

Analysis of Graphite Electrodes for determination of Boron at trace levels

Graphite has many industrial applications. The two most important applications are as electrodes in industries and as moderator in nuclear industry. Graphite being a dual use material, faces export restrictions under the Atomic Energy Act 1962. Analytical Chemistry Division, BARC receives regularly graphite samples for its characterization as nuclear/non nuclear grade. A method has been standardized at Analytical Chemistry Division, BARC for determining trace amounts of boron, which is a marker element for certifying the grade of graphite electrode. The method involves dry ashing of graphite after fixing boron using barium hydroxide and determination by Inductively Coupled Plasma Mass Spectrometer (ICP-MS) using matrix matched standards. The method detection limit is $1 \mu\text{g g}^{-1}$ with a method RSD of 5%. The method was verified by spike recovery experiments. Recoveries were found to be $100 \pm 2\%$ in the concentration range of 1 to $100 \mu\text{g g}^{-1}$. Ion Beam Analysis (IBA) was used to validate the present method. Values were found to be in very good agreement. This method has been applied for the routine analysis of boron content in various graphite electrode samples received from the Customs and Central Excise Department.

Typical Graphite Electrode received for analysis



Validation of ignition temperature and time

Spike Quantity(μg)	Quantity obtained (μg)	% Recovery
25	24.5	98.0
50	48.5	97.0
100	101.0	101.0

Comparison of ICP-MS and IBA results

ACD No	SampleCode	ICPMS ($\mu\text{g g}^{-1}$)	IBA (NRA) ($\mu\text{g g}^{-1}$)
N-9507	3388	43.0 ± 3.0	46.0 ± 1.0
N-9508	3406	51.0 ± 2.7	53.0 ± 1.6
N-9509	3255	55.0 ± 2.5	56.0 ± 1.5
N-9510	3340	46.0 ± 3.0	47.0 ± 1.1
N-8756	3342	65.0 ± 4.0	68.0 ± 2.0
O-9	165	15.0 ± 1.0	16.0 ± 0.5
O-81	202	2.0 ± 0.2	2.0 ± 0.1