

DIN EN ISO 21068-2:2024-11 (E)

Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon - Part 2: Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica (ISO 21068-2:2024)

Contents		Page
European foreword		4
Foreword		5
Introduction		6
1	Scope	7
2	Normative references	7
3	Terms and definitions	7
4	Determination of volatile components by gravimetric methods	7
4.1	General	7
4.2	Determination of the loss on drying at 250 °C (w_{LOD250})	8
4.2.1	Principle	8
4.2.2	Apparatus	8
4.2.3	Procedure	8
4.2.4	Calculation	8
4.3	Determination of the loss on ignition in argon (w_{LOIAr})	8
4.3.1	Principle	8
4.3.2	Apparatus	9
4.3.3	Test assembly	9
4.3.4	Reagents	9
4.3.5	Procedure	10
4.3.6	Calculation	10
5	Determination of the total carbon content	11
5.1	Scope	11
5.2	Combustion techniques	11
5.2.1	Combustion in a resistance furnace with lead borate or tin as decomposing agent	11
5.2.2	Combustion in an induction furnace (IF) with metallic powder as decomposing agent	12
5.3	Detection techniques	13
5.3.1	Coulometry	13
5.3.2	Detection of the released carbon dioxide, CO ₂ , by infrared absorption (IR)	13
5.3.3	Thermal conductivity (TC) method	14
5.4	Expression of results	14
6	Determination of free carbon content	14
6.1	General	14
6.1.1	Direct methods	14
6.1.2	Indirect methods	19
7	Determination of silicon carbide content	22
7.1	General	22
7.2	Determination of silicon carbide, SiC, by indirect method	22
7.2.1	Principle	22
7.2.2	Calculation	22
7.2.3	Precision	23
7.3	Determination of silicon carbide, SiC, by combustion methods	23
7.3.1	Procedure	23
7.3.2	Calculation	23

7.4	Determination of silicon carbide, SiC, by combustion at 750 °C.....	23
7.4.1	Principle.....	23
7.4.2	Residue production.....	23
7.4.3	Determination of the total carbon content of the residue.....	24
7.4.4	Calculation.....	24
8	Determination of total silicon content.....	25
9	Determination of free silicon content.....	25
9.1	Principle.....	25
9.2	Pretreatment with hydrochloric acid.....	25
9.3	Determination of free silicon by hydrogen evolution.....	25
9.3.1	Reagents.....	25
9.3.2	Apparatus.....	26
9.3.3	Mass of test portion.....	26
9.3.4	Procedure.....	27
9.3.5	Blank test.....	28
9.3.6	Calculation.....	28
10	Determination of free silica content.....	29
10.1	Principle.....	29
10.2	Reagents.....	29
10.3	Apparatus.....	29
10.4	Sample preparation.....	30
10.5	Procedure.....	30
10.5.1	Determination.....	31
10.5.2	Calculation and expression of SiO ₂ content.....	31
11	Determination of surface silica content.....	31
12	Expression of results.....	32
13	Test report.....	32
Annex A (informative) Precision data.....		33
Annex B (informative) Examples of certified reference materials for calibration of carbon analyser.....		39
Annex C (normative) Correction of the free carbon content in case of oxidation of SiC.....		40
Bibliography.....		41