Analysis of Trace Elements in High-Purity Graphite Using the Prodigy DC Arc Spectrometer

Introduction

Graphite is one of the softest minerals in existence, has incredible thermal stability (melts at 3650 °C) and is an excellent electrical and thermal conductor. The majority of naturally occurring graphite is processed into a fine powder for the production of materials such as: steels, lubricants, industrial coatings, rubber and plastic additives, brake linings, batteries, electrodes and is used in gas-cooled nuclear reactors.

This application note contains data to demonstrate the ability of the Teledyne Leeman Lab's **Prodigy DC Arc** to determine trace elements in high-purity graphite.

Experimental

Operating Parameters

Standards were in the form of powdered graphite and were analyzed in their native form without the addition of a powdered internal standard. All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** without the use of a Stallwood Jet. Emission from all elements was collected from 0-20 seconds and the remaining instrument and method conditions are listed in Table I.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignite at 6A, ramp to 12A over 5 s, hold at 12A for 15 s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	1/8" diameter pedestal with a boiler cup (ASTM #S-1, #S-2)
Sample	
Sample Size	Hand packed, ~80 mg
Internal Standard	None
Integration Time	0-20 s

All standards were prepared for analysis by mixing each with a prepared chemical solution that was used to improve the efficiency with which samples burn and produce emission. A chemical solution was prepared for addition to each standard once it was hand packed into a sample electrode.

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The solution was prepared by dissolving 0.5 g silver chloride and 0.125 g cesium fluoride into 6.25 mL of ammonium hydroxide in a volumetric flask. After all solids were dissolved, the solution was brought to the volume mark with deionized water. Once standards were hand packed into sample electrodes, 35 µL of the solution described above was added to each standard. All packed electrodes were then dried in an oven at 100 °C for 1 hour.

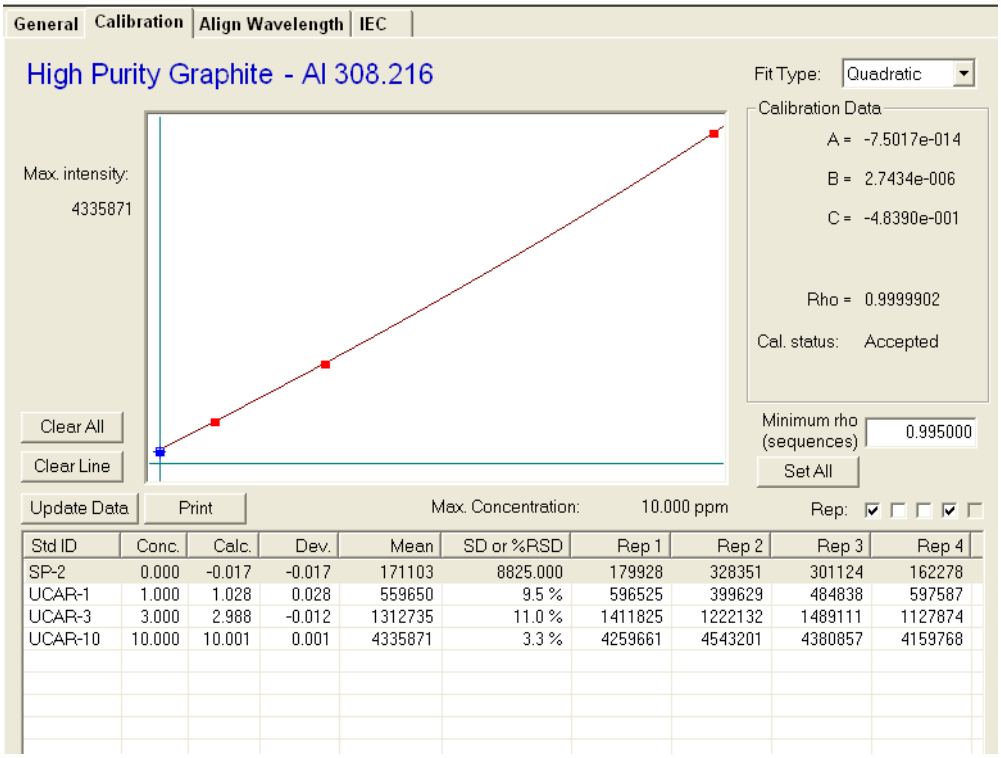
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were a 1/8" diameter pedestal (part # S-1) with a boiler cup (part # S-2). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm analytical gap was used and the position of the electrodes was adjusted during the sample burn to maintain a distance of 4 mm between the sample and the counter electrode.

Calibration

The instrument was calibrated with several high-purity graphite standards that contained the analytes of interest at 0, 1, 3 and 10 ppm. Calibration standards were prepared in this matrix by serial dilution on a weight-to-weight basis from a multi-element stock standard containing 45 elements at 1.21% (MV Laboratories, Inc., Frenchtown, NJ). All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in high-purity cobalt is illustrated in Figure 1 for Al at 308.216 nm. The calibration curve for Al demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve of Al at 308.216 nm in High-Purity Graphite



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
SP-2	0.000	-0.017	-0.017	171103	8825.000	179928	328351	301124	162278
UCAR-1	1.000	1.028	0.028	559650	9.5 %	596525	399629	484838	597587
UCAR-3	3.000	2.988	-0.012	1312735	11.0 %	1411825	1222132	1489111	1127874
UCAR-10	10.000	10.001	0.001	4335871	3.3 %	4259661	4543201	4380857	4159768

Results

Detection Limits

A study was performed to determine the instrument's detection limits for the elements of interest. Detection limits were calculated based on 3 times the standard deviation of 10 replicate measurements of the calibration blank. Results for the detection limit study are listed in Table II in units of parts per million (ppm).

Table II Detection Limits in High-Purity Graphite

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Al	308.216	0.13	0 – 20	Mn	259.373	0.008	0 – 20
As	193.759	0.32	0 – 20	Na	589.592	0.73	0 – 20
B	249.773	0.027	0 – 20	Ni	341.477	0.005	0 – 20
Ca	396.847	0.32	0 – 20	P	253.565	0.055	0 – 20
Cd	226.502	0.021	0 – 20	Pb	283.307	0.026	0 – 20
Cr	283.563	0.010	0 – 20	Sb	231.147	0.034	0 – 20
Cu	327.396	0.043	0 – 20	Si	252.412	0.22	0 – 20
Fe	259.940	0.021	0 – 20	Sn	283.999	0.006	0 – 20
Ga	294.364	0.014	0 – 20	Ti	337.280	0.027	0 – 20
K	766.491	0.61	0 – 20	V	309.311	0.008	0 – 20
Li	670.784	0.020	0 – 20	Zn	213.856	0.009	0 – 20
Mg	285.213	0.058	0 – 20				

Conclusions

The analysis of graphite using the **Prodigy DC Arc** demonstrates that the current-controlled DC Arc power supply, combined with the simultaneous data collection of both peak and background data, provides reproducible sample burns that are reflected in the detection limits obtained for trace elements in a graphite matrix.

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