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)

Contents

Page

1 Scope **1**
2 Normative reference **1**
3 Analysis of surface impurities **1**
  3.1 Sampling **1**
  3.2 Preparation of sample **2**
  3.3 Determination of surface silicon (Si_{surf}) **2**
  3.4 Determination of surface carbon (C_{surf}) **4**
  3.5 Determination of loss on acid treatment (LAT) **9**
  3.6 Determination of surface silicon dioxyde ($SiO_{2\,surf}$) **10**
  3.7 Calculation of the content of residual silicon carbide (SiC_R) . **11**
  3.8 Determination of surface iron (Fe_{surf}) **12**
  3.9 Determination of surface iron by atomic absorption spectrometry (AAS) **13**
  3.10 Determination of surface aluminium oxide ($Al_2O_{3\,surf}$) **15**
  3.11 Determination of surface aluminium oxide by atomic absorption spectrometry (AAS) **15**
  3.12 Determination of surface calcium oxide (CaO_{surf}) and of surface magnesium oxide (MgO_{surf}) **15**
  3.13 Determination of surface magnesium oxide (MgO_{surf}) and of surface calcium oxide (CaO_{surf}) by atomic absorption spectrometry (AAS) **17**
4 Determination of silicon carbide (SiC) in crushed silicon carbide .. **17**
  4.1 Principle **17**
  4.2 Preparation of sample **17**
  4.3 Determination of total carbon (C_{total}) **18**
  4.4 Determination of free carbon (C_{free}) **20**
  4.5 Calculation of the silicon carbide (SiC) content **20**
5 Test report **20**

Annexes

A Other methods of analysis **21**
B Permissible deviation as a result of variations in the measuring technique for the chemical analysis of silicon carbide **22**

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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 $[w(C_{surf})]$ 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 $w(C_{surf})$ 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 ± 5) °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.

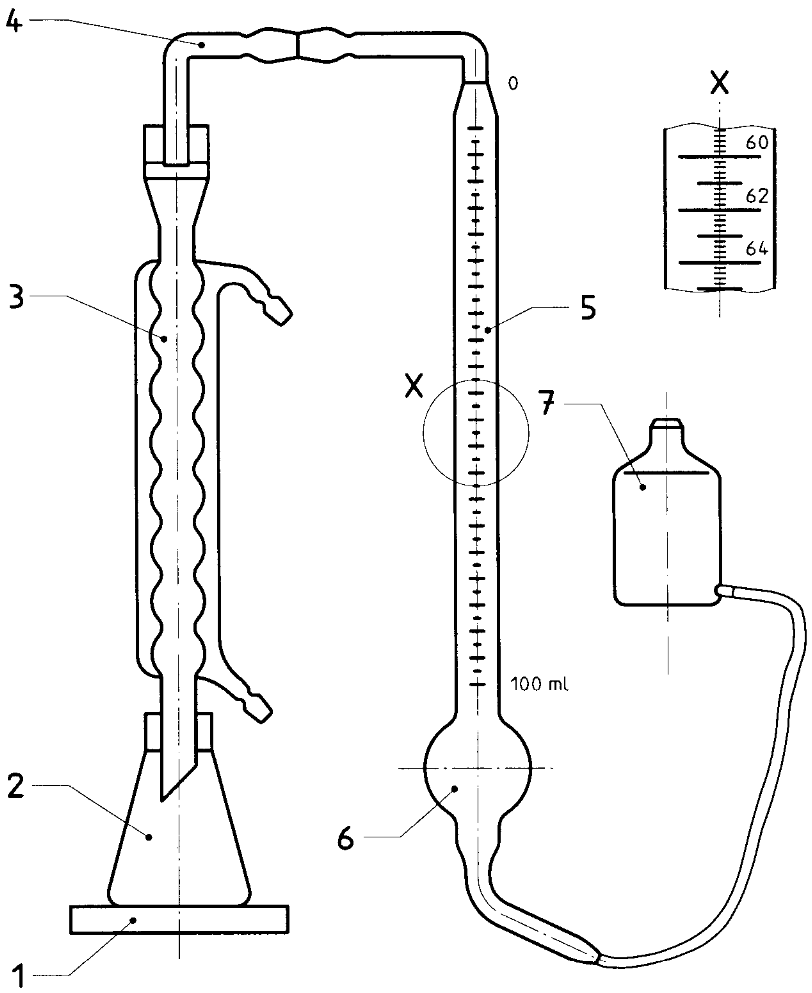
3.3.4 Procedure

From the sample prepared according to 3.2, take a test portion (*m*) of 5 g weighed to within ± 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 ± 1 °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 ± 1 °C.

If tap water does not provide the consistent temperature required to within ± 1 °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.

