Laser Analysis Technologies Pty Ltd

SPECTROLASER APPLICATION

Analysis of Limestone

Limestone, the ubiquitous natural form of calcium carbonate, is used extensively worldwide as a feedstock in the glass, ceramic and cement industries. To ensure production quality in these industries the level of impurities in limestone and other like feedstock is closely monitored during the production process.

The Spectrolaser is a convenient tool to analyse feedstock materials such as limestone where broad elemental range and fast analysis aids the production process.

Sample Preparation

Typically, the Spectrolaser is used to analyse pressed powders which give a measurement precision of <5% RSD[1] for most matrices. This precision level is limited by the heterogeneous properties of naturally occurring materials and is similar to other direct analysis techniques such as XRF. Precision can further be enhanced to <1% RSD by the preparation of the samples in fused beads prior to analysis.

Detectable Elements

The Spectrolaser records the laser plasma emission spectrum from the deep UV (190 nm) to the IR (950 nm) using a patented multi-channel spectrometer system that captures the entire spectral range in a single laser pulse. This enables simultaneous determination of all detectable elements. A broad range of elements are detectable in limestone, depending on the geology of the particular source of the material. Detectable elements include - but are not confined to - Al, Ba, C, Ca, Cr, Fe, H, K, Mg, Mn, Na, O, Si, Sr and Ti.

[1] RSD : Relative Standard Deviation is defined as the standard deviation in a series of measurements divided by the mean vale of the measurement.

Calibration Curves

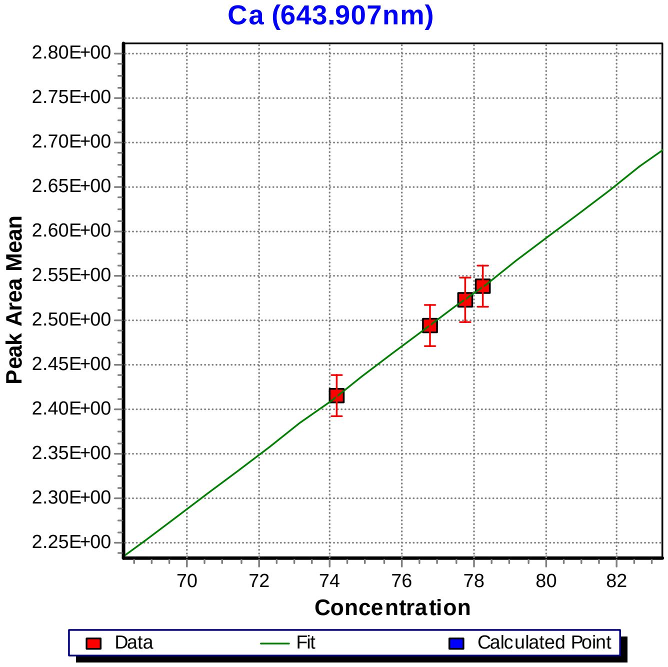
The *Spectrolaser* uses a technique known as Laser Induced Breakdown Spectroscopy (LIBS) to perform the elemental analysis of materials. Calibration involves the use of certified reference materials, the selection of an appropriate elemental optical emission line, and, in most cases, selection of a normalisation emission line. The *Spectrolaser* software automatically constructs calibration curves of the normalised peak area vs elemental concentration present in the reference materials. The concentration is determined in samples of unknown composition by the instrument using comparison with this calibration reference.

When there is a dominant matrix component for which the concentration will remain approximately constant across the calibration set, precision can be improved by normalizing against an emission line corresponding to the optical emission from that element. In the case of limestone calcium or carbon are possible candidates for matrix normalization. Alternatively, since the analysis is performed in air, atmospheric components, for example Nitrogen and Oxygen, will also be present in constant proportion and can be used in this role.

