ANALYSIS OF LIMESTONE USING THE TSI® LIBS DESKTOP ANALYZER

APPLICATION NOTE LIBS-010

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 TSI® LIBS Desktop Analyzer is a convenient tool to analyze feedstock materials such as limestone where broad elemental range and fast analysis aids the production process.

Sample Preparation

Typically, the TSI LIBS Desktop Analyzer is used to analyze 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.

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

Detectable Elements

The TSI LIBS Desktop Analyzer 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.

Calibration Curves

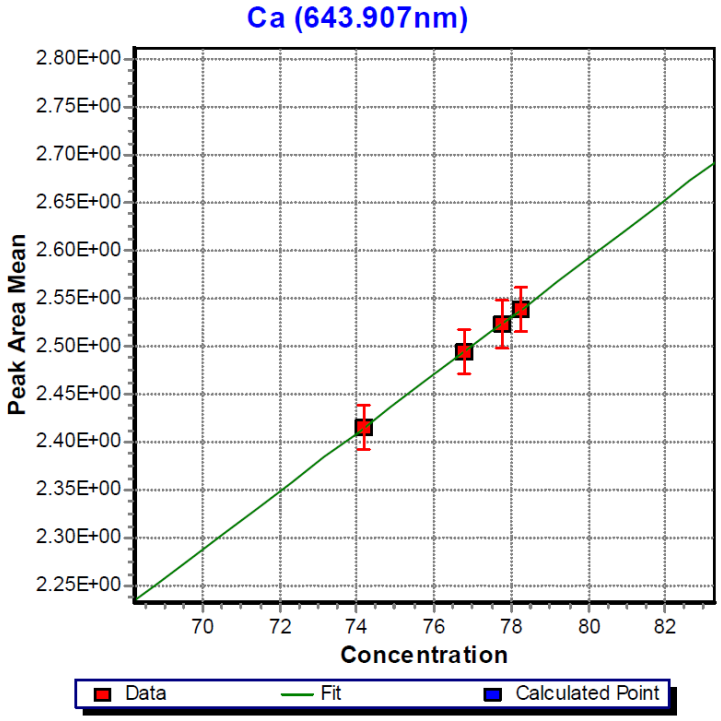
The TSI LIBS Desktop Analyzer uses a technique known as Laser Induced Breakdown Spectroscopy (LIBS) to perform the elemental analysis of materials. Calibration involves the use of certified reference materials, the selection of an appropriate elemental optical emission line, and, in most cases, selection of a normalization emission line. The TSI LIBS Desktop Analyzer software automatically constructs calibration curves of the normalized peak area vs. elemental concentration present in the reference materials. The concentration is determined in samples of unknown composition with the comparison against 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

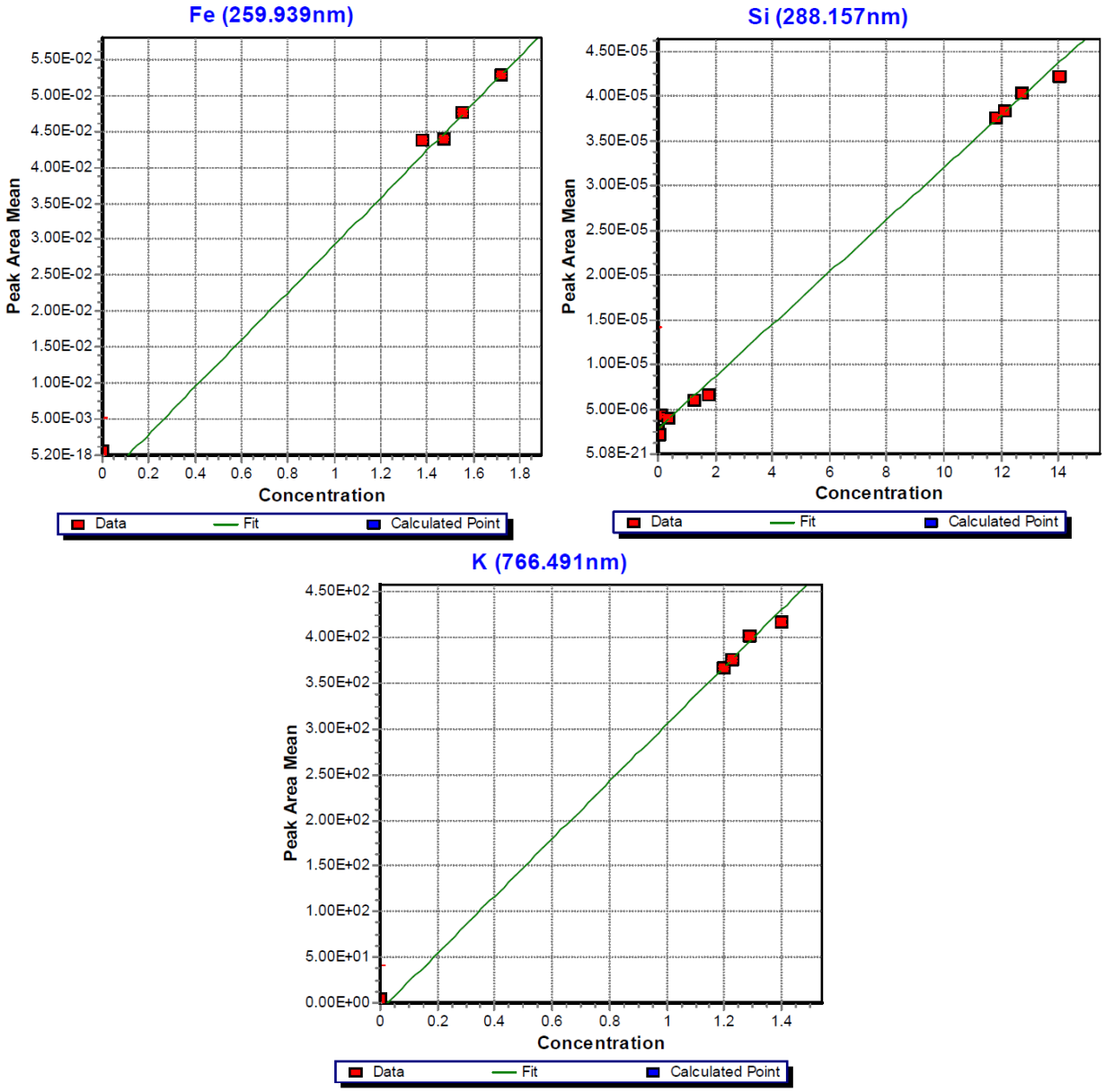
Principal Component

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 %

UNDERSTANDING, ACCELERATED

TSI Incorporated – Visit our website **www.tsi.com** for more information.

USA **Tel:** +1 800 874 2811
UK **Tel:** +44 149 4 459200
France **Tel:** +33 4 91 11 87 64
Germany **Tel:** +49 241 523030
India **Tel:** +91 80 67877200
China **Tel:** +86 10 8251 6588
Singapore **Tel:** +65 6595 6388