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.

$$w(\mathrm{Si_{surf}}) = \frac{0{,}000\,627 \times V \times f}{m} \times 100$$

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 ($\mathrm{C_{surf}}$)

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 ± 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 ± 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

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

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;

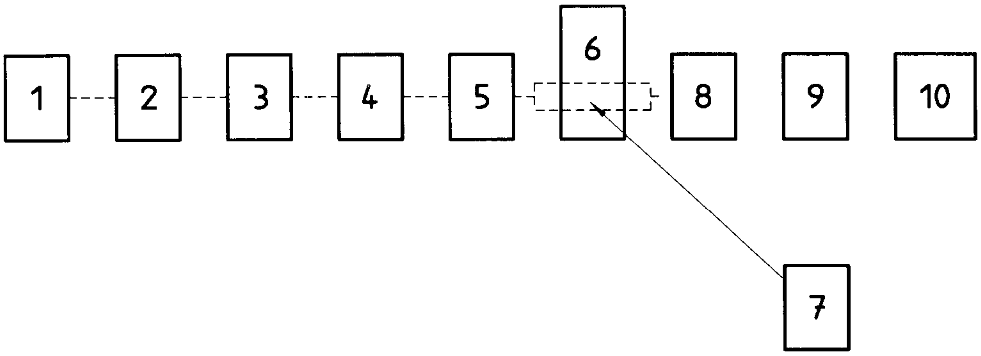
m_2 is the mass of carbon dioxide, in grams;

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

$$0{,}272\,9 = \frac{M_r(\mathrm{C})}{M_r(\mathrm{CO_2})}$$

$$0{,}375\,4 = \frac{M_r(\mathrm{C})}{M_r(\mathrm{O_2})}$$

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_2
5 Drying column containing anhydrous $Mg(ClO_4)_2$
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_2SO_4 (density of 1,84 g/ml)
10 Absorption tube containing CO_2-absorbent lined with glass wool at both ends and with a layer approximately 10 mm to 15 mm thick of $Mg(ClO_4)_2$ covering the CO_2 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 [$Ba(ClO_4)_2$] in distilled or deionized water and make up to 1 l.

3.4.2.2.1.2 Barium carbonate ($BaCO_3$).

3.4.2.2.1.3 Hydrogen peroxide absorbed on urea [$H_2O_2 \cdot CO(NH_2)_2$], e.g. perhydrite tablets.

3.4.2.2.1.4 2-propyl alcohol (isopropyl alcohol) [$CH_3CH(OH)CH_3$].

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 by capable of operating at (1 100 ± 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 ± 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 ± 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*).

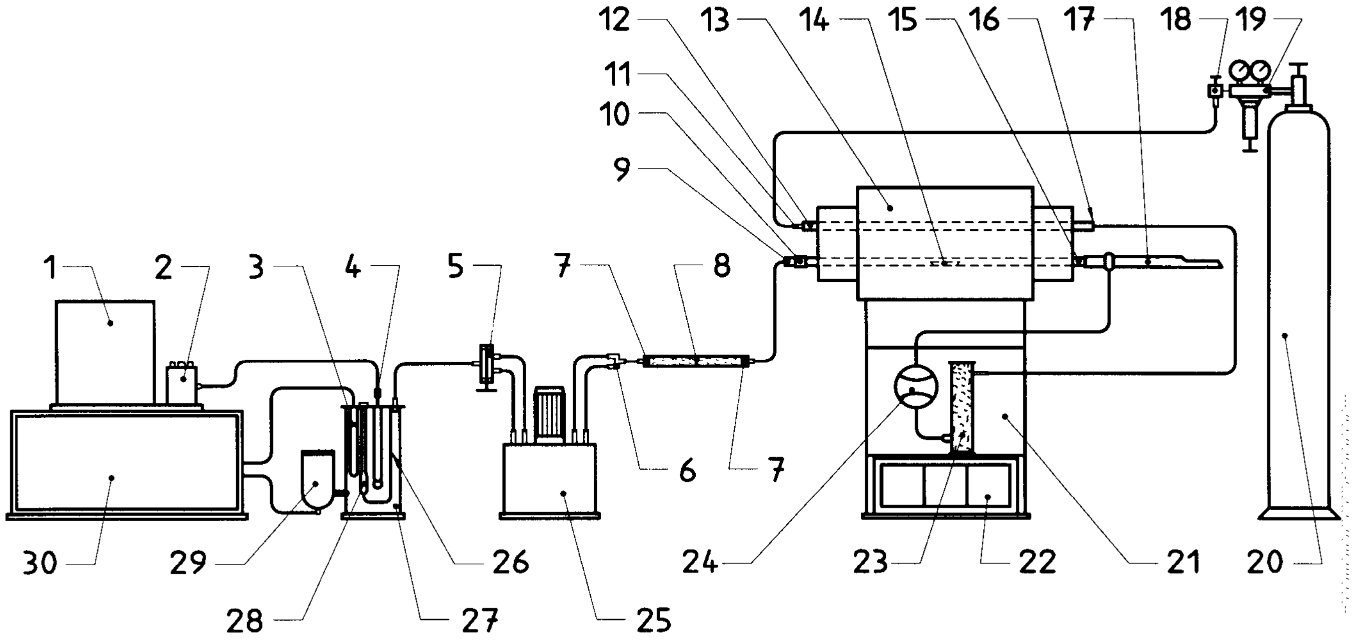
3.4.2.2.4 Expression of results

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

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

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 perhydrite	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 ($2PbO \cdot B_2O_3$), 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.

3.4.2.3.2 Apparatus

Standard laboratory equipment and

3.4.2.3.2.1 Electric furnace, capable operating at (750 ± 20) °C.

3.4.2.3.2.2 Agate mortar.

3.4.2.3.2.3 Other equipment, required for the determination of surface carbon indicated in 3.4.2.2.2.

3.4.2.3.3 Procedure

From the sample prepared according to 3.2, take a 1 g test portion (m_0) weighed to within ± 0,000 1 g and place in a previously calcined crucible. Heat in the furnace for 60 min at (750 ± 20) °C, then allow to cool in a desiccator and weigh to an accuracy of 0,1 mg. Reheat for at least 30 min and re-weigh when the specimen has cooled. If, after the second heating operation, there is a loss in mass, repeat the operation until constant or increasing mass is obtained. Refer, in this case, to the mass of the analytical product obtained after the last heating (m_1).

