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**9286**

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

**iTeh STANDARD PREVIEW**

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

[ISO 9286:1997](https://standards.iteh.ai/catalog/standards/sist/6838f33e-aa08-4696-999b-032aa7dde9bd/iso-9286-1997)

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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.

**iTeh STANDARD PREVIEW**

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

[ISO 9286:1997](#)

<https://standards.it/en/catalog/standards/sist/683873e-ae08-4686-999b-032aa7dde9bd/iso-9286-1997>

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

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# 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<sub>R</sub>) 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 [ $w(C_{\text{surf}})$ ] 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  $w(C_{\text{surf}})$  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 ( $\text{Si}_{\text{surf}}$ )

### 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.**

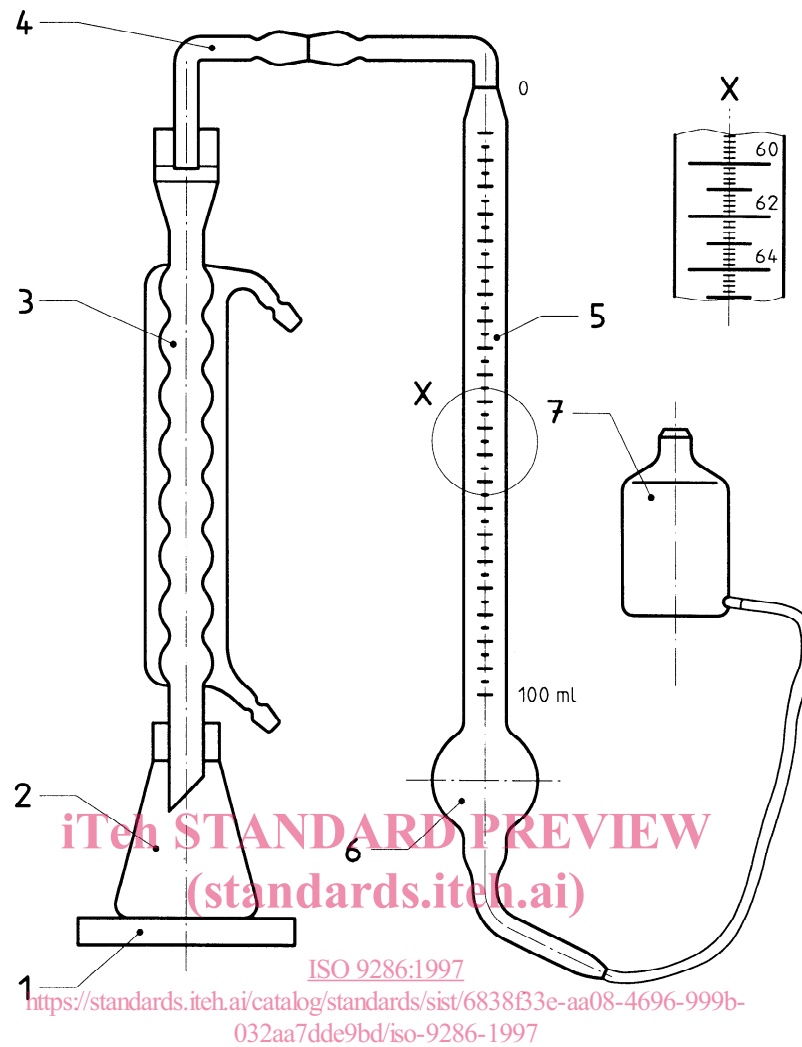
### 3.3.4 Procedure

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  $\pm 3$  °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 ( $C_{\text{surf}}$ )

### 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

ISO 9286:1997

3.4.2.1 Gravimetric method <https://standards.iteh.ai/catalog/standards/sist/6838f33e-aa08-4696-999b-032aa7dde9bd/iso-9286-1997>

#### 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;



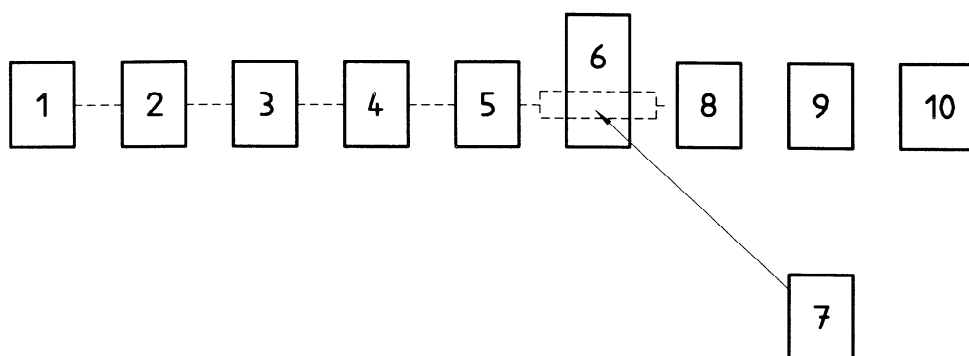
$m_2$  is the mass of carbon dioxide, in grams;

$$m_3 = m_1 - m_0 + 0,272\ 9\ m_2$$

$$0,272\ 9 = \frac{M_r(\text{C})}{M_r(\text{CO}_2)}$$

$$0,375\ 4 = \frac{M_r(\text{C})}{M_r(\text{O}_2)}$$

The method of determination and calculation compensates for possible oxidation of the silicon carbide.



