
Abrasive grains and crude — Chemical analysis of silicon carbide

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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 9286:1997), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the Scope has been shortened so that it only contains the subject of the document and the aspects covered;
- the structure of document has been revised in its entirety due to several new subclauses;
- new [Clause 3](#) "Terms and definitions" has been added;
- [4.3](#) (former 3.2) "Preparation of sample" has been revised;
- [4.4](#) (former 3.4) "Determination of surface carbon ($C_{\text{surf/free}}$)" has been revised, consisting of [4.4.2](#) "Detection by gravimetric method" and [4.4.3](#) "Detection by infrared absorption (IR)" with direct and indirect method;
- [4.5](#) (former 3.6) "Determination of surface silicon dioxide ($\text{SiO}_{2\text{surf}}$)" has been revised, consisting of [4.5.2](#) "Detection by HF/KF dissolving reactions" and [4.5.3](#) "Hydrofluoric acid loss"; and [4.5.4](#) "Molybdenum blue spectrophotometry" has been added;
- [4.6](#) (former 3.3) "Determination of surface silicon (Si_{surf})" has been revised; and [4.6.3](#) "Silver displacement method" and [4.6.4](#) "Molybdenum blue spectrophotometry" have been added;
- former 3.7 "Calculation of the content of residual silicon carbide (SiC_R)" has been moved to [4.10](#);
- former 3.8 and 3.9 for the determination of surface iron have been revised and moved to [4.9](#), consisting of the following detection methods: atomic absorption spectrometry (AAS) and induced coupled plasma (ICP);

- former 3.10 and 3.11 for the determination of surface aluminium oxide have been deleted; and determination of surface aluminium has been added to [4.9](#);
- former 3.12 for the determination of surface calcium oxide and surface magnesium oxide has been deleted; and determination of surface calcium and surface magnesium has been added to [4.9](#);
- former 4.3 "Determination of total carbon" has been revised by adding the detection by infrared absorption (IR) and has been moved to [4.8](#);
- former Annexes A and B have been deleted.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Abrasive grains and crude — Chemical analysis of silicon carbide

1 Scope

This document specifies 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 the determination of the SiC content of crushed crude when the silicon carbide content is greater than 95 % (mass fraction).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9138, *Abrasive grains — Sampling and splitting*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Analysis of surface impurities

4.1 General

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

All reagents used for the analysis specified in this document shall be of minimum p.a. (pro analysis) quality.

4.2 Sampling

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

4.3 Preparation of sample

4.3.1 Fine grains

This applies to the determination of the surface impurities of abrasive grains in their original grain size and state for grain sizes F500 and finer as well as for grain sizes P1500 and finer. The surface chemical analysis shall be carried out on unprocessed abrasive grains.

The sample shall be dried at (110 ± 5) °C until constant mass is obtained.

4.3.2 Coarse Grains

4.3.2.1 Sample preparation for surface impurity determination

This applies to abrasive grains in their original grain size and state for grain sizes F400 and coarser as well as for grain sizes P1200 and coarser.

The sample shall be dried at (110 ± 5) °C until constant mass is obtained.

4.3.2.2 Sample preparation for total and free carbon determination

This applies to abrasive grains in their original grain size and state for grain sizes F150 to F400 as well as for grain sizes P180 to P1200.

The sample shall be dried at (110 ± 5) °C until constant mass is obtained.

Grain sizes F120 and coarser as well as grain sizes P150 and coarser shall be crushed until all the material passes through a 150 µm sieve.

If a steel mortar is used, the quantity of additional iron contaminant in the sample is generally less than 0,1 % (mass fraction) and therefore insignificant. If the quantity of additional iron contaminant is higher than 0,1 % (mass fraction), it is advisable to make a parallel determination on a sample that is iron free (according to 4.9), taking the added iron due to grinding in the steel mortar into account. The sample shall then be ground using a hard metal or boron carbide mortar.

The mass of the sample for analysis shall be corrected based on Formula (1).

$$m_s = \frac{m_{s1} \cdot (100 - c)}{100} \quad (1)$$

where

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m_s is the corrected mass of the sample, in grams (g);

m_{s1} is the mass of the sample containing the metal, dried according to 4.3.2.1, in grams (g);

c is the quantity of iron added due to grinding, mass fraction in per cent (%).

4.3.3 Crude

The crude sample shall be prepared by the following procedure:

Pre-dry an approximate 20 kg sample made up of several pieces in a heating cabinet at (110 ± 5) °C.