Disagglomerate the specimen in the agate mortar and homogenise without further size reduction. Then determine the carbon content [$w(\mathrm{C_R})$] of the calcined residue according to the directions given in 4.3.2.2.

Determine the total carbon content [$w(\mathrm{C_{total}})$] of the sample to be analysed which was prepared according to the directions given in 3.2, in the manner described in section 4.3.2.2.

3.4.2.3.4 Expression of results

The variation in mass during calcination at 750 °C (m_v), expressed as a percentage by mass, is given by the following formula and rounded to the nearest 0,1 % (m/m).

$$m_\mathrm{v} = \frac{m_1 - m_0}{m_0} \times 100$$

where

m_1 is the specimen weight after annealing at 750 °C according to the directions given in 3.4.3.3;

m_0 is the test portion weight (see 3.4.2.3.3), in grams.

In the following equation prefix the result with a negative sign if the mass has decreased and a positive sign if it has increased.

The mass of the calcined residue R, as a percentage by mass, rounded to the nearest 0,1 % (m/m) is

$$R = 100 + m_\mathrm{v}$$

The surface carbon content [$w(\mathrm{C_{surf}})$], as a percentage by mass, rounded to the nearest two decimal places, is calculated using the following equation:

$$w(\mathrm{C_{surf}}) = \frac{w(\mathrm{C_{total}}) - \dfrac{R - w(\mathrm{C_R})}{100}}{1{,}600\,9} - \frac{m_\mathrm{v}}{2{,}664\,1}$$

or

$$w(\mathrm{C_{surf}}) = \frac{w(\mathrm{C_{total}}) - w(\mathrm{C_R})}{1{,}600\,9} - \frac{m_\mathrm{v} \times w(\mathrm{C_R})}{160{,}09} - \frac{m_\mathrm{v}}{2{,}664\,1}$$

The factors in the equations are calculated as follows:

$$1{,}600\,9 = \frac{\dfrac{M_r(SiO_2) - M_r(SiC)}{M_r(C)} + 1}{\dfrac{M_r(SiO_2) - M_r(SiC)}{M_r(C)}}$$

$$2{,}664\,1 = \frac{M_r(SiO_2) - M_r(SiC)}{M_r(C)} + 1$$

3.5 Determination of loss on acid treatment (LAT)

3.5.1 Principle

The loss on acid treatment is understood to be the loss of substance after treatment of the sample to be analysed, with a mixture of sulphuric, hydrofluoric and nitric acids.

3.5.2 Reagents

3.5.2.1 Sulphuric acid solution, 96 % (*m*/*m*).

3.5.2.2 Hydrofluoric acid solution, 40 % (*m*/*m*).

3.5.2.3 Nitric acid solution, 65 % (*m*/*m*).

3.5.2.4 Hydrochloric acid solution, 37 % (*m*/*m*).

3.5.2.5 Hydrochloric acid solution, approximately 4 % (*m*/*m*).

3.5.3 Apparatus

3.5.3.1 PTFE (polytetrafluorethylene) or **platinum crucible.**

3.5.3.2 Sand bath.

3.5.3.3 Porcelain filter crucible, porosity 7 µm.

3.5.3.4 Volumetric flask, 250 ml.

3.5.4 Procedure

From the sample prepared according to 3.2, take a 5 g test portion (m_0) weighed to within ± 0,001 g and place it in the PTFE or platinum crucible. Add 20 drops of sulphuric acid [96 % (*m*/*m*)], 30 ml hydrofluoric acid [40 % (*m*/*m*)] and 10 ml nitric acid [65 % (*m*/*m*)] and leave to evaporate overnight. Mix the residue with 10 ml hydrochloric acid [37 % (*m*/*m*)] and heat at approximately 60 °C for 30 min. Filter using a weighed porcelain filter crucible. Wash the filtration residue three times with diluted hydrochloric acid [4 % (*m*/*m*)] and twice with hot distilled water [at approximately (90-100) °C] and then dry in an oven at 110 °C. After cooling, weigh the crucible and determine the mass of the evaporated residue (m_1). Transfer the filtrate to a 250 ml volumetric flask. Cool, fill to the volume mark with distilled water and mix thoroughly.

Put aside the (V_0) solution for determining Fe, Al_2O_3, Mg and CaO.

3.5.5 Expression of results

Loss on acid treatment (LAT), expressed as a percentage by mass, is given by the equation

$$\text{LAT} = \frac{m_0 - m_1}{m_0} \times 100$$

where

m_0 is the mass of the test portion according to 3.5.4, in grams;

m_1 is the mass of residue according to 3.5.4, in grams.

3.6 Determination of surface silicon dioxide ($SiO_{2\,surf}$)

3.6.1 Principle

This method is suitable for the determination of the surface SiO_2 content in SiC and is based on the reactions shown in 3.6.2.

3.6.2 Reactions

$$SiO_2 + 2\ KF + 4\ HF \rightarrow K_2SiF_6 + 2\ H_2O$$

The K_2SiF_6 is separated and titrated with a caustic soda (NaOH) solution.

$$K_2SiF_6 + 4\ NaOH \rightarrow 2\ KF + 4\ NaF + SiO_2 + 2\ H_2O$$

The method can be applied in the presence of elemental silicon because this does not react under the conditions of this procedure.

