

## Analysis of Trace Elements in Titanium Dioxide Using the Prodigy DC Arc Spectrometer

### Introduction

Titanium (IV) oxide, also known as titanium dioxide (TiO<sub>2</sub>), is found as a white powder with a melting point of 1843 °C. It is the naturally occurring oxide form of titanium and exists in four polymorphic forms: rutile, anatase, brookite and akaogiite. When ground as a fine powder with uniform particle size, titanium oxide has the highest refractive index of any known mineral, which makes it useful in a wide range of applications.

As a component of sunscreen, TiO<sub>2</sub> readily absorbs ultraviolet light without discoloration which enhances the stability of sunscreen and improves its ability to physically block UV light and protect the skin. When used in the production of paper, plastics, coatings or paint pigments, TiO<sub>2</sub> creates finished products with opaque, bright white physical properties. As a component in cosmetic and skin care products, TiO<sub>2</sub> helps to create a final product that is thick and provides UV protection for the skin. Since TiO<sub>2</sub> is both water and oil dispersible, it can be used to manufacture a wide range of skin care products.



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 titanium dioxide (TiO<sub>2</sub>).

### Experimental

#### Operating Parameters

All standards were prepared for analysis by mixing each with high-purity graphite powder such that the ratio of sample to graphite was 2:1. All mixtures were thoroughly blended in a mixer/mill for a minimum of 5 minutes before transferring them into sample electrodes. Each sample was weighed and transferred such that 25 mg of material was loaded into each sample electrode.

Analyses were performed on the Teledyne Leeman Lab's **Prodigy DC Arc** with the use of the Stallwood Jet. The jet gas consisted of a 70:30 mixture of Ar:O<sub>2</sub> such that the total gas flow was 2.5 L/min. A summary of the instrument and method conditions used are listed in [Table I](#).

Table I DC Arc Operating Conditions	
Parameter	Setting
<b>DC Arc Stand</b>	
Current	Ignite at 6 A, hold at 6 A for 20 s, jump to 20 A, hold at 20 A for 30 s
Stallwood Jet	70:30 Ar:O <sub>2</sub> (1.75 LPM Ar/0.75 LPM O <sub>2</sub> )
Analytical Gap	4 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, ~15 mg
Internal Standard	None
Integration Time	Individual time gates were used