Crush the sample in a laboratory jaw crusher to a grain 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 laboratory jaw crusher is ineffective for small quantities. Mix the whole sample until it is homogenous.

Reduce the sample size to 1 kg by using a sample splitter according to ISO 9138.

Grind the 1 kg sample (< 2,5 mm) in a laboratory roll crusher to a grain size smaller than 1 mm. Sieve the sample through a 1 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 laboratory roll crusher is ineffective for small quantities. Mix the whole sample until it is homogenous.

Reduce the sample size to 100 g by using a sample splitter according to ISO 9138.

Grind the 100 g sample (< 1 mm) in a suitable apparatus (e.g. steel mortar) to a grain size smaller than 0,5 mm. Sieve the sample through a 0,5 mm sieve, crush the oversize residue and sieve again. Continue

in this manner until the whole sample has passed through the sieve. Mix the whole sample until it is homogenous.

Reduce the sample size to 25 g by using a sample splitter according to ISO 9138.

Grind the 25 g sample (< 0,5 mm) in the same way as above in order to obtain a grain size distribution smaller than 0,15 mm. Mix the whole sample until it is homogenous.

Dry the finely ground sample until a constant mass is obtained using a heating cabinet at $(110 \pm 5) ^\circ\text{C}$.

If a steel mortar is used, the quantity of additional iron contaminant in the sample is generally less than 0,1 % (mass fraction) and therefore insignificant. If the quantity of additional iron contaminant is higher than 0,1 % (mass fraction), it is advisable to make a parallel determination on a sample that is iron free (according to 4.9), taking the added iron due to grinding in the steel mortar into account. The sample shall be ground using a hard metal or boron carbide mortar.

The mass of the sample for analysis shall be corrected based on [Formula \(1\)](#).

4.4 Determination of surface carbon ($C_{\text{surf/free}}$)

4.4.1 Principle

Depending on the sample preparation, the resulting values are used for the determination of impurity (C_{surf}) or the calculation of silicon carbide content (C_{free}).

For grain sizes finer than F120 and P150: ($C_{\text{surf}} = C_{\text{free}}$).

Determination of carbon is obtained by heating the sample in a stream of oxygen inside a combustion furnace. The released carbon dioxide shall be detected by one of the following methods: gravimetric method (see 4.4.2) or infrared absorption (IR) (see 4.4.3).

4.4.2 Detection by gravimetric method

4.4.2.1 Apparatus

4.4.2.1.1 Combustion apparatus, according to [Figure 1](#).

4.4.2.2 Procedure

Prior to starting the measurements, purge the combustion furnace using a stream of oxygen for 10 min to 15 min. From the sample prepared according to 4.3 take a test portion (m) of 2 g weighed to within ± 1 mg and place it into a previously calcined and weighed combustion boat. Weigh the absorption tube to within ± 1 mg and insert it in the combustion furnace. Place the combustion boat containing the test portion into the hot zone of the tube furnace at $900 ^\circ\text{C}$ to $915 ^\circ\text{C}$. Pass a stream of oxygen for 30 min at a flow rate of 100 ml/min through the combustion apparatus. Remove the absorption tube and weigh it. The increase in mass corresponds to the mass of carbon dioxide (m_2). Determine the mass of the residue in the combustion boat (m_1) within ± 1 mg after cooling it in a desiccator.

4.4.2.3 Expression of results

The content of surface carbon is calculated according to [Formula \(2\)](#).

$$w_{C_{\text{surf}}} = \frac{(0,2729 \cdot m_2) - (0,3754 \cdot m_3)}{m} \cdot 100 \quad (2)$$

where

w_{C_surf} is the content of surface carbon (C_{surf}), mass fraction in per cent (%);

m is the mass of the test portion prior to combustion, in grams (g);

m_1 is the mass of the test portion after combustion, in grams (g);

