Analysis of Trace Elements in Nickel Oxide Using the Prodigy DC Arc Spectrometer

Introduction

Nickel(II) oxide is the chemical compound with the formula NiO. It is notable as being the only well-characterized oxide form of nickel. The mineralogical form of NiO, bunsenite, is very rare and is classified as a basic metal oxide. Several million kilograms are produced annually and used predominantly as an intermediate in the production of nickel alloys.

Nickel(II) oxide is used in the ceramic industry to make frits, ferrites, and porcelain glazes. It is also used as a component in fuel cells and in the nickel-iron battery, known as the Edison Battery. It is the precursor to many nickel salts used as specialty chemicals and catalysts. More recently, NiO was used to make the NiCd rechargeable batteries found in many electronic devices, until it was replaced by the environmentally superior Lithium Ion battery. The sintered oxide is used to produce nickel steel alloys.

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 NiO.

Experimental

Operating Parameters

A series of standards was prepared for analysis by using high-purity NiO, graphite, a carbonate and fluoride-containing buffer and a 45 element stock standard obtained from MV Laboratories. The buffer consisted of a 6:3:1 mixture of graphite to $BaCO_3$ to BaF_2 by weight. Each standard was mixed with this buffer at a ratio of 3:2 by weight. All mixtures were thoroughly blended with a SPEX mixer/mill for a minimum of 10 minutes before hand packing into electrodes.

All analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** in atmosphere 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	Ignition at 12A, hold for 30 s, ramp from 12-16A over 135 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 an undercut cup (ASTM #S-14)
Sample	
Sample Size	Hand packed, ~50 mg
Internal Standard	None
Integration Time	Individual time gates used

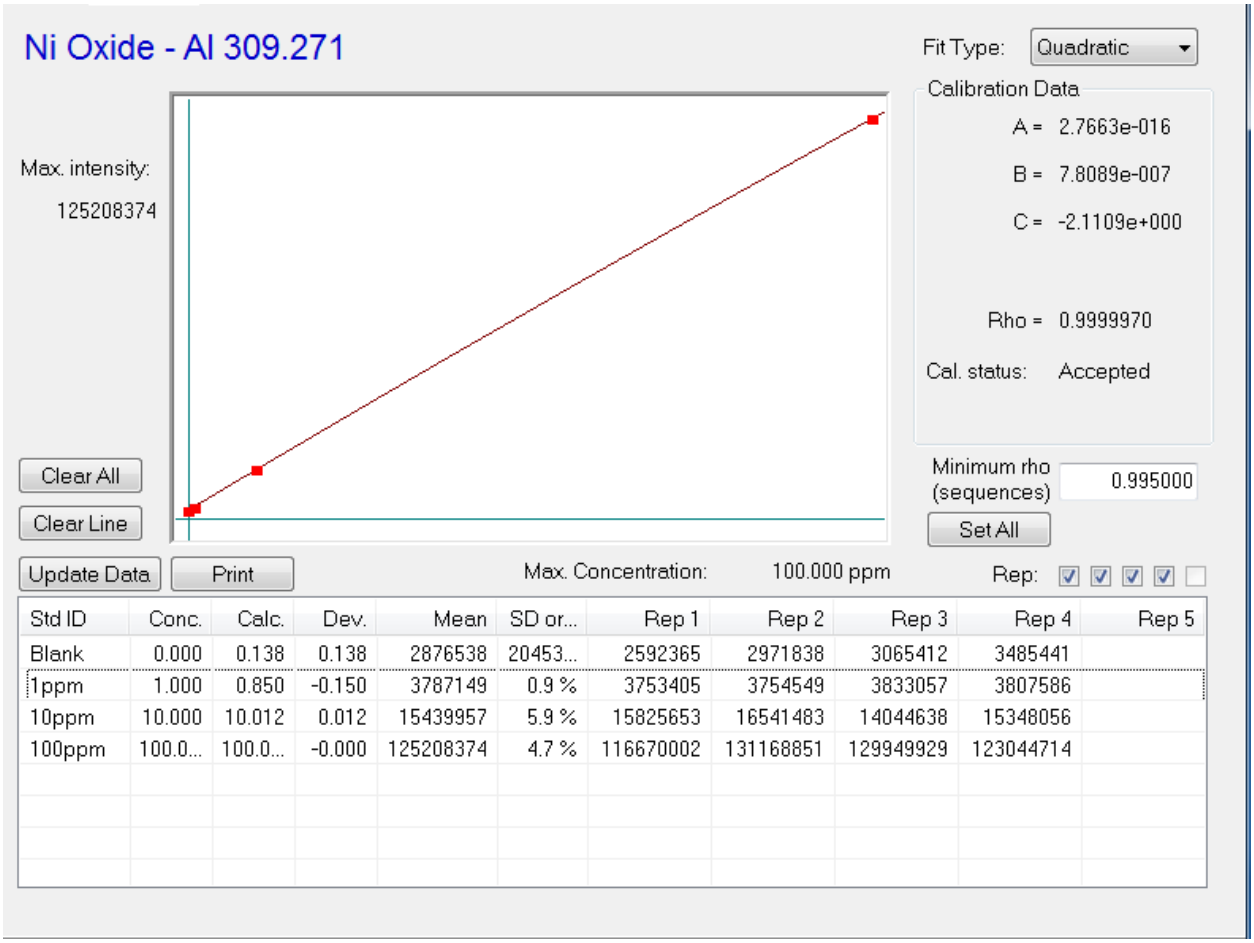
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # S-14). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm analytical 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 NiO 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 from 0 to 100 ppm in the NiO matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in NiO 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 Oxide