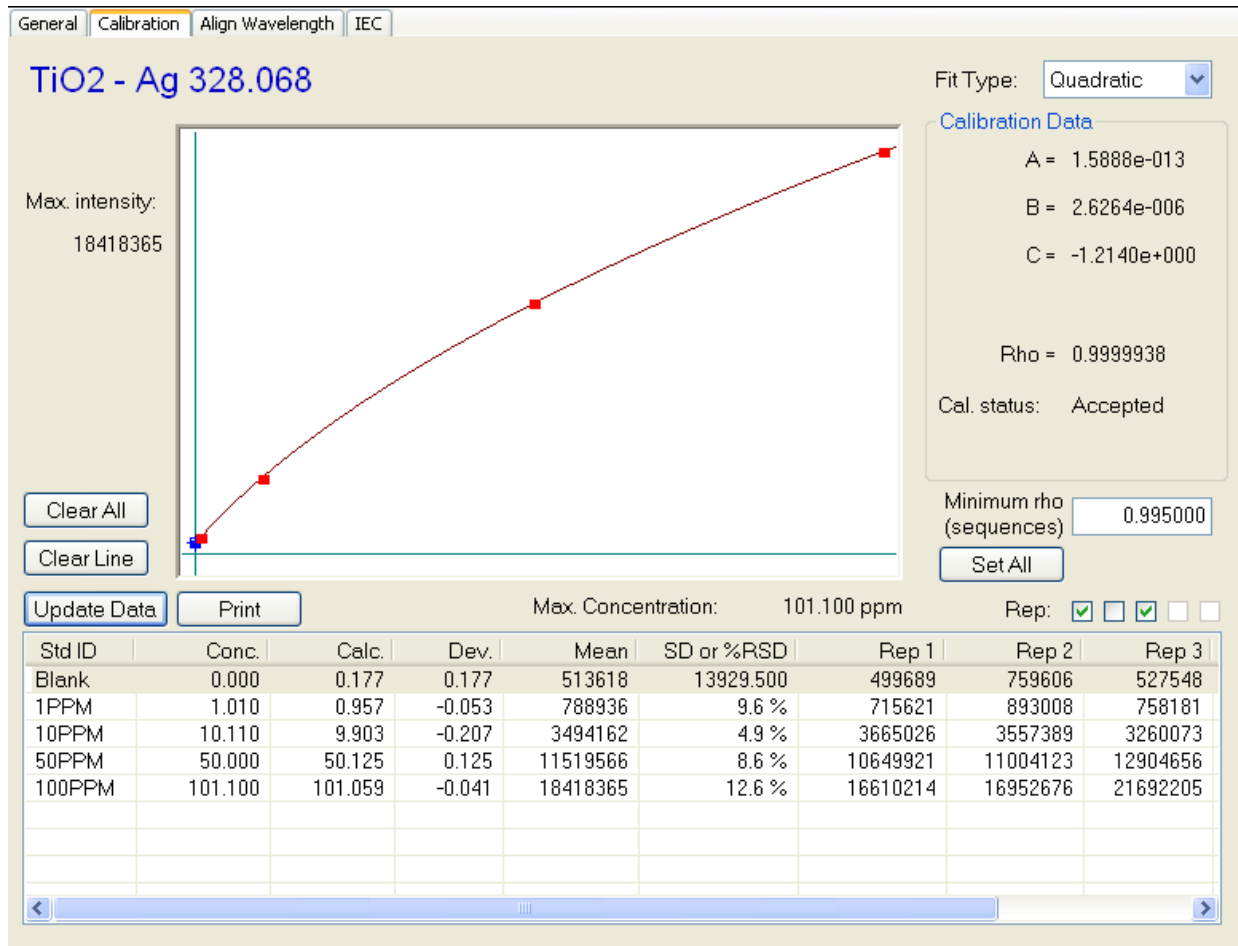
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with a 4 mm x 3 mm undercut cup (part # S-15). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm 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 TiO<sub>2</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, 1.0, 10, 50 and 100 ppm in the TiO<sub>2</sub> matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in TiO<sub>2</sub> is illustrated in Figure 1 for Ag at 328.068 nm. The calibration curve for Ag demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

**Figure 1** Calibration Curve for Ag at 328.068 nm in High-Purity Titanium Dioxide



## 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](#) in units of parts per million (ppm).

Table II Detection Limits in High-Purity Titanium Dioxide							
Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.48	0-25	K	766.490	1.7	0-25
Al	308.215	1.3	0-30	Li	670.784	0.20	0-30
B	249.773	0.75	0-45	Mg	279.553	0.57	0-25
Ba	493.409	4.1	0-25	Mn	280.106	0.37	0-25
Be	234.861	0.11	0-25	Mo	317.035	0.99	0-30
Bi	306.772	0.17	0-15	Na	589.592	0.40	0-30
Ca	422.673	1.2	0-25	Ni	305.082	2.3	0-25
Cd	226.502	0.77	0-15	Pb	283.306	0.57	0-25
Co	238.892	1.9	0-25	Sb	231.147	0.94	0-25
Cr	284.325	0.68	0-25	Sn	286.333	0.38	0-30
Cu	324.754	0.11	0-25	Sr	460.733	2.2	0-20
Fe	302.107	2.8	0-25	V	292.464	0.75	0-40
Ga	294.364	0.75	0-25	W	257.144	4.0	0-30
Ge	265.118	0.59	0-25	Zn	213.856	0.30	0-25

## Conclusions

The analysis of TiO<sub>2</sub> 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 TiO<sub>2</sub> matrix. The TiO<sub>2</sub> material used for this work contained a measurable Si impurity which prevented a detection limit from being calculated for that element.