

Abrasive grains and crude — Chemical analysis of silicon carbide

ICS 25.100.70

Confirmed
July 2008

National foreword

This British Standard reproduces verbatim ISO 9286 : 1997 and implements it as the UK national standard.

The UK participation in its preparation was entrusted to Technical Committee MTE/13, Grinding wheels, abrasive tools, paper and cloths and powders, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled 'International Standards Correspondence Index', or using the 'Find' facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii and iii, a blank page, pages 1 to 22, an inside back cover and a back cover.

Amendments issued since publication

Amd. No.	Date	Text affected

This British Standard, having been prepared under the direction of the Engineering Sector Board, was published under the authority of the Standards Board and comes into effect on 15 June 1997

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ISBN 0 580 27568 X

INTERNATIONAL STANDARD

**ISO
9286**

First edition
1997-01-15

Abrasive grains and crude — Chemical analysis of silicon carbide

*Abrasifs en grains ou en roche — Analyse chimique du carbure
de silicium*



Reference number
ISO 9286:1997(E)

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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.

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.

International Standard ISO 9286 was prepared by Technical Committee ISO/TC 29, *Small tools*, Subcommittee SC 5, *Grinding wheels and abrasives*.

Annexes A and B of this International Standard are for information only.

Abrasive grains and crude — Chemical analysis of silicon carbide

1 Scope

This International Standard covers the chemical analysis of silicon carbide based abrasive grains and crudes. It is applicable for the determination of the surface impurities of abrasives grains and determining the SiC content of crushed crude when the silicon carbide content is greater than 95 % (m/m).

When the grain size of the silicon carbide is greater than or equal to 15 μm , the determination of the loss on acid treatment is carried out according to the method given in 3.5 and the residual SiC content (SiC_R) according to the method given in 3.7.

When the grain size of the silicon carbide is smaller than 15 μm , the determination of the SiC content shall be carried out by the methods described in 4.3 and 4.4.2.1 or 4.4.2.3 and 4.5 because the methods given in 4.2 and 3.7 inevitably give false results due to oxidation.

When the surface carbon content [$w(\text{C}_{\text{surf}})$] is greater than 2 % (m/m), a coulometric determination according to the method described in 3.4.2.3 will give correct results. The gravimetric method described in 3.4.2.1 is only applicable to surface carbon contents which are greater than 2 % (m/m), if ignition is continued to a constant weight or a weight increase. The coulometric method described in 3.4.2.2 cannot be applied in cases where $w(\text{C}_{\text{surf}})$ is more than 2 %.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9138:1993, *Abrasive grains — Sampling and splitting*.

3 Analysis of surface impurities

This clause applies to the determination of the surface impurities of abrasive grains in their original particle size state.

3.1 Sampling

The sample shall be taken from the batch of SiC grains to be analysed by according to the method described in ISO 9138.

3.2 Preparation of sample

The surface chemical analysis shall be carried out on unprocessed abrasive grains. The sample will only be dried at $(110 \pm 5)^\circ\text{C}$ until constant weight is obtained.

3.3 Determination of surface silicon (Si_{surf})

3.3.1 Principle

Volumetric method based on liberation of hydrogen resulting from the attack on silicon by a boiling sodium hydroxide solution.

3.3.2 Reagents

3.3.2.1 Sodium hydroxide solution, approximately 25 % (m/m).

3.3.2.2 Distilled or totally deionized water, to be used as a sealing liquid, acidified by several drops of sulphuric acid and slightly coloured with methyl orange.

3.3.3 Apparatus

Standard laboratory equipment and

3.3.3.1 Apparatus for silicon determination, as shown in figure 1, consisting of a hot plate (1) with a sand bath, a 100 ml wide-necked conical flask (2), a ball condenser having a total length of 40 cm approximately (3), an angled capillary tube (4), a gas burette (5) with an additional reservoir volume of 180 ml to 200 ml (6), a rubber tube connected to a levelling bottle (7).

Place the 100 ml conical flask in an upright position on the electrically heated sand bath. Connect it to the ball condenser by means of the rubber stopper which ensures gas tightness. Place the thermometer (3.3.3.4) in the cooling water using a T-piece. Connect the ball condenser to the upper end of the gas burette by means of a tube passing through the rubber bung. Connect the capillary tube from the lower end of the gas burette to the levelling bottle which contains a sealing liquid.

