●Determination of Inorganic Impurities in Graphite
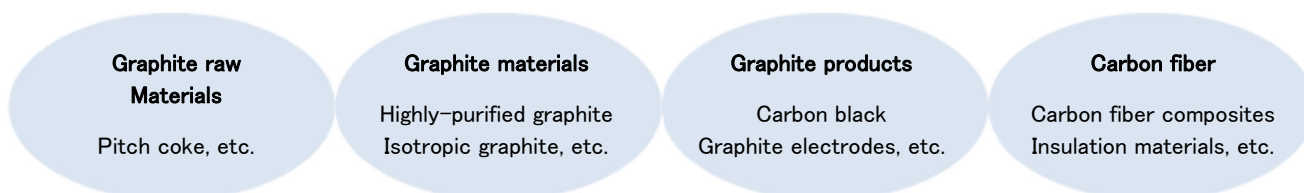
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[Overview]

Graphite materials have been used for many years in various fields due to their excellent heat resistance, chemical resistance, and processability . New materials, such as carbon fibers, have also been developed and applied to leading-edge technologies in aerospace, semiconductor fields and others.

Sumika Chemical Analysis Service, Ltd. developed graphite analysis techniques in the 1990s. Since then, we have continuously improved those techniques to meet the customers' demands for advanced analyses for higher purity of graphite materials. In recent years, we have developed high-sensitive analytical method using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and have provided analysis services using those techniques to a wide range of customers, including manufactures of graphite materials, graphite insulation materials, semiconductors, and others.

[Applicable samples]

Graphite raw Materials	Graphite materials	Graphite products	Carbon fiber
Pitch coke, etc.	Highly-purified graphite Isotropic graphite, etc.	Carbon black Graphite electrodes, etc.	Carbon fiber composites Insulation materials, etc.

We currently use two analysis methods for determining traces of elements in graphite. The main purposes and applicable samples of these methods are described below. Limits of quantification for various elements are listed on the next page.

[Analytical methods]

After samples are pulverized, they are ashed, dissolved, then determination will be carried out.   It is important to select pretreatment method that is appropriate for the target element and limits of quantification. Figure 1 shows basic test procedures.
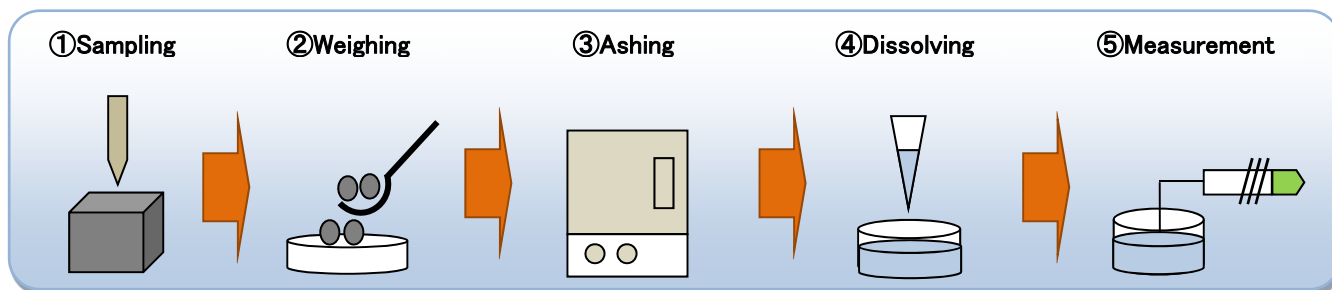
①Sampling   ②Weighing   ③Ashing   ④Dissolving   ⑤Measurement

Fig.1 Basic procedures

1.　Standard method

This analytical method mainly uses Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) and Atomic Absorption Spectroscopy (AAS). We recommend this method to customers who don't know sample compositions and/or impurity contents.   Table 1 shows limits of quantification for various elements.

・ This method is applicable to samples in various forms, such as carbon fibers, insulation materials, and special graphite materials.

・ This method is applicable to a wide range of samples from those with high purity materials to those high matrix samples.

・ About 100 grams of a sample is required for conducting an analysis using this method. (The required amount may vary depending on factors such as the number of elements to be determined.)

2.　High-sensitive method

This method uses Inductively Coupled Plasma Mass Spectrometry (ICP-MS). It can analyze elements at the concentration of ng/g (ppb) level. We recommend this method to customers who need to determine impurity contents with high sensitivity. (Table 2 shows limits of quantification for various elements.)

・This method is applicable to high-purity graphite materials with low residues or impurities. (We might recommend the standard method to customers who need analyses of materials with high matrix samples.)

・This method has been successfully used for analyzing carbon fiber composites and other special materials.

・A relatively small amount—about 10 grams—of a sample is required for this method. (The required amount may vary depending on factors such as the number of elements to be determined.)

Table 1 The Limit of quantification (LOQ) of each item in standard method using ICP–AES, AAS, etc.

Item	LOQ μg/g (ppm)	Item	LOQ μg/g (ppm)	Item	LOQ μg/g (ppm)	Item	LOQ μg/g (ppm)	Item	LOQ μg/g (ppm)
Ag	0.05	Co	0.06	Hg	0.1	Nb	0.1	Ta	0.3
Al	0.08	Cr	0.07	Ho	0.05	Ni	0.1	Tb	0.2
As	0.2	Cs	0.2	I	10	P	1	Te	1
B	0.1	Cu	0.08	In	0.2	Pb	0.2	Ti	0.09
Ba	0.03	Dy	0.05	K	0.1	S	1	Tm	0.5
Be	0.02	Eu	0.05	La	0.05	Sb	0.2	V	0.07
Bi	0.2	F	1	Li	0.01	Sc	0.02	W	0.5
Br	3	Fe	0.04	Lu	0.05	Se	0.2	Y	0.1
Ca	0.04	Ga	0.2	Mg	0.02	Si	0.1	Zn	0.1
Cd	0.07	Gd	0.05	Mn	0.03	Sm	0.05	Zr	0.04
Ce	0.2	Ge	1	Mo	0.2	Sn	0.5	灰分	5
Cl	3	Hf	0.1	Na	0.05	Sr	0.03		

Note: The LOQ values may vary depending on the amount of sample available for analysis, sample composition, residues, etc.

Table 2 The LOQ of each item in high sensitivity method using ICP–MS

Item	LOQ ng/g (ppb)	Item	LOQ ng/g (ppb)	Item	LOQ ng/g (ppb)	Item	LOQ ng/g (ppb)	Item	LOQ ng/g (ppb)
Al	5	Co	5	K	5	P	5	V	1
As	5	Cr	5	Li	5	Pb	5	W	5
B	10	Cs	5	Mg	2	Rb	5	Zn	5
Ba	5	Cu	5	Mn	2	Sr	5	Zr	5
Be	10	Fe	5	Mo	5	Th	0.1		
Bi	5	Ga	5	Na	5	Ti	2		
Ca	5	Ge	5	Nb	5	Tl	5		
Cd	5	In	5	Ni	5	U	0.1		

Note: The LOQ values may vary depending on the amount of sample available for analysis, sample composition, residues, etc.
The quantifiable items in the high sensitivity method differ from those of the standard method depending on the difference in sample pretreatment and analysis principle.

[Keywords]

Carbon, Graphite, CC Composite, Carbon Fiber Reinforced Carbon Composite