ICS 25.100.70
CCS J 43

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 47571-2026

Methods for the chemical analysis of synthetic diamond

人造金刚石化学分析方法

Issued on: April 30, 2026 **Implemented on: November 1, 2026**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

Table of Contents

Foreword

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1.Structure and Drafting Rules of Standardization Documents". Drafting. Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents. This document was proposed by the China Machinery Industry Federation. This document is under the jurisdiction of the National Technical Committee on Standardization of Abrasives and Grinding Tools (SAC/TC139). This document was drafted by: Zhengzhou Abrasives & Grinding Research Institute Co., Ltd., Jinggong Boyan Testing Technology (Henan) Co., Ltd., and Zhongnan Drilling. Stone Co., Ltd., Henan Yellow River Whirlwind Co., Ltd., Henan Houde Diamond Technology Co., Ltd., Beijing University of Science and Technology, Henan United Precision Materials Co., Ltd. The companies mentioned are. Liaocheng Laixin Powder Materials Co., Ltd., Shandong Liaocheng Laixin Powder Materials Technology Co., Ltd., and Hunan Liangcheng New Materials Technology Co., Ltd. The main drafters of this document are. Xing Bo, Chen Xuebin, Bao Hua, Yi Liangcheng, Wang Yuchang, Tan Suling, Li Chengming, Li Pingping, Wang Lei, and Cao Xiaojun. Hu Yujing, Zhang Liang, Qi Yanjie, and Pu

Fenggang. Chemical analysis methods for synthetic diamond

1.Scope This document describes the determination of ash content using a high-temperature calcination method and the determination of surface and ash impurity elements using inductively coupled plasma atomic emission spectrometry. Content of (aluminum, boron, calcium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, molybdenum, sodium, niobium, nickel, phosphorus, lead, silicon, strontium, titanium, vanadium, tungsten, zinc, zirconium) determined by ion chromatography. Determination of inorganic anions (F-, Cl-, NO-2-, Br-, NO-3-, PO3-4-, SO2-4-) by ion chromatography; determination of inorganic cations (Li-, Na-,...) by ion chromatography. The content of NH4, K, Ca2+, and Mg2+ was determined by pulse-heated inert gas melting-infrared absorption/thermal conductivity method, and the oxygen and nitrogen content was determined by secondary method. Ion mass spectrometry (SIMS) for determining boron, nitrogen, and phosphorus content; glow discharge mass spectrometry (GD-MS) for determining impurity element content; and indirect methods for determining... Methods for determining the purity of nominal diamonds. This document applies to the determination of the chemical composition of synthetic diamonds.

3.Terms and Definitions This document does not contain any terms or definitions that need to be defined.

4 Sample Preparation

4.1 Powder/Particle Samples Sampling was performed according to JB/T 3914, and the sample was reduced to 5g~10g. The sample was placed in an oven and dried at 105°C~110°C for 1 hour. Remove the sample and immediately place it in a desiccator to cool to room temperature for later use.

4.2 Flake and block samples For sheet-like and block-like samples, use the original sample state or prepare the sample to a suitable size.

5 Determination of Ash Content by High-Temperature Ignition Method

5.1 Method Principles Under high-temperature calcination conditions, the carbon element (C) of synthetic diamond is oxidized to produce CO2, and the remaining substance is ash. Through precise... Weigh the sample before and after ignition, and characterize the ash content as a percentage of the mass of the residue to the mass of the original dried sample.

5.2 Instruments and Equipment

5.2.1 High-temperature furnace, the maximum heating temperature should be able to reach 1200°C.

5.2.2 Analytical balance with a scale division of 0.0001g.

5.2.3 Quartz dish (or ceramic dish).

5.3 Test Procedure Weigh 3g to 5g of the sample, accurate to 0.0001g, and place it in a quartz dish (or ceramic dish) that has been preheated to constant weight in a high-temperature furnace at 1100°C. Record the total mass of the sample and the quartz (or ceramic) dish. Place the sample container in a high-temperature furnace at 1000°C±20°C for ignition. 2 hours. After removing and cooling slightly, transfer to a desiccator to cool to room temperature, then weigh. Repeat the ignition process (30 minutes each time) until the difference in mass between two consecutive weighings is reached. A weight less than 0.0003g is considered constant, and the final mass is recorded.

