

BORON ANALYSIS IN GLASS, ULEXITE, AND COLEMANITE USING THE TSI® LIBS DESKTOP ANALYZER

APPLICATION NOTE LIBS-001

Materials

Boron is an example of a light element (low atomic number) that is easily measured using Laser Induced Breakdown Spectroscopy (LIBS) but can be difficult to measure using alternative technologies.

Colemanite and ulexite are examples of boron containing ores used as feedstock in the glass industry. In these tests a multinational glass company sent a sample of colemanite to be analyzed with the TSI LIBS Desktop Analyzer while ulexite samples were obtained directly from a USA mine. For comparison, the repeat analysis of borosilicate glasses is also presented.

Analysis Method

The glass samples were ground to <60 microns with a ring mill prior to analysis. The powdered glass, colemanite and ulexite samples were pressed using the LAT 40T hydraulic press and analyzed using the TSI LIBS Desktop Analyzer using 50 laser pulses, corresponding to a 20 second analysis time in this case.

Detectable Elements

The Spectra exhibit strong Boron emission as well as emission from Group I and II elements is observed, notably Li, Na, K, Ca and Mg.

The principal components detected include:
Al, B, Ba, Ca, Fe, K, Li, Mg, N, Na, O, Si, Sr, and Ti.

The detection limit for B is estimated at 0.001% in these materials based on the observed signal-to-noise ratio.

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 by comparison with this calibration reference.

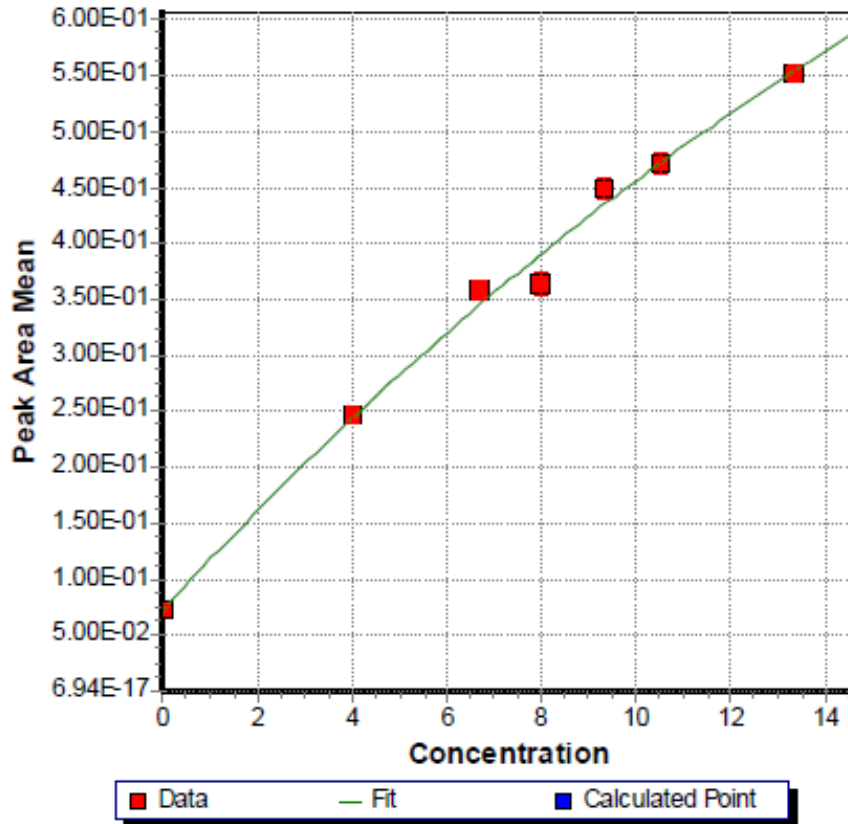


When there is a dominant matrix component for which the concentration will remain approximately constant across the calibration set, it is best to normalize against a line corresponding to the optical emission from that element. Oxygen is a possible candidate for this role in these samples and ratio to O has yielded the best results in this investigation.

