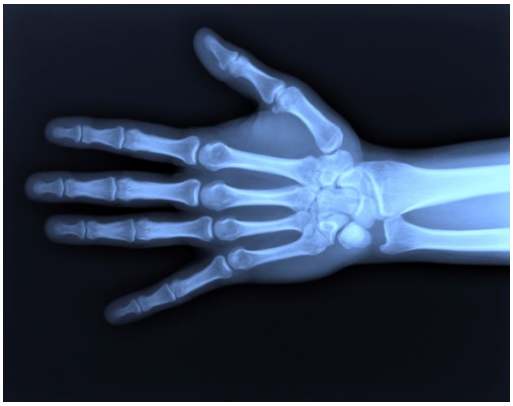
Analysis of Trace Elements in Tungsten Trioxide Using the Prodigy DC Arc Spectrometer

Introduction

Tungsten trioxide is used for many purposes in everyday life. It is frequently used in industry to manufacture tungstates for x-ray screen phosphors, for fireproofing fabrics and in gas sensors. Due to its rich yellow color, WO_3 is also used as a pigment in ceramics and paints.



In recent years, tungsten trioxide has been employed in the production of electrochromic windows, or smart windows. These windows are made of electrically switchable glass that change light transmission properties with an applied voltage. This allows the user to tint their windows, changing the amount of heat or light passing through.

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 tungsten oxide.

Experimental

Operating Parameters

All standards were prepared for analysis by mixing each WO_3 standard with a 1:5 $AgCl_2$/graphite buffer in a ratio of 1:1.5 by weight. Buffer material and all standards were shaken in a SPEX 8000M Mixer/Mill for a minimum of 10 minutes to ensure homogeneity.

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 8A, hold at 8A for 2 s, jump to 15A, hold for 33 s
Stallwood Jet	None
Analytical Gap	3 mm
Electrodes	
Counter Electrode	1/8" diameter and pointed (ASTM #C-1)
Sample Electrode	1/4" diameter and tapered (ASTM #1068-2)
Sample	
Sample Size	Hand packed, ~45 mg
Internal Standard	None
Integration Time	0-35 s for all wavelengths

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

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" diameter cup electrodes with tapered ends (part # 1068-2). 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.

