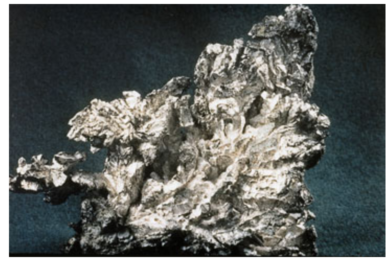
Analysis of Trace Elements in High-Purity Silver using the Prodigy DC Arc Spectrometer

Introduction

Silver is a white, relatively soft metal that has the highest electrical and thermal conductivity of all metals in the periodic table. Its electrical and thermal conductivity properties, along with its monetary value and lustrous appearance, make silver suitable for use in many applications, both in its pure metallic form and as a compound in alloyed metals. As a pure metal, silver is used to produce currency, jewelry, sterling silver silverware, electrical contacts, and industrial catalysts. Layers of silver can be sputtered onto glass surfaces to produce optics that allow varying amounts of light penetration.



As an alloyed metal, silver can be combined with metals such as tin and mercury to produce amalgams used in dentistry applications. Silver alloys are also used in speaker wires, RF connectors, printed circuits, RFID antennas, high-voltage contacts and electrical contacts inside computer keyboards.

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

Experimental

Operating Parameters

Standards were in the form of silver beads and were analyzed in their native form without the addition of graphite or a powdered internal standard. Standards were carefully weighed such that 100 mg of material was transferred into each sample electrode.

All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** in atmosphere without the use of a Stallwood Jet. All elements were integrated using individual time gates and the remaining instrument and method conditions used are listed in Table I.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignition at 13A, hold for 60 s, jump to 16A, hold for 65 s
Analytical Gap	4 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	3/16" diameter with an undercut cup (ASTM #S-15)
Sample	
Sample Size	100 mg
Internal Standard	None
Integration Time	Individual time gates were used

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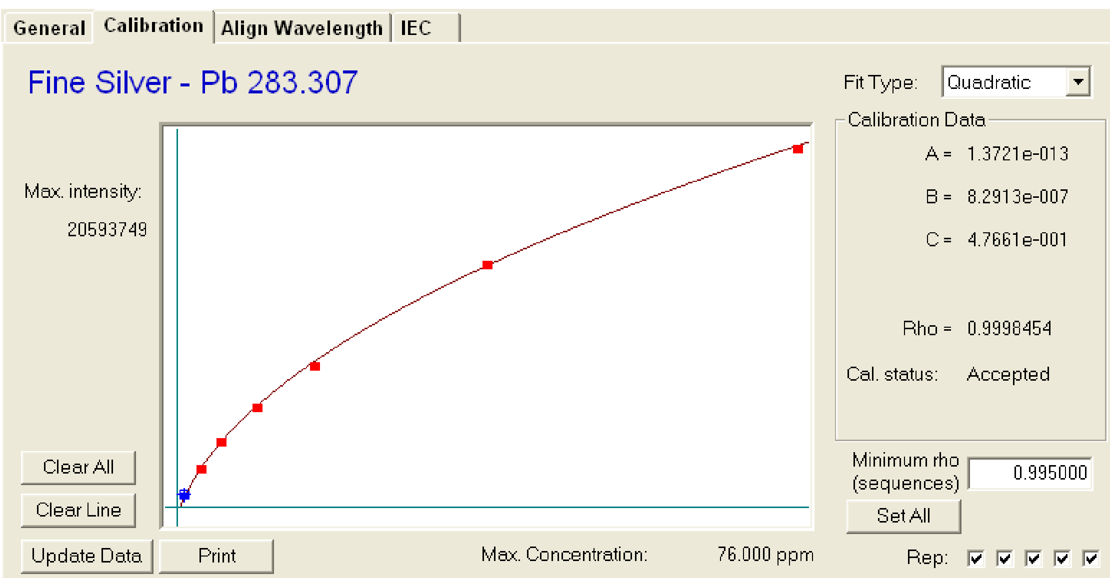
The sample and counter electrodes were purchased from Bay Carbon Inc. (Bay City, MI) and used as received. The sample electrodes used were 3/16" diameter electrodes with undercut cups (ASTM # S-15). The counter electrodes used for all analyses were 1/8" in diameter and pointed (ASTM # C-1). A 4 mm analytical 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 two sets of high-purity silver standards. The first set of standards (Ag-1, Ag-2, Ag-3) contained the analytes of interest at concentrations that ranged from 0.01 to 213 ppm and was used to calibrate the instrument for purposes of calculating detection limits. The second set of standards (Blank, 1, 2, 3, 4, 5 and 6) contained the analytes of interest at concentrations that ranged from 1 to 1050 ppm and was used to determine the instrument's accuracy and working dynamic range in this matrix. All standards were analyzed as received and weighed directly into sample electrodes as described above.

An example of a typical calibration curve for elements measured in high-purity silver is illustrated in Figure 1 for Pb at 283.307 nm. The calibration curve demonstrates typical precision and accuracy for the concentration range over which the instrument was calibrated.

Figure 1 Calibration Curve of Pb at 283.307 nm in High-Purity Silver



Std ...	Conc.	Calc.	Dev.	Mean	SD or ...	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5
Blank	1.000	1.211	0.211	783779	6.7 %	688147	802673	773707	842372	811996
1	3.000	2.976	-0.024	2208000	6.6 %	1942926	2358755	2317916	2188377	2232027
2	5.500	5.633	0.133	3813048	2.4 %	3948464	3755742	3900086	3711196	3749754
3	10.000	9.837	-0.163	5773354	5.2 %	6114091	5897211	5508827	6005995	5340650
4	17.000	16.276	-0.724	8126565	9.8 %	7562656	9267569	8044709	7035448	8722444
5	38.000	38.827	0.827	13967898	6.6 %	14671949	14420772	14077550	14515045	12154176
6	76.000	75.741	-0.259	20593749	3.5 %	16295403	17207007	18297847	21304823	19882676

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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 Ag-1 standard which contained all elements of interest at approximately 0.01 ppm. 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 Silver

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Au	267.595	0.01	90-125
Bi	306.772	0.003	0-60
Cu	324.754	0.01	0-120
Fe	302.064	0.2	0-125
Ni	305.082	0.006	90-125
Pb	283.307	0.007	0-45
Pd	340.458	0.002	90-125
Pt	265.945	0.02	90-125
Sb	217.589	0.2	0-125
Se	203.985	0.3	0-45
Sn	283.999	0.008	0-45
Te	214.275	0.2	0-125
Tl	535.046	0.3	0-25
Zn	481.053	0.02	0-20

Conclusions

The analysis of silver metal using the **Prodigy DC Arc** Spectrometer 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 silver metal matrix. The instrument's superior working dynamic range allows elements to be measured at concentrations that span multiple orders of magnitude without the need for line switching using multiple calibration curves.

It should be noted that the detection limits reported in this application note, reflect not only the precision and sensitivity of the instrument, but the homogeneity and the size uniformity of the standards used in the detection limit study. Standards that are less homogenous and uniform in size will cause the detection limits to deteriorate. The degree of deterioration will depend upon the variation in material size and composition.