

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

### Introduction

Niobium forms oxide compounds with oxidation states at +2 (niobium monoxide, NbO), +3 (niobium trioxide, Nb<sub>2</sub>O<sub>3</sub>), +4 (niobium dioxide, NbO<sub>2</sub>) and +5 (niobium pentoxide, Nb<sub>2</sub>O<sub>5</sub>). Niobium pentoxide is the most commonly occurring oxide and is the precursor material used in all niobium applications. Niobium pentoxide is a white solid that melts at 1512 °C, is relatively nonreactive and is completely insoluble in water. Niobium pentoxide is used in the production of capacitors, alloyed materials and optical glasses. This oxide is also used to produce lithium niobate which is used extensively in telecommunication applications (optical modulators and mobile telephones).



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 niobium oxide. It is recommended that the sample preparation and analysis procedures outlined in this note also be followed for the analysis of niobium metal, with the addition of an initial preparation step to convert niobium metal to its oxide form. The conversion to niobium oxide is recommended because the oxide form melts and boils more smoothly and reproducibly in an arc than does the metallic form.

### Experimental

#### Operating Parameters

All standards were prepared for analysis by mixing each standard with a carrier consisting of a 2:1 blend of high-purity graphite powder and silver chloride. The standards were combined with the carrier such that the ratio of the carrier to sample was 2:1 by weight. All mixtures were thoroughly blended in a mixer/mill for a minimum of 10 minutes before hand packing them into sample 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
<b>DC Arc Stand</b>	
Current	Ignition at 6A, hold for 2 s, jump to 10A, hold for 28 s
Stallwood Jet	None
Analytical Gap	3 mm
<b>Electrodes</b>	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	3/16" diameter with an undercut cup (ASTM #S-15)
<b>Sample</b>	
Sample Size	Hand packed
Internal Standard	None
Integration Time	0-30 s for all wavelengths

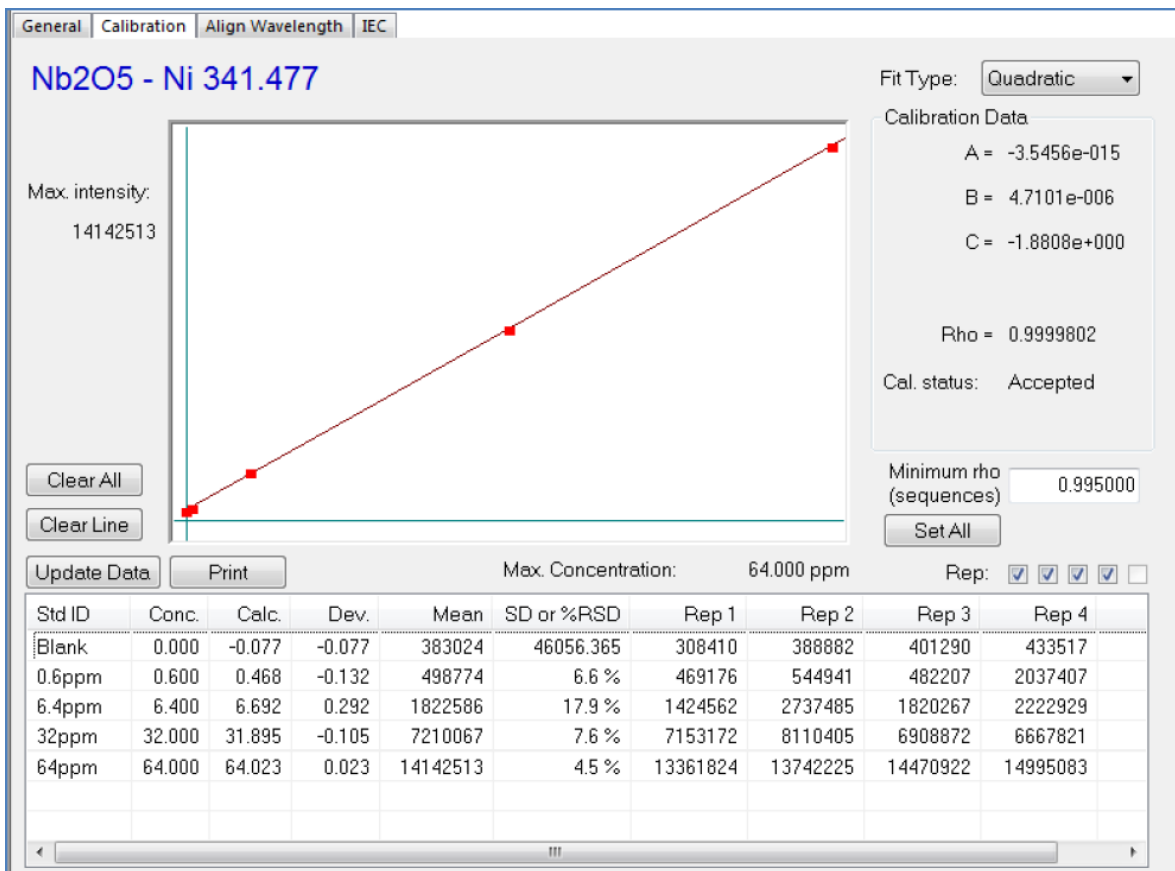
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # S-15). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 3 mm analytical gap was used and the position of the electrodes was adjusted during the sample burn to maintain a distance of 3 mm between the sample and the counter electrode.

### Calibration

The instrument was calibrated with several high-purity Nb<sub>2</sub>O<sub>5</sub> 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, 0.6, 6.4, 32 and 64 ppm in the Nb<sub>2</sub>O<sub>5</sub> matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in uranium oxide is illustrated in Figure 1 for B at 249.678 nm. The calibration curve for B 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 Niobium Oxide



## 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 Niobium Oxide							
Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Al	396.153	1.25	0-30	Li	670.784	0.63	0-30
As	193.759	5.70	0-30	Mg	279.553	0.49	0-30
B	249.773	0.44	0-30	Mn	279.482	0.73	0-30
Ba	493.409	2.01	0-30	Na	588.995	1.80	0-30
Be	234.861	0.07	0-30	Ni	341.477	1.28	0-30
Bi	223.061	1.28	0-30	Pb	283.307	0.46	0-30
Ca	393.366	3.40	0-30	Sb	206.833	2.64	0-30
Cd	228.802	1.20	0-30	Si	288.160	7.49	0-30
Co	345.351	0.83	0-30	Sn	283.999	1.55	0-30
Cr	425.435	0.15	0-30	Sr	460.733	1.10	0-30
Cu	327.396	2.40	0-30	Te	214.275	2.85	0-30
Fe	271.903	1.39	0-30	Ti	323.452	1.21	0-30
Ga	294.364	0.87	0-30	V	318.540	1.79	0-30
Ge	270.963	3.23	0-30	Zn	213.856	0.22	0-30
K	766.491	4.17	0-30	Zr	343.823	7.23	0-30

## Conclusions

The analysis of niobium 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 niobium oxide matrix.