#### Key

- 1 Oxygen source
- 2 Flow regulating valve
- 3 Flow meter
- 4 Column for the absorption of CO<sub>2</sub>
- 5 Drying column containing anhydrous Mg(ClO<sub>4</sub>)<sub>2</sub>
- 6 Tube furnace
- 7 Combustion tube (outlet lined with ferruginous glass wool)
- 8 Column containing 1 mm sized granules of zinc
- 9 Gas washing bottle containing H<sub>2</sub>SO<sub>4</sub> (density of 1,84 g/ml)
- 10 Absorption tube containing CO<sub>2</sub>-absorbent lined with glass wool at both ends and with a layer approximately 10 mm to 15 mm thick of Mg(ClO<sub>4</sub>)<sub>2</sub> covering the CO<sub>2</sub> absorbent

**Figure 2 — Combustion apparatus for the determination of carbon (resistance furnace)**

### 3.4.2.2 General coulometric method

#### 3.4.2.2.1 Reagents

##### 3.4.2.2.1.1 Barium perchlorate solution.

Dissolve approximately 200 g of barium perchlorate [Ba(ClO<sub>4</sub>)<sub>2</sub>] in distilled or deionized water and make up to 1 l.

##### 3.4.2.2.1.2 Barium carbonate (BaCO<sub>3</sub>).

##### 3.4.2.2.1.3 Hydrogen peroxide absorbed on urea [H<sub>2</sub>O<sub>2</sub>·CO(NH<sub>2</sub>)<sub>2</sub>], e.g. perhydrite tablets.

##### 3.4.2.2.1.4 2-propyl alcohol (isopropyl alcohol) [CH<sub>3</sub>CH(OH)CH<sub>3</sub>].

##### 3.4.2.2.1.5 Granulated soda lime.

##### 3.4.2.2.1.6 Buffer solutions for calibrating the pH meter, in accordance with the manufacturer's instructions.

##### 3.4.2.2.1.7 Oxygen, 99,99 % (V/V).

### 3.4.2.2.2 Apparatus

Standard laboratory equipment and

#### 3.4.2.2.2.1 Combustion unit, as shown in figure 3.

The combustion furnace shall be capable of operating at  $(1\ 100 \pm 30)$  °C.

#### 3.4.2.2.2.2 Thermocouple, with indicator to measure the furnace temperature.

#### 3.4.2.2.2.3 Flow meter.

For assembly and operation refer to the manufacturer's instructions. To commission a new apparatus or for occasional checking, carry out several determinations using a reference sample of known surface carbon content in the manner indicated in 3.4.2.2.3 before testing the required sample. The carbon content determined must correspond to admissible limits to the carbon content of the reference sample.

### 3.4.2.2.3 Procedure

From the sample prepared according to 3.2 take a 0,2 g test portion ( $m_0$ ) weighed to within  $\pm 0,000\ 1$  g and place in a combustion boat which has been previously calcined to remove all carbon. Heat the combustion furnace to  $(850 \pm 20)$  °C and insert the boat. Record the internal temperature and adjust the furnace temperature correspondingly. It is customary to use gas fractionating which means that only a fraction, (usually one tenth) of the gas to be analysed is used; except where the surface carbon content is very low, in which case gas fractionating is not used.

Adjust the stream of oxygen so as to prevent the surrounding air from being introduced. At the temperature given above, the determination of the surface carbon takes 10 min. Each series of analyses shall be preceded by a blank value determination using a pre-ignited boat (without gas fractionating). Calculate the mean value ( $l'$ ) from three separate determinations.

As a general rule, the blank test gives a carbon content which varies between 0,01 % ( $m/m$ ) and 0,02 % ( $m/m$ ).

