Analysis of Trace Elements in Silicon Dioxide Using the Prodigy DC Arc Spectrometer

Introduction

Silicon dioxide, also known as silica, can be produced by oxidizing the surface of silicon material or can be found naturally occurring in the form of sand or quartz. Silica is a good electrical insulator, has high thermal stability and is resistant to abrasion. For these reasons, silica finds use in the production of glass for beverage bottles, drinking glasses and windows. It is also used as the primary component in optical fibers used for telecommunication and in ceramic materials such as porcelain and stoneware.

Silica can be manufactured into several different forms, one of which is fused silica. Fused silica, a high-purity grade of silica, often refers to silicon dioxide that is 99.4-99.9% pure. At 1830 °C, this material has a high melting point which, combined with its dielectric and insulating properties, make it useful in many electronic applications.

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 silicon dioxide.

Experimental

Operating Parameters

All standards were prepared for analysis by mixing with high-purity graphite such that the ratio of sample to graphite was 1:1. The mixtures were thoroughly blended with a SPEX mixer/mill for a minimum of 10 minutes before hand packing into sample electrodes.

All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** in air without the use of the Stallwood Jet. The remaining instrument and method conditions used are listed in Table I.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignite at Ignite at 6A, hold at 6A for 15s, jump to 15A, hold at 15A for 100s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	3/16" diameter and pointed (ASTM #C-2)
Sample Electrode	3/16" diameter with an undercut cup (ASTM #S-15)
Sample	
Sample Size	Hand packed, ~15 mg
Internal Standard	None
Integration Time	Individual time gates were used

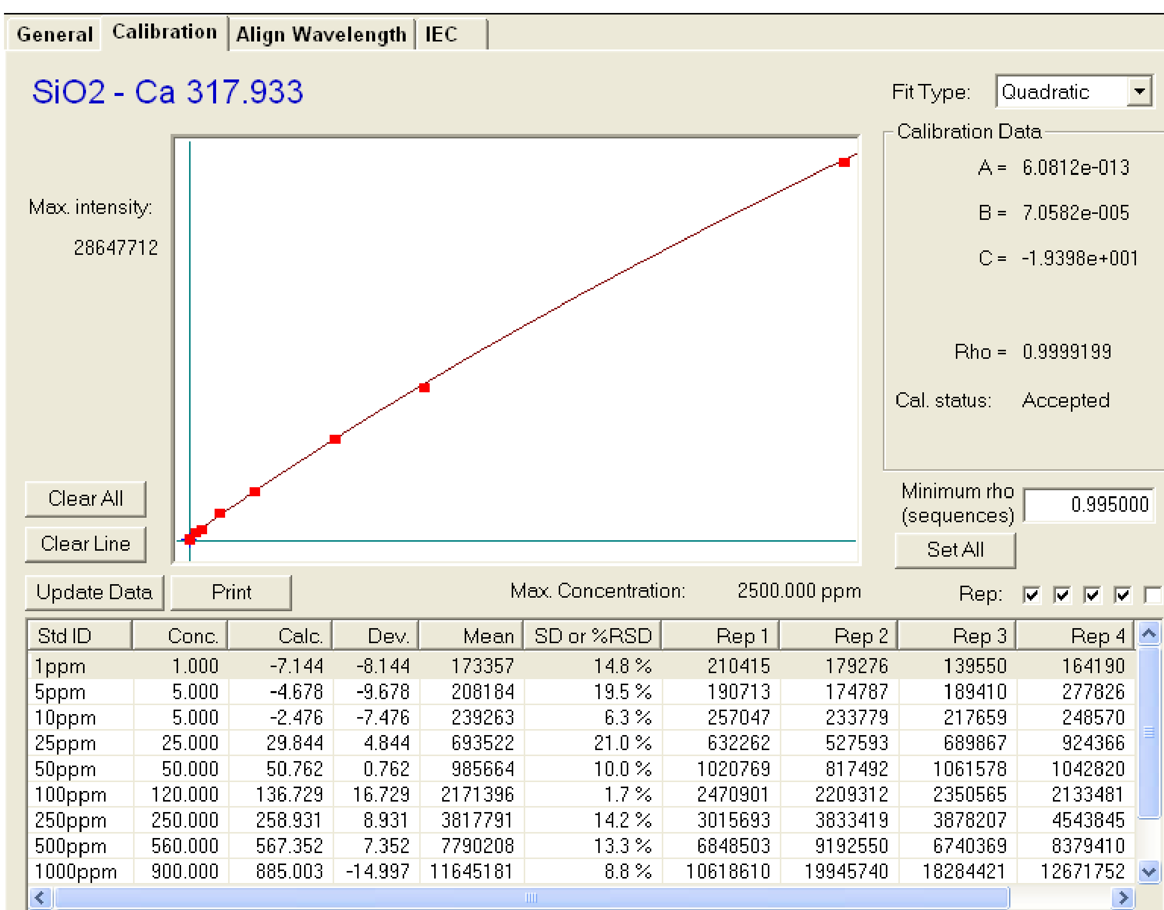
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16” in diameter with an undercut cut (part # S-15). The counter electrodes used for all analyses were 3/16” in diameter and pointed (part # C-2). A 4 mm analytical 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 silicon dioxide standards that were spiked with a multielement stock standard containing 45 elements at 1.21% (MV Laboratories, Inc., Frenchtown, NJ). Calibration standards were prepared in this matrix by serial dilution on a weight-to-weight basis such that the analytes of interest were present at 0, 1.0, 5.0, 10, 25, 50, 100, 250, 500, 1000, 2500 and 5000 ppm in the silicon dioxide matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example of a typical calibration curve for elements measured in silicon dioxide is illustrated in Figure 1 below. Figure 1 contains a calibration curve for Ca at 317.933 nm and demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve for Ca at 317.933 nm in High-Purity Silicon Dioxide