An alternative analysis method is to use Chemometric correlation analysis. In this method the TSI LIBS Desktop Analyzer's software automatically constructs calibration curves using correlation of the entire observed spectrum with a material property (in this case the B concentration). Two correlation methods are available: Partial Least Squares or Principal Component Analysis.

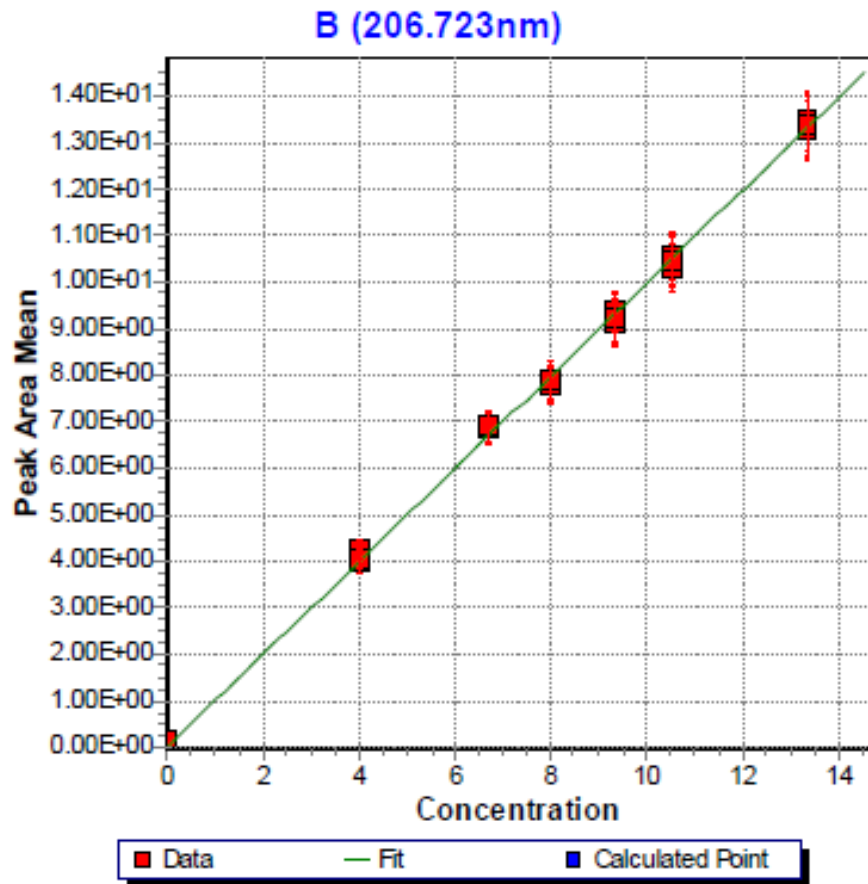
Boron Calibration

B (206.723nm)



Boron Calibration using the 206.723 nm emission line normalized to the O emission at 777nm, ulexite example.

Boron Calibration - Chemometric



Boron calibration using principal component regression (PCR)
Chemometric analysis, ulexite example.

Multiple Analysis Tests

1. Small bead glass samples. Nominal B_2O_3 concentration 5.5%

Analysis B	B_2O_3 (%)
1	5.4
2	5.6
3	5.5
4	5.5
5	5.4
6	5.6
Average	5.5
SD	0.1
Nominal Value	5.5

2. Large pieces of glass. Nominal B2O3 concentration 4.5%

Analysis B	$_2\text{O}_3$ (%)
1	4.6
2	4.6
3	4.5
4	4.5
5	4.6
6	4.6
Average	4.6
SD	0.1
Nominal Value	4.5

3. Colemanite. Nominal B2O3 concentration 47.8 %

Analysis B	$_2\text{O}_3$ (%)
1	48.1
2	47.4
3	47.7
4	46.6
5	49.3
6	48.7
Average	47.8
SD	0.52
Nominal Value	47.8

Precision can be further improved using longer analysis times and our TSI LIBS Desktop Analyzer XY scanning options.



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