
**Refractory products — Determination
of reduced species in carbon
containing refractories by XRD**

*Produits réfractaires - Détermination par DRX des espèces réduites
dans les réfractaires contenant du carbone*



This document is a preview generated by EKO



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

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
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Apparatus	2
5 Instrument performance	2
6 Sampling	2
7 Procedure	3
7.1 Sample preparation	3
7.2 Measuring parameters	3
7.3 Qualitative analysis	3
7.4 Quantitative analysis	4
7.4.1 General	4
7.4.2 Quantitative phase determination using Rietveld Refinement	4
7.5 Rationalisation of results	5
7.5.1 Calculation refinement for α -Si ₃ N ₄ , β -Si ₃ N ₄ , Si ₂ ON ₂ , BN and AlN	5
8 Limitations of method	6
9 Test report	6
Bibliography	7

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 of 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 www.iso.org/iso/foreword.html.

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.

Refractory products — Determination of reduced species in carbon containing refractories by XRD

1 Scope

This document describes methods for the determination of mineralogical phases often present as additives or reaction products in carbon containing or graphitic refractory products by X-ray Diffraction (XRD) using a Bragg-Brentano diffractometer. It includes details of sample preparation and general principles for qualitative and quantitative analysis of mineralogical phase composition. Quantitative determination of α - Si_3N_4 , β - Si_3N_4 , AlN, aluminium metal, Al_4C_3 , silicon metal, boron carbide and BN are described. The problems encountered with some determinations are highlighted.

Additional reduced species present in some refractories could include $\text{Al}_2\text{O}_3\cdot\text{AlN}$ solid solutions (so called Alons), $\text{Si}_3\text{N}_4\cdot\text{SiO}_2$ solid solutions and $\text{Si}_3\text{N}_4\cdot\text{Al}_2\text{O}_3$ solid solutions (Sialons). The presence of some of these solid solution components will cause problems with both identification and quantification as they are not well-defined structures.

NOTE For rationalisation of nitrogen containing phases, the total nitrogen content, analysed in accordance with EN 12698-1 is used.

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 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

ISO 10081-1, *Classification of dense shaped refractory products — Part 1: Alumina-silica*

ISO 10081-2, *Classification of dense shaped refractory products — Part 2: Basic products containing less than 7 % residual carbon*

ISO 10081-3, *Classification of dense shaped refractory products — Part 3: Basic products containing from 7 % to 50 % residual carbon*

ISO 10081-4, *Classification of dense shaped refractory products — Part 4: Special products*

EN 13925-1, *Non-destructive testing. X-ray diffraction from polycrystalline and amorphous materials. General principles*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 836, ISO 10081-1, ISO 10081-2, ISO 10081-3, ISO 10081-4, EN 13925-1 and the following apply.