

amount of powder, taken from a crushed and ground *item* (3.3).

3.5

test piece

piece of material for the test, prepared by compaction into an adequate holder from *test portion* (3.4).

3.6

diluent

silica brick without alpha-quartz

EXAMPLE Used silica bricks in the working area served for more than 10 years, containing tridymite and a small amount of glass phase, and alpha-quartz is not detected

4 Principle

The X-ray diffraction peak intensity of a phase to be measured in multi-phase materials is proportional to the phase content. Silica bricks are mainly composed of phases of tridymite, cristobalite and alpha -quartz. The quantitative analysis of alpha-quartz phase in silica bricks is conducted by the external standard method.

5 Apparatus

5.1 X-ray diffractometer, in accordance with in ISO 23071:2021, Clauses 4 and 5.

5.2 Grinder and suitable grinding media, which shall not contaminate the test portion, e.g. tungsten carbide or zirconia, grinding the item(s) into powder with particle size no more than 0,030 mm.

5.3 Analytical balance, with a sensitivity of 0,1 mg.

5.4 Drying oven, capable of being controlled at $(110 \pm 5) ^\circ\text{C}$, or other device which has an equivalent heating effect.

5.5 Cavity holder, powder holder and punch.

6 Alpha-quartz reference materials

The alpha-quartz content of the reference materials, e.g. NIST 1878b, shall not be less than 95 %; other phases shall not be observed through X-ray diffraction or polarizing microscopy. Alternatively, a series of reference materials qualified may be used for establishment of a working curve, e.g. GSB S2021004.

7 Sampling

A sample shall consist of at least four items, obtained by sampling in accordance with ISO 5022, or a sampling plan agreed upon by the interested parties.

8 Procedure

8.1 Number of test pieces

A test portion shall be prepared for each item. Five test pieces shall be prepared for each test portion.

If the test portion is insufficient for preparing five test pieces, reuse the measured materials after re-homogenising and re-shaping for subsequent measurements.