If silicates and/or silicides are present, there may be a reaction leading to higher $SiO_{2\,surf}$ results. Therefore, it must be checked as to whether this method is applicable in such cases.

3.6.3 Reagents

3.6.3.1 Potassium fluoride/hydrofluoric acid solution (KF/HF), 125 g KF is dissolved in 800 ml HF, 40 % (*m*/*m*).

3.6.3.2 Hydrochloric acid, 18 % (*m*/*m*) solution.

3.6.3.3 Wash solution, 100 g KCl is dissolved in 1 l of distilled water (use either a fresh solution or solution cooled down to approximately 10 °C).

3.6.3.4 Caustic soda (NaOH), standardized solution, 0,1 mol/l.

3.6.3.5 Phenolphthalein indicator, 1 % (*m*/*m*) alcohol solution.

3.6.3.6 Blue litmus paper.

3.6.3.7 Ice cubes.

3.6.4 Apparatus

3.6.4.1 PTFE or **platinum crucible.**

3.6.4.2 Sand bath, adjustable to ± 5 °C.

3.6.4.3 PE (polyethylene) Büchner funnel, (45 mm, consisting of two parts).

3.6.4.4 PE suction bottle.

3.6.4.5 Ashless type filter paper, (45 mm diameter, dense against $BaSO_4$).

3.6.4.6 Ashless filter paper flake.

3.6.4.7 Rubber spatula.

3.6.4.8 Conical flask, 300 ml.

3.6.4.9 pH-meter, with temperature compensation.

3.6.5 Procedure

Weigh, to within ± 0,001 g, 1 g (m_0) of the sample prepared according to 3.1 into a platinum or PTFE crucible. Add 15 ml KF/HF solution, then 5 ml HCl [18 % (m/m)] and heat on the sand bath for 2,5 h.

During the operation, the volume of liquid must not be significantly reduced. This is achieved by keeping the temperature of the sand bath at (50 ± 5) °C.

Next, place the crucible in a basin of cold water containing ice cubes to speed up cooling. Put a paper filter upon the bottom of the Büchner funnel, and then fill half of the funnel with filter flakes. These are first saturated with wash solution and carefully compacted by pressing slightly. Then filter the cold crucible contents, cleaning the crucible carefully using a rubber spatula and rinsing with the cold wash solution. Wash the residue on the filter with the cold wash solution (approximately 10 °C) and continue to wash until blue litmus no longer shows red coloration. Transfer the residue, together with the filter flakes to a 300 ml conical flask, dilute with hot distilled water, add 10 drops of phenolphthalein solution and titrate to the first persistent red coloration with NaOH (0,1 mol/l).

In the case of coloured solutions a pH-meter with temperature compensation can be used. The end point is at pH 8,2.

A blank determination is to be made and taken into account when evaluating.

3.6.6 Expression of results

$SiO_{2\,surf}$ content, expressed as a percentage by mass, is given by the equation

$$w\left(\mathrm{SiO}_{2\,\mathrm{surf}}\right) = \frac{\left(V - V_0\right) \times 1{,}502}{m_0 \times 1000} \times 100$$

where

m_0 is the mass of the test portion, according to 3.6.5, in grams;

V is the volume NaOH (0,1 mol/l) consumption in millilitres (sample);

V_0 is the volume NaOH (0,1 mol/l) consumption in millilitres (blank test);

1,502 is the conversion factor of millilitres of NaOH (0,1 mol/l) into milligrams of SiO_2;

1 000 is the conversion factor, grams to milligrams.

3.7 Calculation of the content of residual silicon carbide (SiC_R)

The calculation of the content of residual SiC is based on the results of the determination of surface carbon (C_{surf}), in 3.4 and of the determination of the loss on acid treatment (LAT) in 3.5, namely:

$$w(\mathrm{SiC_R}) = 100 - \left[w(\mathrm{C_{surf}}) + \mathrm{LAT}\right]$$

where

$w(\mathrm{C_{surf}})$ is the surface carbon content, expressed as a percentage by mass, according to 3.4;

LAT is the loss on acid treatment, expressed as a percentage by mass, according to 3.5.

3.8 Determination of surface iron ($\mathrm{Fe_{surf}}$)

3.8.1 Principle

Determination of surface iron by phenanthroline-1,10 spectrometry.

3.8.2 Reagents

3.8.2.1 Standard iron solution ($1\,\mathrm{ml} \mathrel{\hat=} 1\,\mathrm{mg}\ \mathrm{Fe_2O_3}$).

Prepare 1 l of solution by dissolving 4,91 g ferrous(II) ammonium sulphate, $\mathrm{Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O}$ in $\mathrm{H_2SO_4}$ (0,05 mol/l) and make up with the acid to 1 l. Adjust the strength of the solution using the titrated oxidizing solution.

3.8.2.2 Hydroxyl ammonium chloride solution, 10 % (*m*/*m*).

3.8.2.3 Ammonium acetate solution, 30 % (*m*/*m*).

3.8.2.4 Phenanthroline-1,10 solution, 0,1 % (*m*/*m*).

Dissolve 1 g phenanthroline monohydrate-(1,10) in 15 ml of ethanol 95 % (*V*/*V*) and make up to 1 l with water.