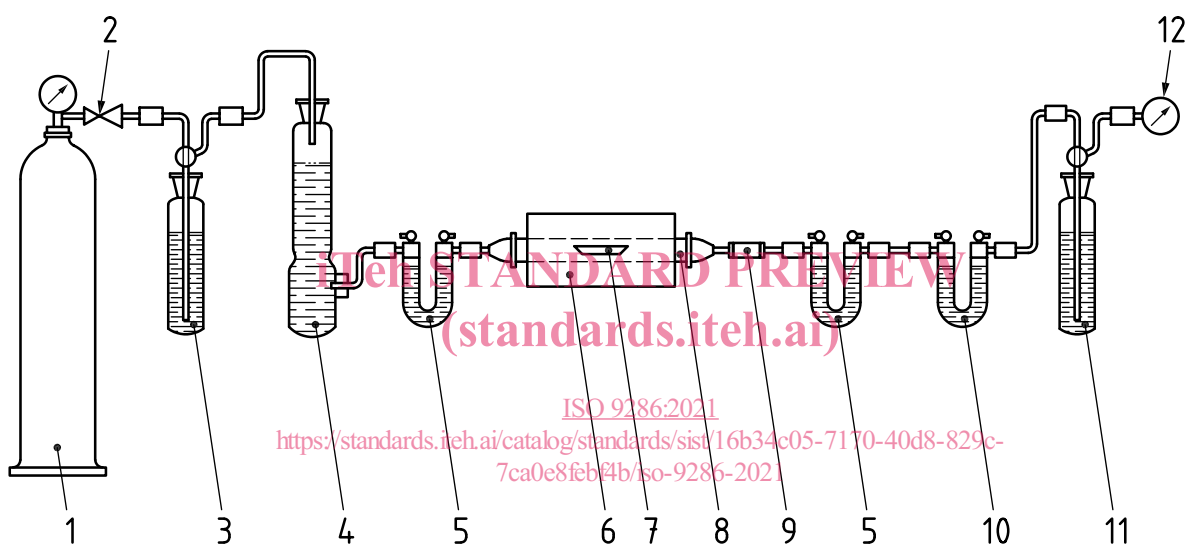
m_2 is the mass of carbon dioxide, in grams (g);

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

0,272 9 is the quotient of the molecular mass of carbon $M_r(C)$ and carbon dioxide $M_r(CO_2)$;

0,375 4 is the quotient of the molecular mass of carbon $M_r(C)$ and oxygen $M_r(O_2)$.

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



Key

- 1 oxygen cylinder
- 2 pressure controlling valve
- 3 gas washing bottle containing concentrated sulfuric acid
- 4 drying tower containing sodium hydroxide on a carrier
- 5 absorption U-tube containing magnesium perchlorate
- 6 resistance furnace controllable to 1 000 °C
- 7 combustion boat
- 8 combustion tube
- 9 tube filled with percarbamide
- 10 absorption U-tube containing sodium hydroxide on a carrier and magnesium perchlorate
- 11 gas washing bottle containing 5 % palladium (II) chloride solution
- 12 manometer

Figure 1 — Combustion apparatus for gravimetric determination using resistance furnace

4.4.3 Detection by infrared absorption (IR)

4.4.3.1 Direct method

4.4.3.1.1 Principle

The method may be applied if a consideration of silicon carbide oxidation is not necessary.

4.4.3.1.2 Apparatus

Different types of instruments for carbon determination analysis by combustion and IR detection are available. It consists of an oxygen cleaner, a combustion tube, a gas refiner and an IR analyser.

4.4.3.1.3 Procedure and expression of results

From the sample prepared according to 4.3, take a test portion (m). Follow the instructions for the instrument available and report the detected surface or free carbon.

4.4.3.2 Indirect method

4.4.3.2.1 Principle

The method shall be applied if a consideration of silicon carbide oxidation is necessary.

4.4.3.2.2 Apparatus

Use the apparatus as specified in 4.4.3.1.2.

4.4.3.2.3 Procedure

From the sample prepared according to 4.3, take a test portion (m). Follow the instructions for the instrument available. After combustion, cool the test portion in a desiccator and weigh the test portion (m_1) to an accuracy of ± 1 mg.

4.4.3.2.4 Expression of results

The content of surface or free carbon is calculated according to Formula (3).

$$w_{C_surf/free} = \frac{m_C - 0,3754 \cdot (m_1 - m + m_C)}{m} \cdot 100 \quad (3)$$

where

- $w_{C_surf/free}$ is the content of surface or free carbon ($C_{surf/free}$), mass fraction in per cent (%);
- m_C is the mass of carbon detected as CO_2 by the instrument, in grams (g);
- m_1 is the mass of the test portion after combustion, in grams (g);
- m is the mass of the test portion prior to combustion, in grams (g);
- 0,3754 is the quotient of the molecular mass of carbon $M_r(C)$ and oxygen $M_r(O_2)$.