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ISO 9286:1997

AS/NZS 4616

Australian/New Zealand Standard™

**Abrasive grains and crudes—
Chemical analysis of silicon carbide**



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AS/NZS 4616:1999

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Industrial Diamond Association of Australia
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Australian/New Zealand Standard™

Abrasive grains and crudes— Chemical analysis of silicon carbide

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PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee ME/33, Abrasives to supersede, in part, AS 1487—1975, *Abrasive grain size*.

This Standard is identical with and has been reproduced from ISO 9286:1997, *Abrasive grains and crude—Chemical analysis of silicon carbide*.

The objective of this Standard is to provide a method for the determination of surface impurities of abrasive grains and the silicon carbide content of crushed crude when the silicon carbide content is greater than 95% by weight.

This Standard is one of a series of adopted ISO Standards applicable to bonded and coated abrasive products.

As this Standard is reproduced from an International Standard, the following applies:

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<i>Reference to International Standard or other Publication</i>		<i>Australian/New Zealand Standard</i>	
ISO		AS/NZS	
9138	Abrasive grains—Sampling and splitting	4520	Abrasive grains—Sampling and splitting

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AUSTRALIAN/NEW ZEALAND STANDARD

Abrasive grains and crudes—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.

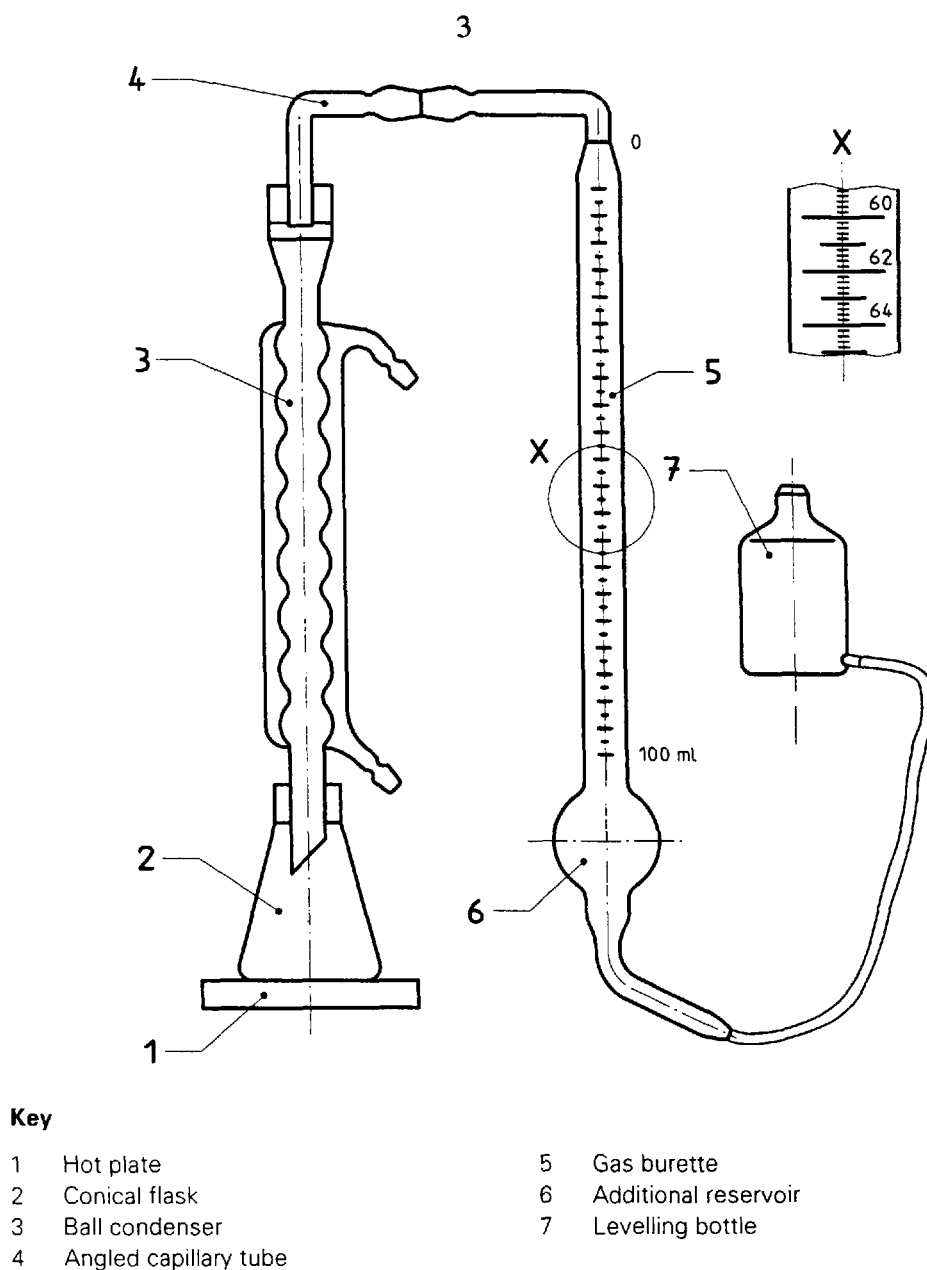


Figure 1 — Apparatus for determining silicon

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.

3.3.5 Expression of results

The content of surface silicon expressed as a percentage by mass is calculated using the following equation. The result will be rounded to two decimal places.

$$w(\text{Si}_{\text{surf}}) = \frac{0,000\,627 \times V \times f}{m} \times 100$$

where

- V is the volume of hydrogen collected in the gas burette, in millilitres;
- m is the weight of test portion according to 3.3.4, in grams;
- 0,000 627 is the conversion factor of the volume of hydrogen gas, in millilitres, to the weight of silicon, in grams;
- f is the correction factor for reducing the hydrogen gas volume to normal conditions of temperature and pressure: 0 °C and 1 013 hPa.

f can be obtained by reference to correction tables applicable to gases, taking into account the indicated temperature and the steam pressure above the sealing liquid.

3.4 Determination of surface carbon (C_{surf})

3.4.1 Principle

Gravimetric or coulometric determination of carbon dioxide obtained by the heating of the surface carbon in a stream of oxygen inside a combustion furnace.

3.4.2 Test methods

3.4.2.1 Gravimetric method

3.4.2.1.1 Apparatus

3.4.2.1.1.1 Combustion apparatus, as shown in figure 2.

3.4.2.1.2 Procedure

Prior to starting the measurements, purge the combustion train using a stream of oxygen for 10 min to 15 min. Take from the sample prepared according to 3.2, a test specimen (m_0) of 2 g weighed to within $\pm 0,000\,1$ g and place it in a previously calcined and weighed combustion boat. Weigh the absorption tube and insert it in the combustion train. Place the combustion boat containing the sample into the hot zone of the tube furnace at (900 to 915) °C. Pass a stream of oxygen for 30 min at a flow rate of 100 ml/min through the apparatus then remove the absorption tube and weigh it. The increase in mass corresponds to the weight of carbon dioxide (m_2). Finally determine the weight of the residue in the combustion boat (m_1) within $\pm 0,000\,1$ g.

3.4.2.1.3 Expression of results

The amount of surface carbon expressed as a percentage by mass, is calculated using the equation

$$w(C_{\text{surf}}) = \frac{(0,272\,9 \times m_2) - (0,375\,4 \times m_3)}{m_0} \times 100$$

where

- m_0 is the mass of the test portion prior to combustion (equal to the mass of sample weighed on 3.4.2.1.2), in grams;
- m_1 is the mass of the sample after combustion, in grams;