3.8.2.5 Sulphuric acid, 48 % (*m*/*m*).

3.8.2.6 Tartaric acid solution, 10 % (*m*/*m*).

3.8.2.7 Congo-red paper, according to 3.8.4.2.

3.8.3 Apparatus

3.8.3.1 Spectrophotometric absorptiometer.

3.8.3.2 Volumetric flask, 100 ml.

3.8.3.3 Pipette, 25 ml.

3.8.4 Procedure

3.8.4.1 Preparation of calibration curve

Prepare a series of standard solutions to cover the expected range of iron concentrations.

Determine their absorbance in the same manner as used for the test solution (see 3.8.4.2). Prepare a calibration curve showing the absorbance values in the terms milligrams of Fe per 250 ml of solution.

3.8.4.2 Determination

Sample a 25 ml aliquot (V_1) of the filtrate obtained during the loss on acid treatment determination (V_0, see 3.5.4) into a 100 ml volumetric flask (V_2).

Add 10 ml of hydroxyl ammonium chloride solution and 10 ml of tartaric acid solution [10 % (*m*/*m*)]. Add NH_4OH dropwise until the solution causes an alkaline reaction on the Congo-red paper. Add the H_2SO_4 [48 % (*m*/*m*)] dropwise until the solution begins to cause an acid reaction on the Congo-red paper. Add ammonium acetate solution until this solution causes an alkaline reaction, then add an additional 5 ml. Add 10 ml of the phenanthroline-1,10 solution. Bring up to the mark with water. Measure the absorbance of the solution at a wavelength around 500 nm. Use a blank test solution as a reference for measuring the absorbance.

3.8.5 Expression of results

Using the absorbance measured from the sample solution, interpolate the amount of surface iron in the sample solution from the calibration curve.

Iron content, expressed as a percentage by mass, is given by the equation

$$w\left(\mathrm{Fe_{surf}}\right) = \frac{m_1 \times V_2}{1\,000 \times m_0 \times V_1} \times 100$$

where

m_1	is the mass, in milligrams of iron in 250 ml, read from the calibration curve;
m_0	is the mass of the test portion according to 3.5.4, in grams;
V_1	is the volume sampled by pipette, according to 3.8.4.2, in millilitres;
V_2	is the volume of the test solution, according to 3.8.4.2, in millilitres;
1 000	is the conversion factor, grams to millilitres.

3.9 Determination of surface iron by atomic absorption spectrometry (AAS)

The surface iron content [$w(\mathrm{Fe_{surf}})$] can also be determined on the filtrate obtained from the loss on acid treatment (LAT) described in 3.5.4 using atomic absorption spectrometry (AAS).

3.10 Determination of surface aluminium oxide ($Al_2O_{3\,surf}$)

3.10.1 Principle

Determination of surface Al_2O_3 by using 8-hydroxyquinoline spectrometry.

3.10.2 Reagents

3.10.2.1 Tartaric acid solution, 2 % (*m*/*m*).

3.10.2.2 Ammonia solution, 25 % (*m*/*m*).

3.10.2.3 Ammonia solution, 6 % (*m*/*m*).

3.10.2.4 Ammonium sulphide solution, 5 % (*m*/*m*).

3.10.2.5 Potassium cyanide solution, 3 % (*m*/*m*).

3.10.2.6 8-hydroxyquinoline solution, 5 g/l.

Dissolve 0,5 g weighed to the nearest 0,001 g of 8-hydroxyquinoline in 15 ml of acetic acid and dilute to 100 ml with distilled water.

3.10.2.7 Aluminium standard solution.

Dilute one 0,1 g ampule of Al in 1 l of distilled water.

3.10.2.8 Methylene chloride

3.10.3 Apparatus

3.10.3.1 Spectrometric absorptiometer.

3.10.3.2 Volumetric flasks, of 100 ml and 1 l.

3.10.3.3 Conical flask, 100 ml.

3.10.3.4 Pipette, 50 ml.

3.10.3.5 Separating funnel, 100 ml.

3.10.3.6 Filter paper, medium and coarse grades, ashless.

3.10.4 Procedure

Using a pipette, transfer 50 ml (V_1) of the stock solution (V_0) obtained according to 3.5.4 to a 100 ml volumetric flask.

Add 5 ml of tartaric acid solution, shake and add as much ammonia solution [25 % (m/m)] as is necessary to render the solution alkaline (approximately 10 ml). Bring to the boil and mix with 5 ml ammonium sulphide solution and boil again for a short time. (Proceed with caution, as the solution tends to overflow easily.)

After cooling, dilute with distilled water to 100 ml (V_2) and mix thoroughly.

Remove the precipitate by filtration, using a dry, medium grade, ashless filter paper.

Discard the first 10 ml of the filtrate (it is imperative to carry out the operation in a fume cupboard as H_2S is given off). Using a pipette, transfer 20 ml (V_3) of the solution to a 100 ml conical flask. Add 5 ml of potassium cyanide solution and 3 ml ammonia [6 % (m/m)] and bring to the boil and then add 2 ml of 8-hydroxyquinoline solution. Boil again for a short time taking care to avoid any overflowing. Allow to solution to cool slowly to room temperature. Then add 50 ml methylene chloride and transfer the mixture to the 100 ml separating funnel. Quickly rinse the conical flask with water and add the rinsing solution to the separating funnel.

Shake briefly. Allow the gas to be given off and shake vigorously for 30 s. Collect the organic phase and transfer to a dry vessel using a dry, coarse grade, filter paper. Discard the first few millilitres. Measure the absorbance in relation to methylene chloride at a wavelength of 405 nm.

