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

Introduction

High-purity nickel is predominantly used in the production of metal alloys. It is also used in the production of many domestic and worldwide consumer products including rechargeable batteries, magnets, catalysts and coins, including the United States five cent piece. Nickel, in its powdered form, is mixed with iron or copper metal powders to increase the density of auto parts such as clutch plate holders, rotors and gears.

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 nickel.

Experimental

Operating Parameters

Standards were in the form of powdered metal and were analyzed in their native form without the addition of graphite or a powdered internal standard. Standards were carefully weighed such that 15 mg of metal powder was transferred into each appropriate sample electrode. All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc**. The instrument was operated using the instrument and method parameters listed in Table I. Standards were burned in atmosphere and all elements were integrated using individual time gates.

Table I DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignition at 6A, hold for 15 s, jump to 15A, hold for 85 s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	3/16" diameter with 4 mm x 3 mm undercut cup (ASTM #S-15)
Sample	
Sample Size	Hand packed, ~15 mg
Internal Standard	None
Integration Time	Individual time gates used

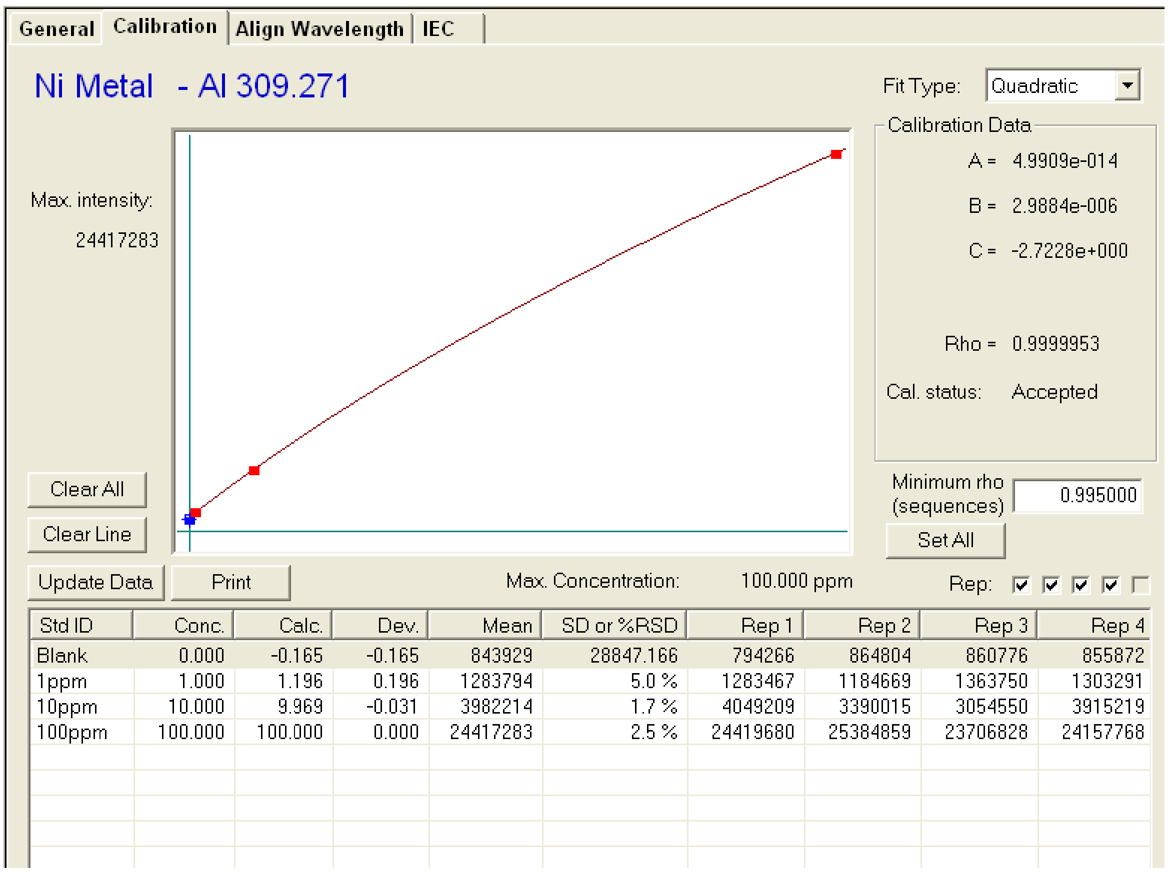
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with a 4 mm x 3 mm undercut cup (part # S-15). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm 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 nickel powdered metal standards that contained the analytes of interest at 0, 1.0, 10 and 100 ppm. Calibration standards were prepared in this matrix by serial dilution on a weight-to-weight basis from a multielement stock standard containing 45 elements at 1.21% (MV Laboratories, Inc., Frenchtown, NJ). All standards were carefully weighed and thoroughly blended in a mixer/mill for a minimum of 5 minutes before weighing them into sample electrodes as described above.

An example calibration curve for elements measured in high-purity nickel is illustrated in Figure 1 for Al at 309.271 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 309.271 nm in High-Purity Nickel Metal



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
Blank	0.000	-0.165	-0.165	843929	28847.166	794266	864804	860776	855872
1ppm	1.000	1.196	0.196	1283794	5.0 %	1283467	1184669	1363750	1303291
10ppm	10.000	9.969	-0.031	3982214	1.7 %	4049209	3390015	3054550	3915219
100ppm	100.000	100.000	0.000	24417283	2.5 %	24419680	25384859	23706828	24157768

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, along with the wavelengths and integration times used for the data collection. All results are listed in units of parts per million (ppm).

Table II Detection Limits in High-Purity Nickel

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.12	0-50	Li	670.784	0.50	0-50
Al	309.271	0.48	0-90	Mg	279.553	0.37	0-50
As	193.759	3.2	0-100	Mn	257.610	0.095	0-50
B	249.678	0.49	0-30	Mo	317.035	0.56	0-90
Ba	493.409	0.45	0-30	Na	588.995	0.97	0-30
Be	234.861	0.18	0-30	P	253.565	1.1	0-70
Bi	306.772	0.28	0-35	Pb	283.307	0.31	0-40
Ca	393.366	0.55	0-40	Sb	217.589	1.7	0-80
Cd	214.438	0.32	0-20	Se	203.985	4.6	0-100
Co	238.892	2.6	0-90	Si	251.612	0.78	0-100
Cr	283.563	0.58	0-90	Sn	283.999	0.24	0-80
Cu	327.396	0.055	0-60	Sr	407.771	3.5	0-100
Fe	259.940	0.44	0-90	Te	214.275	1.0	0-35
Ga	287.424	0.21	0-60	Ti	334.941	0.49	0-90
Ge	270.963	0.59	0-90	V	318.540	0.44	0-90
In	325.609	1.7	0-60	Zn	334.502	1.6	0-20
K	766.491	2.4	0-100	Zr	339.198	3.8	0-100

Conclusions

The analysis of nickel metal 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 nickel matrix.