

3<sup>rd</sup> March 2003

## **SPECTROLASER APPLICATION**

### **SODIUM ANALYSIS IN CALCINED ALUMINA**

#### **MATERIAL**

Powdered samples of high-purity Calcined Alumina were received from a Canadian site of a multi-national aluminium producer for analysis with the Spectrolaser 1000M. A subset of 8 samples with the same particle size and matrix (all with a client sample ID CAR-60RG) was chosen for these tests to determine the efficacy of the Spectrolaser in characterizing the sodium content in these materials.

#### **ANALYSIS METHOD**

Each of the samples was pressed, as received, in 40 mm sample cups using the LAT 40T hydraulic press to a pressure of 30 tonnes and for a dwell time 30secs. Each pellet was analysed 5 times using 100 laser pulses corresponding to a 20 second analysis time for each measurement.

#### **DETECTABLE ELEMENTS**

Detectable elements in the materials include the principal components Al, O, Na and Ca. Trace levels of Fe, Mg, Si and K are observable in some of the samples.

## *Calibration and Analysis Tests*

Two different calibration methods were used on the sample data – traditional single variate OES (optical emission spectroscopy) analysis utilising sodium elemental emission as well as partial least squares chemometric analysis. The results from both methods are reported for comparison.

Using the traditional OES analysis method the Spectrolaser software is used to select sodium emission lines which are then processed using peak integration - and in this case normalisation to the dominant Al component. The Spectrolaser software automatically constructs calibration curves of the normalised peak areas vs concentration of sodium in the alumina standards. The concentration of sodium in the unknown samples is then measured by the instrument by using this calibration reference.

Chemometric techniques use statistical methods to look for correlations between element concentrations and spectral features over the entire observed spectrum. The partial least squares method is used in this instance to establish the correlation of the measured spectra from the alumina standards to the given sodium concentration in each standard. The resulting correlation is then used by the instrument software to calculate the level of sodium in unknown samples subsequently analysed by the instrument.

## *Calibration Curves*

Calibration curves for the two analysis methods are shown in Figure 1 and 2. Both methods yield excellent and reproducible instrument calibration.

## **Reproducibility**

The two calibration methods were used to analyse the data obtained from five measurements of a known alumina standard. The comparison is shown in the table below.

	OES (589nm Na line, normalised to 396nm Al line)	Chemometric
1	0.43	0.41
2	0.39	0.40
3	0.43	0.41
4	0.40	0.39
5	0.43	0.41
Mean	<b>0.42</b>	<b>0.40</b>
SD	0.02	0.01
Reference Value	0.41	

*Table 1: Comparison of Na<sub>2</sub>O concentration in an alumina standard as measured by the Spectrolaser and analysed using OES and chemometric calibration techniques.*

Both methods yield acceptable rsd. In the OES case three measurements would normally be undertaken and the average reported - resulting in a measurement RSD < 5%.

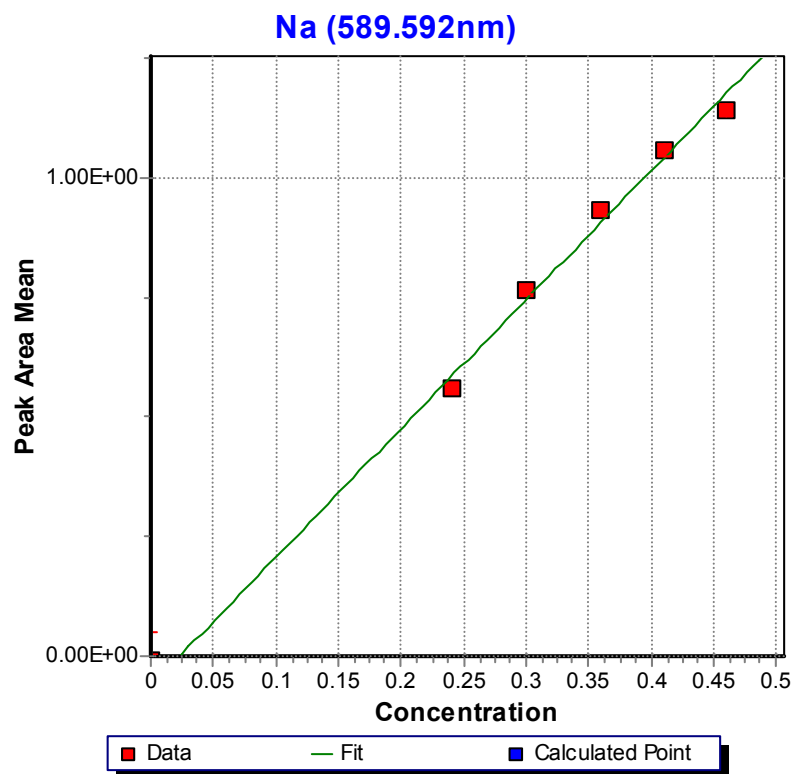


Figure1. Calibration curve for  $\text{Na}_2\text{O}$  in Calcined Alumina using the 589nm Na emission line.

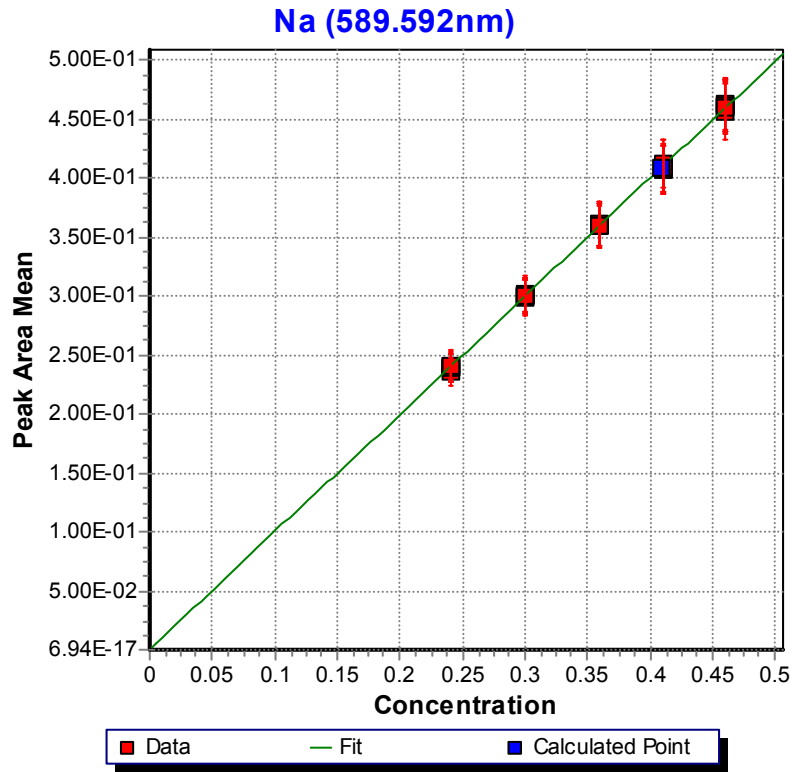


Figure 2: Calibration curve for  $\text{Na}_2\text{O}$  in Calcined Alumina using chemometric techniques

# Sample Spectra