For carrying out the calibration, take from 1 ml (0,1 mg of Al) to a maximum of 10 ml (1 mg of Al) and mix with tartaric acid solution and the ammonia solution [25 % (m/m)]. Proceed as described above. Take 10 ml of the filtrate on precipitation of the sulphide and proceed as above. Prepare a calibration curve by plotting the absorbance values in terms of milligrams of Al per 50 ml of methylene chloride.

3.10.5 Expression of results

Al_2O_{3surf} content, expressed as a percentage by mass, is given by

$$w\left(Al_2O_{3\,surf}\right) = 1{,}889\,5 \times \frac{m_1 \times V_0 \times V_2}{m_0 \times 1\,000 \times V_1 \times V_3} \times 100$$

where

m_1 is the aluminium, in milligrams per 50 ml of methylene chloride;
m_0 is the test portion according to 3.5.4, in grams;
V_0 is the volume of stock solution according to 3.5.4, in millilitres;
V_1, V_3 are volumes sampled by pipette (in the present case V_1 = 50 ml and V_3 = 20 ml);
V_2 is the volume measured in 3.10.4 (in the present case, V_2 = 100 ml);
1,889 5 is the conversion factor, Al to Al_2O_3;
1 000 is the conversion factor, grams to milligrams.

3.11 Determination of surface aluminium oxide by atomic absorption spectrometry (AAS)

One can, alternatively, determine the surface aluminium oxide content ($Al_2O_{3\,surf}$) by atomic absorption spectrometry (AAS) using the filtrate obtained in 3.5.4 at the time of the determination of loss on acid treatment (LAT).

3.12 Determination of surface calcium oxide (CaO_{surf}) and of surface magnesium oxide (MgO_{surf})

3.12.1 Principle

After the separation of Fe and Al, determine the surface CaO by EDTA titration with the presence of a calceine indicator and then the surface CaO and surface MgO together be EDTA titration in the presence of methylthymol blue indicator. Calculate the MgO_{surf} content from the measured results.

3.12.2 Reagents

3.12.2.1 Ammonium chloride solution, 2 % (*m*/*m*).

3.12.2.2 Ammonia solution, 25 % (*m*/*m*).

3.12.2.3 Calceine indicator, sieved.

Grind 0,1 g of calceine together with 0,06 g of thymolphthaleine and 10 g of potassium chloride.

3.12.2.4 EDTA solution, (disodium salt of ethylene diamine tetra acetic acid), 0,05 mol/l.

3.12.2.5 Hydrochloric acid, 37 % (*m*/*m*).

3.12.2.6 Methylthymol blue indicator.

Grind 0,2 g of methylene thymol blue with 20 g of potassium nitrate.

3.12.2.7 Potassium hydroxide solution, 25 % (*m*/*m*).

3.12.2.8 Triethanolamine solution, 50 % (*V*/*V*).

3.12.3 Apparatus

3.12.3.1 Pipette, 50 ml.

3.12.3.2 Beaker, 400 ml.

3.12.3.3 Volumetric flask, 250 ml.

3.12.3.4 Conical flask, 500 ml.

3.12.3.5 Burette, 10 ml.

3.12.3.6 Filter paper, coarse grade, ashless.

3.12.4 Procedure

3.12.4.1 Separation of iron and aluminium

Transfer 150 ml (V_1) of the stock solution (V_0) obtained in 3.5.4 to a 400 ml beaker by means of a pipette. Add 2 g NH_4Cl, bring to the boil and precipitate the hydroxides by adding dropwise the ammonia solution until a pH of 6,8 to 7,0 is reached. Filter through a coarse ashless filter paper and wash with an ammonium chloride solution [2 % (m/m)]. If necessary, evaporate the filtrate to less than 250 ml. After cooling, transfer the solution to a 250 ml volumetric flask and make up to the mark (V_2).

3.12.4.2 Calcium titration

Pour 100 ml (V_3) of solution V_2 (see 3.12.4.1) into a 500 ml conical flask. Add 5 ml of triethanolamine solution and 10 ml of potassium hydroxide solution and then dilute to approximately 200 ml. After having added approximately 0,015 g of calceine indicator, titrate with an EDTA solution (0,05 mol/l) by means of a 10 ml burette until the colour changes from fluorescent green to non-fluorescent violet.

3.12.4.3 Combined titration of calcium and magnesium

Pour 100 ml (V_3) of the test solution (V_2) (see 3.12.4.1) into a 500 ml conical flask. Add 10 drops of hydrochloric acid [37 % (m/m)], 20 ml triethanolamine solution and 25 ml of ammonia solution [25 % (m/m)]. Dilute to 200 ml.

Add approximately 0,04 g of methylthymol blue indicator and titrate with an EDTA solution (0,05 mol/l) by means of a 10 ml burette, until the blue colour disappears.