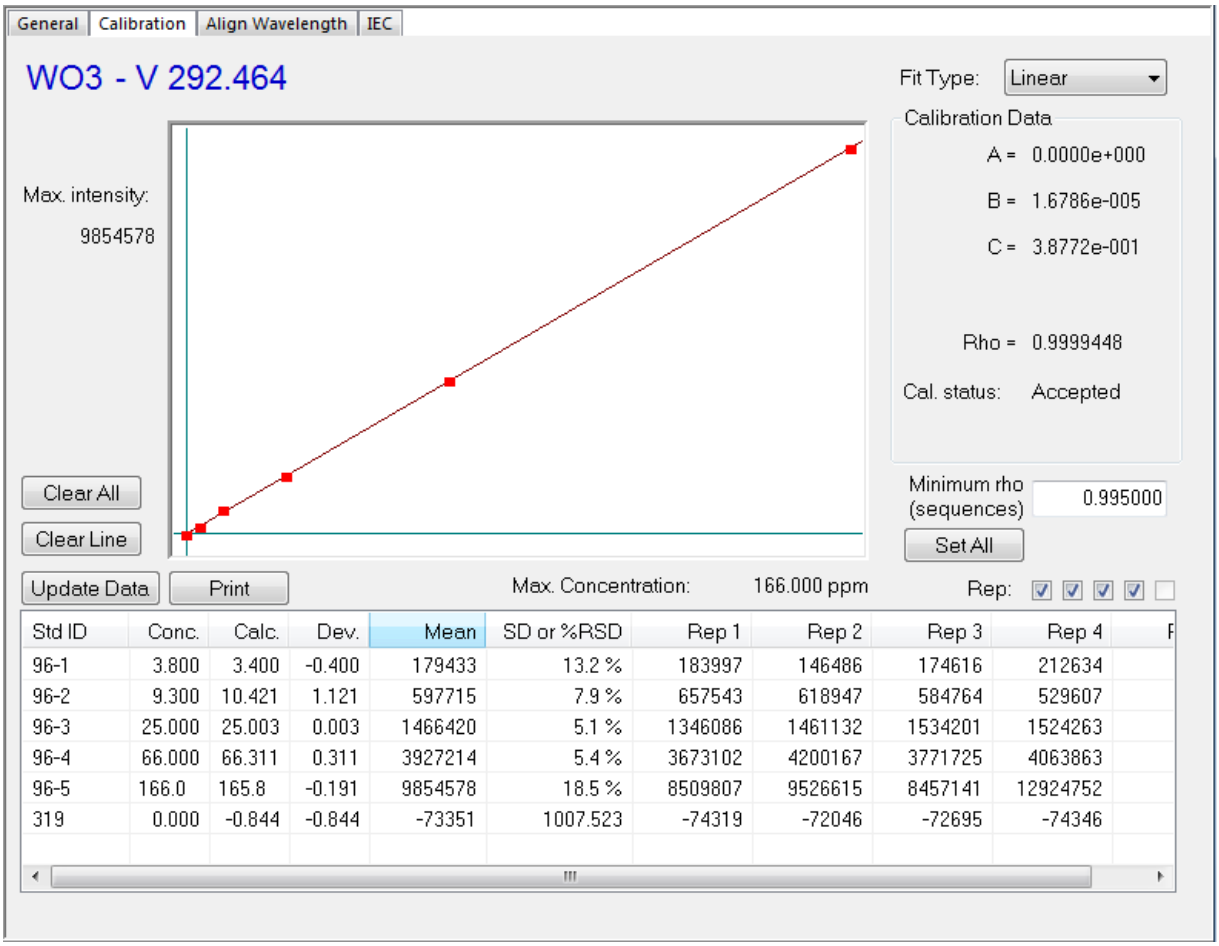
Calibration

The instrument was calibrated with several high-purity WO_3 standards that contained the analytes of interest at concentrations between 0 and 169 ppm. All standards were weighed, mixed and prepared for analysis as described above.

Two nearly pure standards were used for calibration and detection limit studies. Prior to use, each of the two nearly pure materials were measured as samples to determine which material was most appropriate for use as a calibration blank for each analyte.

An example calibration curve for elements measured in WO_3 is illustrated in Figure 1 for V at 292.464 nm. The calibration curve for V demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve of V at 292.464 nm in High-Purity Tungsten Oxide



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
96-1	3.800	3.400	-0.400	179433	13.2 %	183997	146486	174616	212634
96-2	9.300	10.421	1.121	597715	7.9 %	657543	618947	584764	529607
96-3	25.000	25.003	0.003	1466420	5.1 %	1346086	1461132	1534201	1524263
96-4	66.000	66.311	0.311	3927214	5.4 %	3673102	4200167	3771725	4063863
96-5	166.0	165.8	-0.191	9854578	18.5 %	8509807	9526615	8457141	12924752
319	0.000	-0.844	-0.844	-73351	1007.523	-74319	-72046	-72695	-74346

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 a 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 Tungsten Oxide

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Al	309.271	0.59	0-35	Mn	257.610	0.92	0-35
As	193.759	1.91	0-35	Mo	313.259	0.42	0-35
Bi	306.772	0.23	0-35	Ni	341.477	0.46	0-35
Ca	396.847	0.61	0-35	Pb	283.307	0.81	0-35
Cd	214.438	0.21	0-35	Sb	231.147	1.04	0-35
Co	240.725	0.086	0-35	Si	251.612	2.35*	0-35
Cr	284.325	0.24	0-35	Sn	317.502	0.35	0-35
Cu	324.754	1.65*	0-35	Ti	323.452	0.10	0-35
Fe	259.940	2.98*	0-35	V	292.464	0.17	0-35
Mg	279.553	0.62	0-35				

*Indicates contaminations; detection limits for these elements should be lower than stated

Conclusions

The analysis of WO_3 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 tungsten oxide matrix.

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