Std ID	Conc.	Calc.	Dev.	Mean	SD or...	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5
Blank	0.000	0.138	0.138	2876538	20453...	2592365	2971838	3065412	3485441	
1ppm	1.000	0.850	-0.150	3787149	0.9 %	3753405	3754549	3833057	3807586	
10ppm	10.000	10.012	0.012	15439957	5.9 %	15825653	16541483	14044638	15348056	
100ppm	100.0...	100.0...	-0.000	125208374	4.7 %	116670002	131168851	129949929	123044714	

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 Nickel Oxide

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.12	0-115	Mg	279.553	0.30*	0-135
Al	309.271	0.72*	0-155	Mn	260.569	0.14	0-150
As	193.759	2.55	0-150	Mo	317.035	0.50	0-150
B	249.678	0.38	0-155	Na	589.592	2.12*	0-75
Be	234.861	0.25	0-155	Nb	316.340	2.89	90-165
Bi	306.772	0.12	0-90	Pb	283.307	0.18	0-90
Ca	393.366	0.32*	0-145	Sb	217.589	0.48	0-155
Cd	326.106	0.99	0-25	Se	203.985	2.22	0-140
Co	346.580	2.71	0-155	Si	251.612	1.32*	90-160
Cr	427.480	0.49	0-155	Sn	283.999	0.063	0-155
Cu	324.754	1.00*	0-145	Sr	407.771	0.43	0-145
Fe	259.940	2.20*	0-155	Te	214.275	0.68	0-145
Ga	417.206	0.18	0-150	Ti	334.941	0.23	0-155
Ge	270.963	0.42	0-155	V	318.540	0.11	0-155
In	325.609	0.92	0-145	Zn	334.502	1.91	0-55
K	766.491	0.48	0-40	Zr	339.198	2.41	90-160
Ag	328.068	0.12	0-115	Mg	279.553	0.30*	0-135

*Contaminants present in blank; actual DLs should be lower than stated

Conclusions

The analysis of high-purity nickel oxide 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 NiO matrix.

Sales/Support: 800-634-9942 • **Main**: 603-886-8400
110 Lowell Rd, Hudson, NH 03051
www.teledyneleemanlabs.com