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
1ppm	1.000	-7.144	-8.144	173357	14.8 %	210415	179276	139550	164190
5ppm	5.000	-4.678	-9.678	208184	19.5 %	190713	174787	189410	277826
10ppm	5.000	-2.476	-7.476	239263	6.3 %	257047	233779	217659	248570
25ppm	25.000	29.844	4.844	693522	21.0 %	632262	527593	689867	924366
50ppm	50.000	50.762	0.762	985664	10.0 %	1020769	817492	1061578	1042820
100ppm	120.000	136.729	16.729	2171396	1.7 %	2470901	2209312	2350565	2133481
250ppm	250.000	258.931	8.931	3817791	14.2 %	3015693	3833419	3878207	4543845
500ppm	560.000	567.352	7.352	7790208	13.3 %	6848503	9192550	6740369	8379410
1000ppm	900.000	885.003	-14.997	11645181	8.8 %	10618610	19945740	18284421	12671752

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 7 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 Silicon Dioxide							
Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.020	0-25	K	766.491	0.95	0-40
Al	308.216	2.5	0-60	Li	670.784	0.21	0-115
As	193.759	4.5	0-25	Mg	277.983	0.45	0-45
B	249.773	1.0	0-115	Mn	280.106	0.17	0-60
Ba	455.404	0.15	0-50	Mo	313.259	0.50	0-60
Be	313.107	0.011	0-115	Na	588.995	0.15	0-50
Bi	306.772	0.14	0-60	Nb	309.418	0.74	0-105
Ca	317.933	1.4	0-45	Ni	305.082	1.2	0-60
Cd	326.106	0.59	0-25	Pb	283.307	0.64	0-25
Co	345.351	5.2	0-60	Sb	217.589	1.4	0-25
Cr	283.563	0.40	0-60	Sn	317.502	0.31	0-45
Cu	324.754	0.059	0-45	Sr	407.771	1.2	0-60
Fe	259.940	1.0	0-60	Ti	334.941	1.7	0-60
Ga	294.364	0.18	0-45	V	310.230	0.082	0-60
Ge	303.906	0.15	0-35	Zn	213.856	0.46	0-30
In	325.609	0.074	0-30	Zr	339.198	1.7	0-100

Conclusions

The analysis of silicon dioxide 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 silicon dioxide matrix.

The oxide powder contained Co and W impurities that degraded the detection limits for those elements. The contamination for W was significant enough that a detection limit could not be calculated.