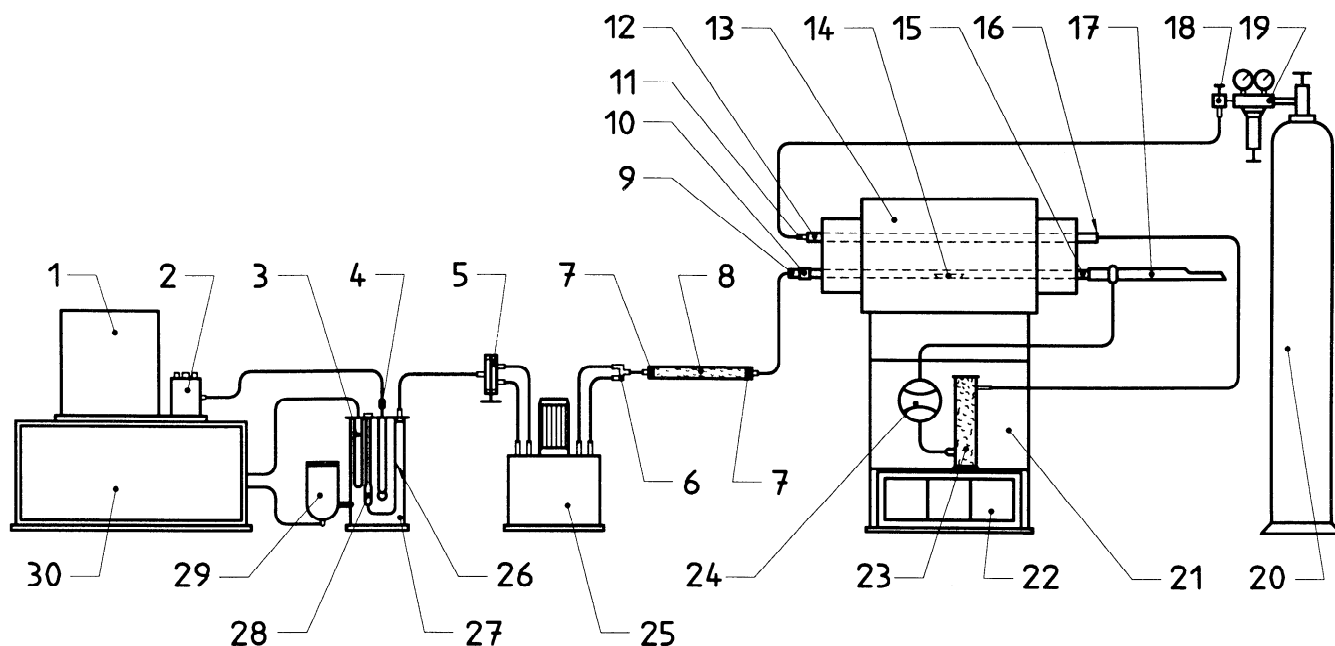
#### 3.4.2.2.4 Expression of results

The surface carbon content ( $C_{\text{surf}}$ ), expressed as a percentage by mass, is calculated according to the following equation, the result being rounded to the second decimal place.

$$w(C_{\text{surf}}) = \frac{(l \times f) - (l' \times f') \times x}{m_0 \times 1000} \times 100$$

where

- $l$  is the mean value of the number of pulses found in the sample;
- $f$  is the gas fractionation coefficient;
- $l'$  is the mean value of the number of pulses found in the blank determination according to 3.4.2.2.3;
- $f'$  is the gas fractionating coefficient used in the blank tests;
- $m_0$  is the weight of the test portion (see 3.4.2.2.3), in grams;
- $x$  is the proportionality coefficient, depending on the apparatus which gives the conversion of the number of pulses into milligrams of carbon;
- 1 000 is the conversion factor, grams to milligrams.



### Key

1	pH-meter	16	4 mm sealing piece
2	Titrating agent	17	Specimen entry point
3	Cathode	18	Precision regulating device
4	Detector	19	Pressure reducing valve
5	Piston valve	20	High pressure oxygen bottle
6	T-piece	21	Current transformer
7	Cotton wadding	22	Temperature controlling device
8	Tube containing perhydrate	23	Soda lime vessel
9	Glass connection with quartz wool	24	Flow meter
10	Coupling hose	25	Metering pump
11	8 mm adapter	26	Inlet tube
12	Ceramic tube	27	Absorption cell
13	Combustion furnace	28	Mixing chamber
14	Combustion boat	29	Anode
15	Combustion tube	30	Coulometer

**Figure 3 — Apparatus for determining carbon content using the coulometric method**

### 3.4.2.3 Particular coulometric method

Coulometric method taking account of SiC oxidation and/or which can be used when the surface carbon content is greater than 2 % (*m/m*).

#### 3.4.2.3.1 Reagents

Reagents indicated in 3.4.2.2.1 and

**3.4.2.3.1.1 Lead borate** ( $2\text{PbO}\cdot\text{B}_2\text{O}_3$ ), obtained by melting 45 g lead oxide (PbO) and 7 g of anhydrous boric oxide at 950 °C for 10 min.

Pour the molten mass on to a clean aluminium plate and allowed to cool. Pulverize the lead borate obtained to a powder.