3.3.3.2 Barometer.

3.3.3.3 Thermometer, for measuring ambient temperature.

3.3.3.4 Immersion thermometer, for checking the temperature of the cooling liquid.

3.3.3.5 Thermostat.

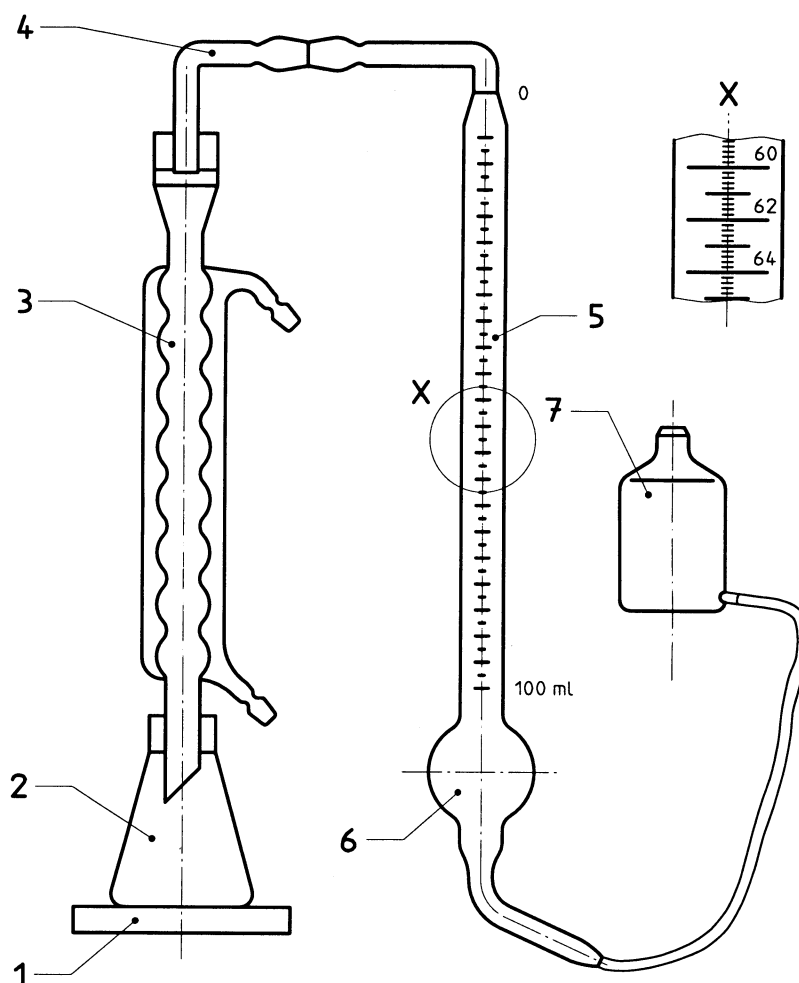
3.3.4 Procedure

From the sample prepared according to 3.2, take a test portion (m) of 5 g weighed to within $\pm 0,001$ g and place in the conical flask. Reduce the test portion (m) to less than 5 g if the volume of hydrogen produced exceeds the capacity of the burette due to the high content of surface silicon.

Prior to setting the starting level in the gas burette, allow the cooling water to circulate in the ball condenser for at least 10 min until the temperature is constant to within $\pm 1^\circ\text{C}$.

At the time of recording the final level in the gas burette, the temperature of the cooling water shall be identical to that at the start within $\pm 1^\circ\text{C}$.

If tap water does not provide the consistent temperature required to within $\pm 1^\circ\text{C}$, a thermostat shall be inserted into the cooling water circuit.



Key

- | | | | |
|---|-----------------------|---|----------------------|
| 1 | Hot plate | 5 | Gas burette |
| 2 | Conical flask | 6 | Additional reservoir |
| 3 | Ball condenser | 7 | Levelling bottle |
| 4 | Angled capillary tube | | |

Figure 1 — Apparatus for determining silicon

Record the ambient temperature in the immediate vicinity of the gas burette to an accuracy of 0,1 °C. If this temperature is not constant the gas burette shall be fitted with a thermostat control.

Add 40 ml of the sodium hydroxide solution, at ambient temperature, to the test portion to be analysed in the conical flask. Connect the flask immediately to the ball condenser, then rapidly adjust the sealing liquid to the zero point of the gas burette using the levelling bottle. Without changing the position of the levelling bottle connect the capillary tube to the condenser and record the initial level of the sealing liquid in the burette to an accuracy of 0,1 ml, after having accurately adjusted the level in the burette and in the levelling bottle. Heat the contents of the conical flask and boil for 90 min. During the heating and boiling, protect the gas burette against thermal radiation. When the boiling period is over, remove the sand bath and the hot plate.

Cool the conical flask still connected to the condenser in a container filled with cold water. Replace the water as often as necessary in order to bring the conical flask and its contents down to the ambient temperature recorded at the beginning of the analysis.

Check that the temperature is identical in both the conical flask and the condenser then adjust the levels in the levelling bottle and the burette and record the liquid level in the burette. Next, record the ambient temperature and the barometric pressure. The difference in the ambient temperature between the beginning and the end of the analysis must not exceed ± 3 °C.