

ISO 21068-2:2008-08 (E)

Chemical analysis of silicon-carbide-containing raw materials and refractory products - Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon

| Contents | | Page |
|--------------------|---|-------------|
| Foreword | | v |
| Introduction | | vi |
| 1 | Scope | 1 |
| 2 | Normative references | 1 |
| 3 | Terms and definitions | 1 |
| 4 | Determination of change in mass | 2 |
| 4.1 | General | 2 |
| 4.2 | Determination of the loss on drying at 250 °C (LOD250) gravimetric method | 2 |
| 4.3 | Determination of the loss on calcination in argon (LOI _{Ar}) | 3 |
| 4.4 | Determination of the change in mass by heat pretreatment in air | 5 |
| 4.5 | Determination of the change in mass at 750 °C (LOI ₇₅₀) | 6 |
| 4.6 | Determination of loss on ignition at 850 °C (LOI ₈₅₀) | 7 |
| 4.7 | Determination of loss on ignition at 1 050 °C (LOI _{1 050}) | 7 |
| 5 | Determination of the total carbon content | 8 |
| 5.1 | General | 8 |
| 5.2 | Combustion techniques | 9 |
| 5.3 | Detection techniques | 12 |
| 5.4 | Detection methods | 17 |
| 6 | Determination of free carbon content | 24 |
| 6.1 | General | 24 |
| 6.2 | Sample decomposition by combustion | 24 |
| 6.3 | Detection techniques | 24 |
| 6.4 | Direct detection methods | 24 |
| 6.5 | Indirect detection methods | 29 |
| 6.6 | Direct determination of free carbon by wet oxidation | 33 |
| 7 | Determination of silicon carbide content, SiC | 33 |
| 7.1 | General | 33 |
| 7.2 | Determination of silicon carbide, SiC, by indirect quantitative method | 34 |
| 7.3 | Determination of silicon carbide, SiC, by direct quantitative method | 34 |
| 7.4 | Determination of silicon carbide SiC by ignition method at 750 °C | 35 |
| 7.5 | Determination of silicon carbide, SiC, by chemical method | 36 |
| 8 | Determination of total silicon content via silica | 38 |
| 8.1 | Principle | 38 |
| 8.2 | Reagents | 38 |
| 8.3 | Apparatus | 38 |
| 8.4 | Procedure | 39 |
| 8.5 | Blank test | 39 |
| 8.6 | Calculation | 40 |
| 9 | Determination of free silicon content | 40 |

| | | |
|---|--|----|
| 9.1 | Principle | 40 |
| 9.2 | Pretreatment with hydrochloric acid | 40 |
| 9.3 | Silicon determination by hydrogen evolution | 40 |
| 9.4 | Silicon determination by silver displacement | 43 |
| 10 | Determination of silica content, SiO ₂ | 45 |
| 10.1 | General | 45 |
| 10.2 | Determination of free and/or combined silica content, SiO ₂ | 45 |
| 10.3 | Determination of free silica, SiO ₂ | 46 |
| 10.4 | Determination of surface silicon dioxide, SiO ₂ | 46 |
| 11 | Expression of results | 46 |
| 12 | Test report | 46 |
| Annex A (informative) Examples of CRMs for calibration of carbon analyser | | 47 |
| Bibliography | | 48 |