3.12.5 Expression of results

1 ml of EDTA solution (0,05 mol/l) $\triangleq$ 0,002 g Ca = 0,002 8 g CaO

CaO_{surf} content, expressed as a percentage by mass, is given by the equation

$$w\left(\mathrm{CaO_{surf}}\right)=\frac{0{,}002\,8\times V_4\times V_0\times V_2}{m_0\times V_1\times V_3}$$

1 ml of EDTA solution (0,05 mol/l) $\triangleq$ 0,001 2 g Mg $\approx$ 0,002 g MgO

Mg_{surf} content, expressed as a percentage by mass, is given by the equation

$$w\left(\mathrm{Mg_{surf}}\right)=\frac{0{,}002\times\left(V_5-V_4\right)\times V_0\times V_2}{m_0\times V_1\times V_3}\times 100$$

where

m_0 is the test portion according to 3.5.4, in grams;

V_0 is the volume of stock solution according to 3.5.4, in millilitres;

V_1 is the volume taken from the stock solution according to 3.12.4.1, in millilitres;

V_2 is the volume of test solution according to 3.12.4.1, in millilitres;

V_3 is the volume taken from test solution according to 3.12.4.2 and 3.12.4.3, in millilitres;

V_4 is the volume of EDTA solution for the Ca titration, in millilitres;

V_5 is the volume of EDTA solution for the combined Ca and Mg titration, in millilitres.

3.13 Determination of surface magnesium oxide (MgO_{surf}) and of surface calcium oxide (CaO_{surf}) by atomic absorption spectrometry (AAS)

The MgO_{surf} and CaO_{surf} contents can be determined alternatively by atomic absorption spectrometry (AAS) using the filtrate obtained according to 3.5.4 at the time of the determination of the loss on acid treatment (LAT).

4 Determination of silicon carbide (SiC) in crushed silicon carbide

4.1 Principle

Determination of the total and free carbon on the crushed sample. On the basis of these determinations, calculate the SiC content.

4.2 Preparation of sample

4.2.1 Crushing and grinding

Pre-dry approximately 20 kg of sample made up of several pieces in an oven at (110 ± 5) °C (the mass depends on the quantity required for the subsequent grinding and quartering operations).

Crush the sample in a laboratory jaw crusher to a particle size smaller than 2,5 mm. Sieve the sample through a 2,5 mm sieve, crush the oversize residue and sieve again. Continue in this manner until the whole sample has passed through the sieve. Crush the last coarse grains in a steel mortar as the jaw crusher is ineffective for small quantities. Mix the whole sample until homogeneous.

Then take a 1 kg sample of the crushed product. This operation can be performed using a sample splitter of the type described in ISO 9138.

Grind the 1 kg, < 2,5 mm particle size sample in a laboratory roll crusher in order to obtain a product with a particle size smaller than 1 mm by proceeding in the same manner as above until all the sample passes through a 1 mm sieve. Mix the ground sample to less than 1 mm and then take a 100 g sample using a sample splitter.

Continue to grind with the aid of a suitable apparatus (e.g. a steel mortar) until the particle size is less than 0,5 mm. Sieve the 100 g sample having a particle size smaller than 1 mm through a 0,5 mm sieve. Grind the oversized residue in portions (3 g to 4 g at a time in a steel mortar). Continue grinding until no residue remains in the 0,5 mm sieve. Mix carefully and reduce the sample to 25 g with the aid of a sample splitter.

Grind the 25 g sample having a particle size smaller than 0,5 mm in the same way as above in order to obtain a size distribution < 0,15 mm and mix carefully.

4.2.2 Drying

Dry the finely ground sample for analysis until a constant weight is obtained in the manner described in 4.2.1 using an oven at (110 ± 5) °C.

4.2.3 Correction of the sample mass

If a steel mortar is used, the amount of iron added to the sample during grinding is generally less than 0,1 % (*m*/*m*) and is, therefore insignificant. If the quantity of iron is greater than 0,1 % (*m*/*m*), it is advisable to make a parallel determination on a sample that is iron free (see 3.8) and to take account of the corresponding result. When determining the iron content, it is preferable to grind the sample in a hard metal or boron carbide mortar.

As the accuracy of measurement shall be 0,01 % (*m*/*m*) maximum, if the amount of iron due to grinding is assumed to approach 1 % (*m*/*m*), then the mass of the sample for analysis has to be corrected on the basis of the following equation taking into account the quantity of iron (this formula is applicable to all analytical determinations):

$$m_0 = \frac{m_1 \times [100 - w(\text{Fe})]}{100}$$

where

m_0 is the corrected mass of the sample in grams;

m_1 is the mass of analysis sample containing the metal, dried according to 4.2.2, in grams;

$w(\text{Fe})$ is the quantity of iron added due to grinding as a percentage by mass of the analysis sample.

4.3 Determination of total carbon (C_{total})

4.3.1 Principle

Gravimetric or coulometric determination of the total carbon after adding a fusion mixture under oxygen.

4.3.2 Test methods

4.3.2.1 Gravimetric method

4.3.2.1.1 Reagents

Same reagents as indicated in 3.4.2.2.1 and

4.3.2.1.1.1 Triplumbic tetraoxide (Pb_3O_4), used as fusion mixture.

4.3.2.1.1.2 Aluminium oxide (Al_2O_3), particle size between 0,1 mm and 0,3 mm.

4.3.2.1.2 Apparatus

Same apparatus as stated in 3.4.2.1.1 and

4.3.2.1.2.1 Weighed flask.

4.3.2.1.3 Procedure

Take from the sample prepared according to 3.2, a test portion (m_0) of 0,25 g weighed to within ± 0,000 1 g and put it into the weighed flask. Add 2,0 g Pb_3O_4 and mix carefully. Transfer the mixture to a combustion boat, the bottom of which has been covered with Al_2O_3 grains (see 4.3.2.1.1.2).

Purge the apparatus for 10 min to 15 min with a stream of oxygen before carrying out the combustion process. Weigh the absorption tube and re-install it in the apparatus.

