

Analysis of Trace Elements in Titanium Dioxide with the Prodigy DC Arc

INTRODUCTION

Titanium (IV) oxide, also known as titanium dioxide (TiO_2), is 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_2 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_2 creates finished products with opaque, bright white physical properties. As a component in cosmetic and skin care products, TiO_2 helps to create a final product that is thick and provides UV protection for the skin. Since TiO_2 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 Labs **Prodigy DC Arc** to determine trace elements in high purity titanium dioxide (TiO_2).

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.



All analyses were performed on the Teledyne Leeman Labs **Prodigy DC Arc** with the use of the Stallwood Jet. The jet gas consisted of a 70:30 mixture of Ar:O₂ such that the total gas flow was 2.5 L/min. A summary of the instrument and method conditions used are listed in Table 1.

Table 1. DC Arc Operating Conditions

Parameter	Setting
DC Arc Stand	
Current	Ignite at 6 A, hold at 6 A for 20s, jump to 20 A, hold at 20 A for 30s
Stallwood Jet	Ar:O ₂ mixture at 70:30 ratio
Analytical Gap	4 mm
Electrodes	
Sample Electrode	3/16" in diameter with 4 mm x 3 mm undercut cup
Counter Electrode	1/8" diameter and pointed
Sample	
Sample Size	25 mg
Internal Standard	None
Integration Time	Variable by wavelength, shown in Table 2 below

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 Standards

The instrument was calibrated with several high-purity TiO₂ standards that were spiked with a multi-element 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 TiO₂ matrix. All standards were weighed, mixed and prepared for analysis as described above.

Calibration Curves

An example calibration curve for elements measured in TiO₂ 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.

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 2 in units of parts per million (ppm).

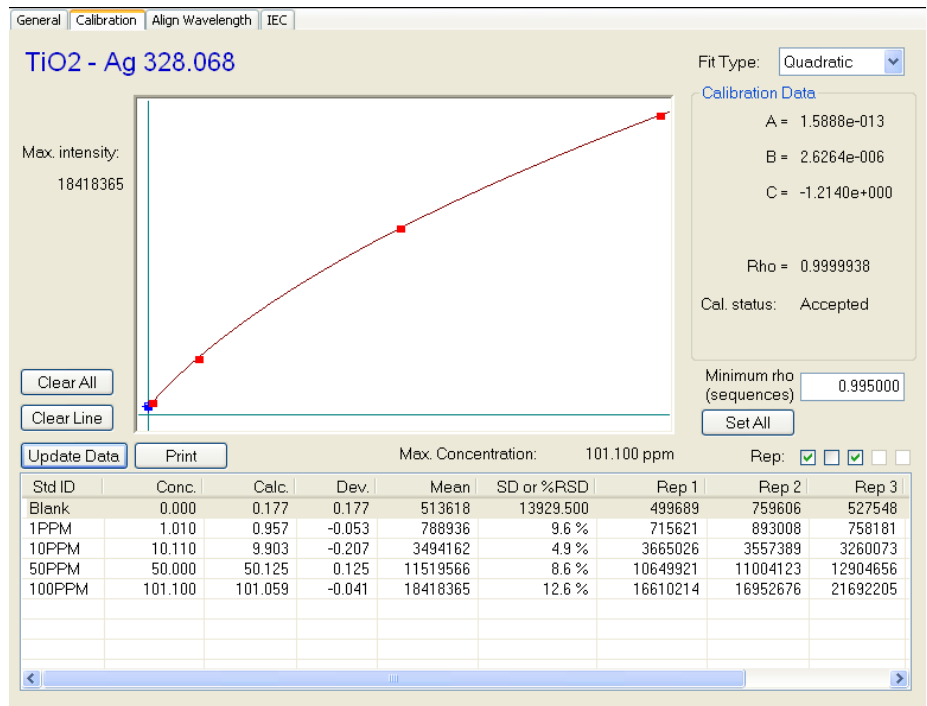


Figure 1. Calibration Curve of Ag at 328.068 nm in High Purity TiO₂

Table 2. Detection Limits in High Purity TiO₂

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 TiO₂ 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 which is reflected in the detection limits obtained for trace elements in a TiO₂ matrix. The TiO₂ material used for this work contained a measurable Si impurity which prevented a detection limit from being calculated for that element.