5.4 Experimental Data Processing The ash content is expressed as its mass fraction (w_ash), calculated according to formula (1).

5.5 Tolerance The allowable error is shown in Table 1.

6.Determination of Surface Impurity Elements by Inductively Coupled Plasma Atomic Emission Spectrometry

6.1 Measurement Range The measurement ranges for various surface impurity elements are shown in Table 2.

6.2 Method Principles The sample was treated with a mixture of nitric acid and hydrofluoric acid to dissolve surface impurities under heating conditions. The standard curve method was used, and inductively coupled plasma was employed. Intra-atomic emission spectrometry (ICP-OES) was used to determine the elemental impurities (aluminum, boron, calcium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, molybdenum, sodium) on the surface of synthetic diamonds. Contents of niobium, nickel, phosphorus, lead, silicon, strontium, titanium, vanadium, tungsten, zinc, and zirconium.

6.3 Reagents and Materials Unless otherwise specified, only reagents confirmed to be of superior purity should be used in the analysis. For standard stock solutions, certified reference materials or standard reference materials should be used preferentially. Preliminary sample.

6.3.1 Nitric acid (rho=1.41g/mL).

6.3.2 Hydrochloric acid (rho=1.19g/mL).

6.3.3 Hydrofluoric acid (rho=1.15g/mL).

6.3.4 Nitric acid (1 1).

6.3.34 Mixed standard solution A. 40 μg/mL. Transfer

10.00 mL of each standard stock solution to a 250 mL volumetric flask, dilute to the mark with water, and mix well. 1 mL of this solution contains 40 μg of each element to be measured. Niobium, silicon, and tungsten standard stock solutions are alkaline matrices; phosphorus standard stock solution... It contains potassium, so standard solutions of niobium, silicon, tungsten, and phosphorus need to be prepared separately.

6.3.35 Mixed standard solution B. 20 μg/mL. Transfer

5.00 mL of each standard stock solution to a 250 mL volumetric flask, dilute to the mark with water, and mix well. 1 mL of this solution contains 20 μg of each element being analyzed. Niobium, silicon, and tungsten standard stock solutions are basic matrices; phosphorus standard stock solution... It contains potassium, so standard solutions of niobium, silicon, tungsten, and phosphorus need to be prepared separately.

6.4 Instruments and Equipment

6.4.1 Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-OES) It should meet the following performance indicators.

6.4.2 Electric heating acid removal device It has multiple pores and is compatible with PTFE digestion vessels. The heating temperature range is from room temperature to 250°C.

6.4.3 Analytical Balance The graduation value is no greater than 0.0001g.

6.5 Test Procedure

6.5.1 Preparation of analytical solutions Weigh 4-5g of the sample, accurate to 0.0001g, and place it in a 100mL polytetrafluoroethylene digestion vessel. Moisten with a small amount of water and add... Add 20 mL of nitric acid (1.1), sonicate to disperse the sample evenly, add

0.5 mL of hydrofluoric acid (1.1), cover with a plastic cap, and place at a temperature of [temperature missing]. Heating was performed in an electric acid-removing apparatus at 80°C for 2 hours. The digestion tube was then removed and allowed to cool to room temperature. The sample and solution were then completely disposed of. Transfer to a 100 mL plastic volumetric flask, dilute to volume with water, and mix well. Filter dry through a plastic funnel and slow-speed filter paper or membrane into a PTFE precipitate. Place in a cup or centrifuge tube. This analytical solution is used for the determination of surface impurity elements.

6.5.2 Preparation of blank test solution Prepare a blank solution according to

6.5.1 without adding a sample.

6.5.3 Plotting Working Curves According to the elemental content, transfer mixed standard