Place the combustion boat containing the sample in the hot zone (at 1 100 °C to 1 200 °C) of the tube furnace. Then pass a stream of oxygen through for 25 min (300 ml of O_2/min).

Remove the absorption tube, cool it to room temperature and weigh. The increase in mass corresponds to the carbon dioxide produced.

4.3.2.1.4 Expression of results

Total carbon content, expressed as a percentage by mass, is given by the equation

$$w(C_{total}) = \frac{(m_2 - m_1) \times 0{,}272\,9}{m_0} \times 100$$

where

m_1 is the mass of the absorption tube before combustion, according to 4.3.2.1.3, in grams;

m_2 is the mass of the absorption tube after combustion, according to 4.3.2.1.3, in grams;

m_0 is the mass of the test portion, according to 4.2.3, in grams.

$$0{,}272\,9 = \frac{M_r(\mathrm{C})}{M_r(\mathrm{CO_2})}$$

4.3.2.2 Coulometric method

4.3.2.2.1 Reagents

Same reagents as stated in 3.4.2.2.1 and 3.4.2.3.1.

4.3.2.2.2 Apparatus

Same apparatus as stated in 3.4.2.2.2.

4.3.2.2.3 Procedure

Take from the sample prepared according to 4.2, a test portion (m_0) weighing 0,04 g to within ± 0,000 1 g, place in a combustion boat and cover with 1 g of lead borate. Place the combustion boat in the tube furnace, which has been previously heated to a temperature of (1 050 ± 30) °C. (It should be borne in mind that the internal furnace temperature is not always the same as that of the combustion chamber.) Adjust the flow of oxygen so as to prevent the introduction of air into the system. Fractionate the gas, that is to say, only use a fraction of the gas for analytical purposes (generally 1/10th). Combustion will be completed after about 5 min. For the coulometric determination of the carbon dioxide produced, proceed in the manner indicated in 3.4.2.2.

Determine the carbon content of the lead borate three times using identical amounts of lead borate, the same method and the same duration for combustion of the sample. Use the mean of the three determinations as the blank test value and deduct it from the total carbon content found in the analysis sample.

4.3.2.2.4 Expression of results

Total carbon content, expressed as a percentage by mass, is given by the equation

$$w(\mathrm{(C_{total}}) = \frac{(l \times f) - (l' \times f') \times x}{m_0 \times 1\,000} \times 100$$

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 given by the blank test, according to 4.3.2.2.3;

f' is the gas fractionation coefficient used for blank determination;

m_0 is the mass of the test portion, according to 4.2.3, in grams;

x is the proportionality coefficient, depending on the apparatus, for converting pulses into milligramms of carbon;

1 000 is the conversion factor, grams to milligrams.

4.4 Determination of free carbon (C_{free})

4.4.1 Principle

Gravimetric or coulometric determination of the free carbon by calcination of the sample in a stream of oxygen without any additives.

If, in the coulometric determination the oxidation of the SiC must be taken into account, then it is necessary to determine the variation in the weight of the calcined residue and the total quantity in the residue. The gravimetric method also takes into account the oxidation of the SiC.

4.4.2 Test methods

4.4.2.1 Gravimetric method

Analyse the sample, prepared according to 4.2, in the manner described in 3.4.2.1.

4.4.2.2 General coulometric method

Analyse the sample, prepared according to 4.2, in the manner described in 3.4.2.2.

4.4.2.3 Special coulometric method

This coulometric method takes into account the oxidation of the SiC and/or may be used if C_{free} content is greater than 2 % (*m/m*).

Analyse the sample, prepared according to 4.2, in the manner described in 3.4.2.3.

4.5 Calculation of the silicon carbide (SiC) content

From the measurement results obtained, calculate the quantity of silicon carbide (SiC) in % by mass using the following equation

$$w(\mathrm{SiC}) = \left[w(\mathrm{C_{total}}) - w(\mathrm{C_{free}})\right] \times 3{,}338\,3$$

where

$w(\mathrm{C_{total}})$ is the total carbon content, as a percentage by mass, according to 4.3;

$w(\mathrm{C_{free}})$ is the free carbon content, as a percentage by mass, according to 4.4.

$$3{,}338\,3 = \frac{M_\mathrm{r}(\mathrm{SiC})}{M_\mathrm{r}(\mathrm{C})}$$

Round the result nearest 0,1 %.

5 Test report

The test report shall, in addition to the results, indicate the method used (reference to this International Standard), all the test conditions and all the details of procedures which are not provided for in this International Standard or which are optional, as well as all incidents likely to have had an effect on the results.

Annex A

(informative)

Other methods of analysis

In addition to the methods described in this International Standard radiation fluorescence analysis (RFA) or atomic absorption spectrometry (AAS) may also be used, provided a correlation has been made with this International Standard.

Annex B
(informative)

Permissible deviation as a result of variations in the measuring technique for the chemical analysis of silicon carbide

When checking the measured results allowance must be made for the variations in the measuring technique.

The permissible deviations given in table B.1 have been determined on the basis of the standard deviations resulting from a co-operative test carried out by ISO.

Table B.1 — Permissible deviations on test results

Measured component	**Deviation** % (*m*/*m*)
Silicon carbide	± 0,40
Total carbon	± 0,10
Free carbon	± 0,07
Iron	± 0,07

ICS 25.100.70

Descriptors: tools, abrasives, non-metallic abrasives, silicon carbides, chemical analysis, determination of content, impurities.

Price based on 22 pages