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

Introduction

Zinc oxide is an inorganic compound with the formula ZnO. It is a white powder that is insoluble in water and is widely used as an additive in numerous materials and products such as: plastics, ceramics, glass, cement, lubricants, paints, adhesives, sealants, pigments, foods, batteries, ferrites, fire retardants, ointments and first aid tapes. Zinc oxide occurs naturally as the mineral zincite, but a majority of zinc oxide is produced synthetically.

The primary materials science application for ZnO is as a wide bandgap semiconductor of the II-VI semiconductor group. As a semiconductor, ZnO has several favorable properties, including good transparency, high electron mobility, wide bandgap and strong room temperature luminescence. These properties are proving useful in emerging applications for transparent electrodes in liquid crystal displays, and in energy-saving or heat-protecting windows. Zinc oxide is also used in electronics and for the manufacture of thin-film transistors and light-emitting diodes.

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 ZnO.

Experimental

Operating Parameters

A series of standards from 0 to 100 ppm was prepared for analysis by using high-purity ZnO, graphite and a 45 element stock standard obtained from MV Laboratories. Each standard was mixed with graphite at a ratio of 1:1 by weight. Each mixture was 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	Ignite at 14A, hold at 14A for 30s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (# C-1)
Sample Electrode	1/4" diameter with an undercut cup (# S-4)
Sample	
Sample Size	Hand packed, ~50 mg
Internal Standard	None
Integration Time	0-30 s for all wavelengths

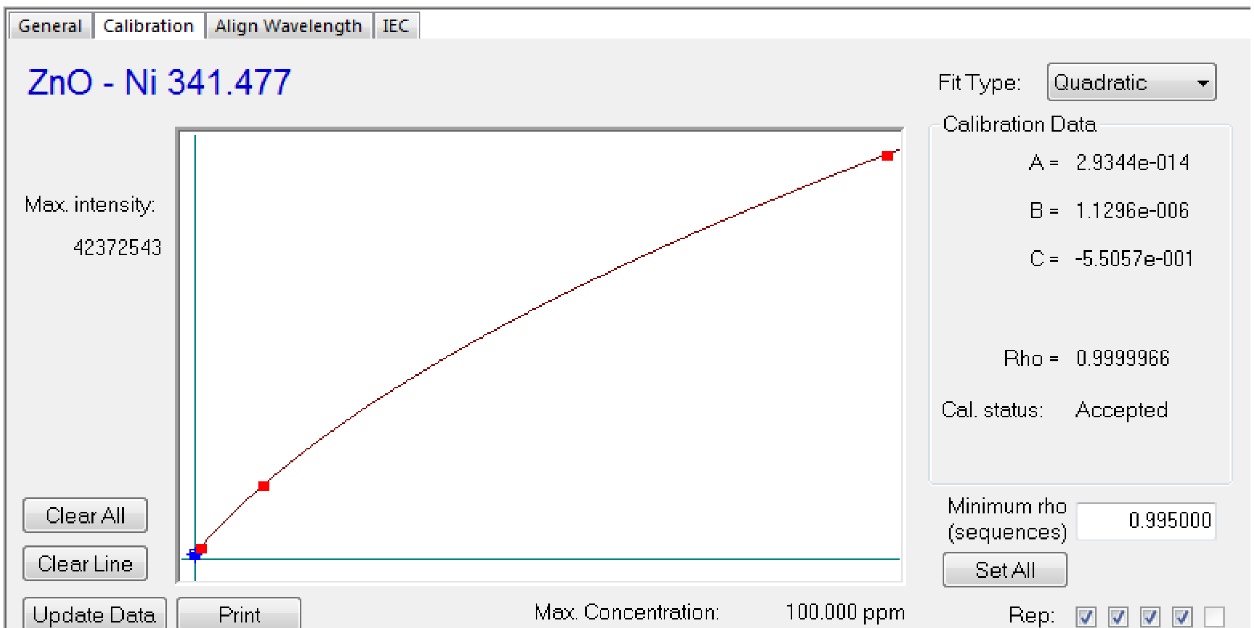
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 1/4" in diameter with an undercut cup (part # S-4). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm analytical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

An example calibration curve for elements measured in ZnO is illustrated in Figure 1 for Ni at 341.477 nm. The calibration curve for Ni demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve of Ni at 341.477 nm in High-Purity Zinc Oxide



Std ID	Conc.	Calc.	Dev.	Mean	SD or...	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5
Blank	0.000	0.147	0.147	608120	13069...	818049	580692	458344	575397	
1ppm	1.000	0.837	-0.163	1191860	9.7 %	1353701	1095595	1126284	1628717	
10ppm	10.000	10.016	0.016	7781087	7.1 %	8322891	8231214	7626937	6943307	
100ppm	100.0...	100.0...	-0.000	42372543	1.6 %	43436832	42022370	42368047	41662926	

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 Zinc Oxide

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	338.289	0.02	0-30	K	766.491	0.16	0-30
Al	396.153	0.21	0-30	Li	670.784	0.03	0-30
As	193.759	1.19	0-30	Mg	279.553	0.16*	0-30
B	249.678	0.03	0-30	Mn	279.827	0.12	0-30
Ba	455.404	0.11	0-30	Na	588.995	0.20	0-30
Be	234.861	0.004	0-30	Ni	341.477	0.40	0-30
Bi	306.772	0.008	0-30	Pb	283.307	0.46*	0-30
Ca	393.366	0.23*	0-30	Sb	217.589	0.13	0-30
Cd	326.106	0.14	0-30	Se	203.985	0.77	0-30
Co	345.351	0.08	0-30	Sn	286.333	0.04	0-30
Cr	427.480	0.16	0-30	Sr	407.771	0.71	0-30
Cu	327.396	0.33*	0-30	Te	214.275	0.78	0-30
Fe	302.064	1.05*	0-30	Ti	336.121	0.35	0-30
Ga	294.364	0.02	0-30	V	318.540	0.09	0-30
Ge	303.906	0.05	0-30				

*Contaminants present in the blank material; actual detection limits should be lower than stated

Conclusions

The analysis of high-purity zinc 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 ZnO matrix.