Example Calibration Curves

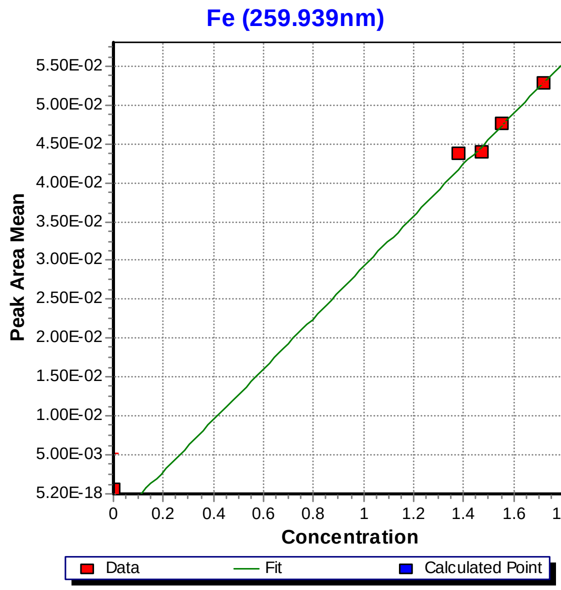
Principal Component

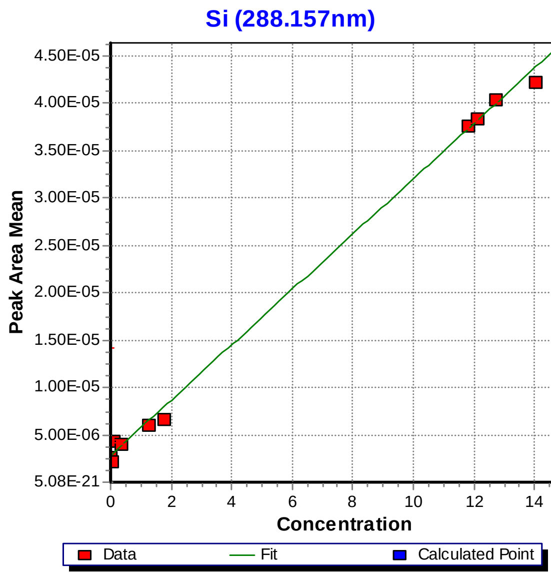
CaO 70-80%,

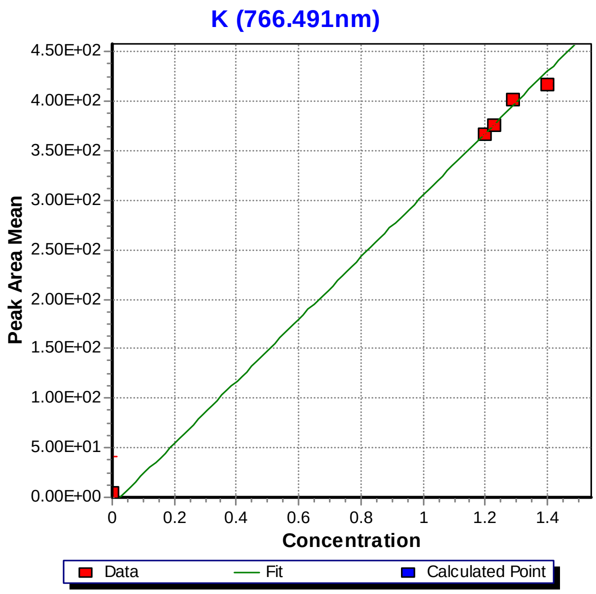


Example Calibration Curves of Minor Components

Fe, K, and Si







Reproducibility

A typical limestone analysis using pressed pellets

	MgO	Al_2O_3	SiO_2	SO_3	K_2O	Fe_2O_3
Measurement	293nm	309.27nm	263.1nm	PLS	766.491nm	259.939nm
1	1.4	4.6	12.3	0.22	1.27	1.47
2	1.3	4.7	11.8	0.26	1.19	1.47
3	1.6	4.5	12.0	0.26	1.20	1.45
4	1.5	5.0	12.2	0.26	1.19	1.46
5	1.4	4.2	11.9	0.21	1.24	1.42
6	1.5	4.7	12.0	0.22	1.22	1.47
7	1.6	4.5	12.2	0.22	1.25	1.44
8	1.4	4.0	11.9	0.21	1.26	1.41
Average (SD)	**1.5 (0.1)**	**4.5 (0.3)**	**12.0 (0.2)**	**0.23 (0.02)**	**1.23 (0.03)**	**1.45(0.02)**
Reference	**1.55**	**4.62**	**12.14**	**0.21**	**1.23**	**1.47**

Typical detection Limits – Calcium matrices

Element	Detection Limit*
Na	0.001 %
Ca	0.001 %
Mg	0.001 %
Fe	0.003 %
Al	0.003 %
K	0.002
Ti	0.002
Si	0.003%