Analysis of Trace Elements in Aluminum Oxide Using the Teledyne Leeman Labs Prodigy DC Arc

Introduction

Aluminum oxide is an amphoteric oxide with the chemical formula Al_2O_3. It is commonly referred to as alumina (α-alumina), or corundum in its crystalline form, as well as many other names, reflecting its widespread occurrence in nature and industry. Its most significant use is in the production of aluminum metal; it is also used as an abrasive due to its hardness, and as a refractory material due to its high melting point. There is also a cubic y-alumina with important technical applications.

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 Al_2O_3.

Experimental

Operating Parameters

A series of standards was prepared for analysis by using high-purity Al_2O_3, graphite and MV Laboratories common element mix. Each standard was mixed with high-purity graphite at a ratio of 1:2 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
DC Arc Stand	
Current	Ignite at 6A, hold at 6A for 20s; jump to 15A and hold for 115s
Stallwood Jet	None
Analytical Gap	4 mm
Electrodes	
Counter Electrode	3/16" diameter and pointed (part #U-3951)
Sample Electrode	3/16" diameter with an undercut cup (part #L-4006)
Sample	
Sample Size	Hand packed, ~30 mg
Internal Standard	None
Integration Time	Individual time gates were used

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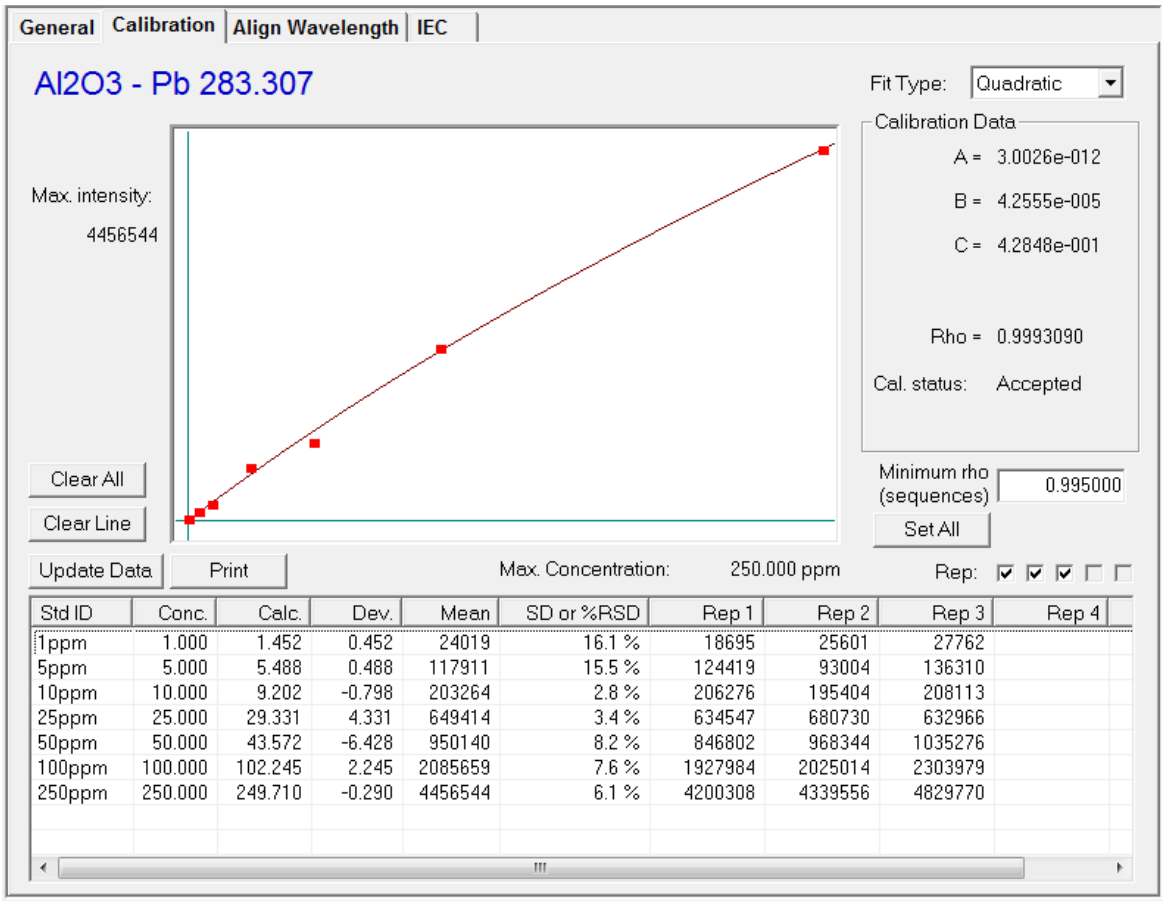
The sample and counter electrodes were purchased from Bay Carbon Inc (Bay City, MI) and used as received. The sample electrodes used were 3/16" in diameter with an undercut cup (part # L-4006). The counter electrodes used for all analyses were 3/16" in diameter and pointed (part # U-3951). A 4 mm analytical 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 Al_2O_3 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 1 to 250 ppm in the Al_2O_3 matrix. All standards were weighed, mixed and prepared for analysis as described above.

An example calibration curve for elements measured in Al_2O_3 is illustrated in Figure 1 for Pb at 283.307 nm. The calibration curve for Pb demonstrates typical precision and accuracy for the concentrations over which the instrument was calibrated.

Figure 1 Calibration Curve of Pb at 283.307 nm in High-Purity Al_2O_3



Std ID	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3	Rep 4
1ppm	1.000	1.452	0.452	24019	16.1 %	18695	25601	27762	
5ppm	5.000	5.488	0.488	117911	15.5 %	124419	93004	136310	
10ppm	10.000	9.202	-0.798	203264	2.8 %	206276	195404	208113	
25ppm	25.000	29.331	4.331	649414	3.4 %	634547	680730	632966	
50ppm	50.000	43.572	-6.428	950140	8.2 %	846802	968344	1035276	
100ppm	100.000	102.245	2.245	2085659	7.6 %	1927984	2025014	2303979	
250ppm	250.000	249.710	-0.290	4456544	6.1 %	4200308	4339556	4829770	

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 Al_2O_3

Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)	Element	Wavelength (nm)	Detection Limit (ppm)	Integration Time (s)
Ag	328.068	0.025	0-40	Li	670.784	0.21	0-90
As	189.042	12.31	0-40	Mg	280.270	1.65*	0-45
B	249.678	0.71	0-115	Mn	280.106	0.095	0-50
Ba	455.404	0.42	0-75	Mo	317.035	0.083	0-60
Be	234.861	0.029	0-60	Na	588.995	0.92*	0-60
Bi	306.772	0.043	0-30	Nb	322.548	4.43	0-60
Ca	393.366	1.44*	0-60	Ni	341.477	0.13	0-60
Cd	326.106	0.31	0-20	Pb	283.307	0.18	0-30
Co	345.351	0.34	0-65	Sb	259.806	1.03	0-45
Cr	425.435	0.38	0-50	Si	251.612	5.56*	0-70
Cu	324.754	1.57*	0-45	Sn	317.502	0.15	0-45
Fe	248.815	0.19	0-55	Ti	334.941	0.54	0-105
Ga	294.364	0.044	0-40	V	318.540	0.35	0-90
Ge	303.906	0.52	0-50	Zn	213.856	0.47	0-45
In	325.609	0.10	0-35	Zr	327.927	7.30	0-115
K	766.491	0.41	0-35				

Conclusions

The analysis of high-purity aluminum 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 an Al_2O_3 matrix.

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