

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

## Introduction

Iron(III) oxide or ferric oxide is the inorganic compound with the formula  $\text{Fe}_2\text{O}_3$ . It is one of the three main oxides of iron; the other two being iron(II) oxide ( $\text{FeO}$ ) (which is rare), and iron(II, III) oxide ( $\text{Fe}_3\text{O}_4$ ). Iron (II, III) oxide occurs naturally as the mineral magnetite. Iron(III) oxide, also known as hematite, is a dark red, ferromagnetic mineral that is readily attacked by acids. Often referred to as rust, iron(III) oxide is the main source of iron for the steel industry.



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  $\text{Fe}_2\text{O}_3$ .

## Experimental

### Operating Parameters

A series of standards was prepared for analysis by using high-purity  $\text{Fe}_2\text{O}_3$ , graphite, a barium containing buffer and a 45 element stock standard obtained from MV Laboratories. The buffer used is a mix of 6:3:1 of graphite to  $\text{BaCO}_3$  to  $\text{BaF}_2$  by weight. Each standard was mixed with buffer at a ratio of 3:2 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
<b>DC Arc Stand</b>	
Current	Ignite at 12A, hold at 12A for 30 s; ramp from 12A to 16A over 135 s
Stallwood Jet	None
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 (# S-14)
<b>Sample</b>	
Sample Size	Hand packed, ~50 mg
Internal Standard	None
Integration Time	Individual time gates were used

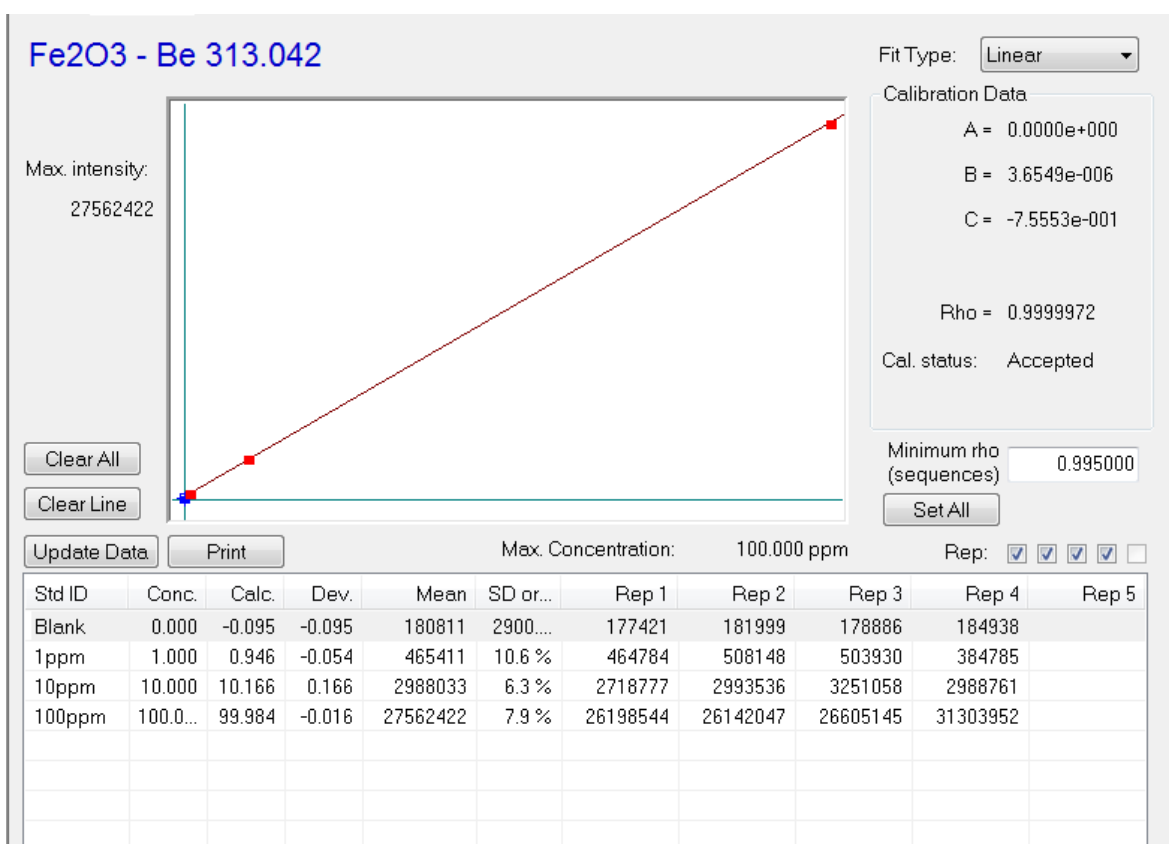
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # S-14). The counter electrodes used for all analyses were 1/8" in diameter and pointed (part # C-1). A 4 mm analytical 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 Fe<sub>2</sub>O<sub>3</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 from 0 to 100 ppm in the Fe<sub>2</sub>O<sub>3</sub> matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in Fe<sub>2</sub>O<sub>3</sub> is illustrated in Figure 1 for Be at 313.042 nm. The calibration curve for Be demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

**Figure 1** Calibration Curve of Be at 313.042 nm in Iron 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 Iron Oxide							
Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Al	308.216	0.12	0-130	Mn	279.827	0.17	0-165
As	193.759	1.82	0-140	Mo	281.615	0.78	60-165
B	208.959	3.40	0-130	Na	589.592	0.26*	0-60
Be	313.042	0.04	0-165	Nb	316.340	1.13	60-165
Bi	223.061	0.03	0-25	Ni	305.082	0.77	0-165
Ca	422.673	0.61*	0-65	Pb	283.307	0.12	0-25
Cd	228.802	0.01	0-25	Sb	231.147	1.68	0-75
Co	345.351	0.52	0-145	Se	203.985	7.99	0-130
Cr	425.435	0.51	0-165	Si	288.160	0.72	0-165
Cu	327.396	1.19*	0-90	Sn	283.999	0.69	0-165
Ga	294.364	5.08	0-85	Sr	407.771	0.24	0-75
Ge	270.963	1.81	0-100	Ti	336.121	0.45	60-165
K	766.491	1.70	0-50	Tl	535.046	0.23	0-25
Li	670.784	0.94	0-50	V	310.230	0.60	60-150
Mg	280.270	0.60*	0-165	Zr	349.621	9.22	0-165

\* Contaminants present in blank; actual DLs should be lower than stated

## Conclusions

The analysis of high-purity iron 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 Fe<sub>2</sub>O<sub>3</sub> matrix.

As indicated in [Table II](#), no detection limits were reported for Ag. The powdered metal used in this application note contained a measurable Ag impurity which degraded the detection limit that would have been obtained in a pure metal matrix. As an example of this degradation, the Ag detection limit obtained in cobalt oxide, using the same graphite, BaCO<sub>3</sub> and BaF<sub>2</sub> preparation described in this work, resulted in a detection limit of 0.15 ppm.