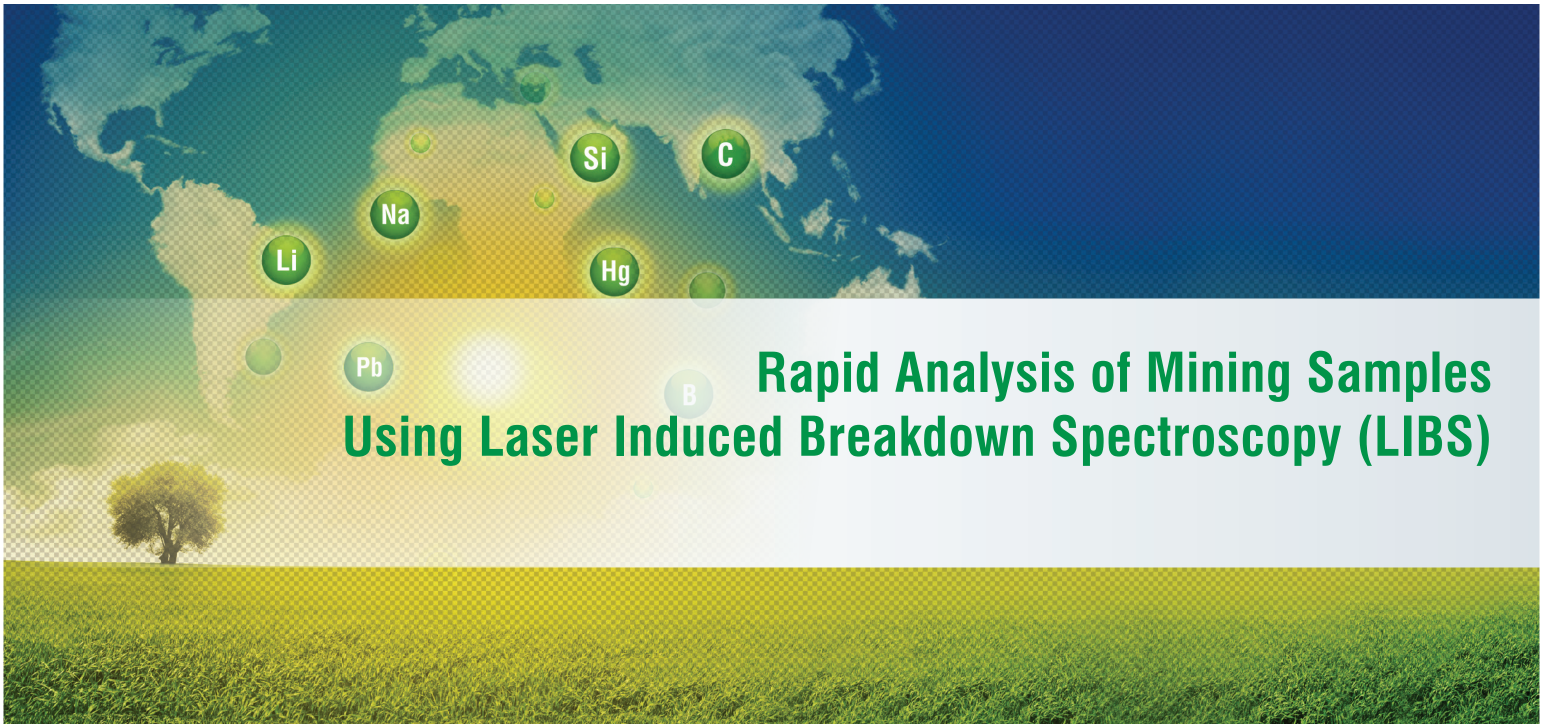
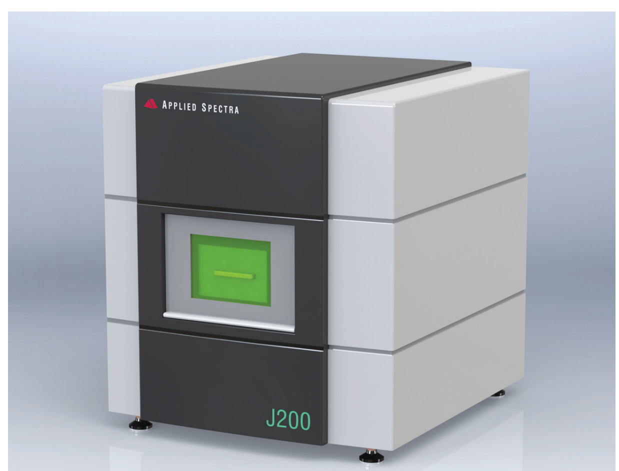



# Rapid Analysis of Mining Samples Using Laser Induced Breakdown Spectroscopy (LIBS)

## Introduction

The mining industry's contribution to the world economy has steadily grown over the last decade due to rapid growth in emerging economies such as China, Russia, India, and Brazil. A variety of natural resources are mined including oil, coal, iron ore, gold, silver, nickel, and copper. Most mining activities involve many processes that include collecting raw materials from mining sites and getting these materials into a form that can be treated and eventually concentrated.




Applied Spectra Inc.'s J200 LIBS Instrument

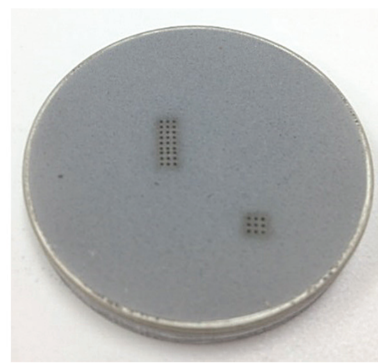
To evaluate the effectiveness of these different processes that lead up to final mining products, critical elements are quantified and monitored by several analytical techniques. These techniques typically consist of XRF, GFAA, ICP-AES, ICP-MS, SEM, and SIMS.

Laser-induced breakdown spectroscopy (LIBS) is a rapidly-emerging analytical technique and has garnered increasing interest over the past few years from a variety of industries. LIBS offers many attractive analytical advantages with respect to other techniques for the mining industry. LIBS can detect elements from H – Pu, which includes non-metals, such as H, N, F, and O, that are often difficult or impossible to discern with other types of analysis. In addition, compared to XRF, LIBS provides high sensitivity for lighter elements (B, Li, C, K, Ca, Mg, Al, Si, etc.) that are critical for several mining applications.

Certified Reference Materials (CRMs) from African Mineral Standards (AMIS) were analyzed to demonstrate LIBS as an effective analytical method for mining applications. The J200 LIBS instrument, from Applied Spectra, Inc., equipped with a 266 nm Nd:YAG (ns) laser and a broadband detector was used for the analysis.

## Sample Analysis

Using the J200 LIBS instrument, 8 mining reference samples were analyzed under optimized conditions. The samples were pressed into pellets, similar to how XRF samples would be prepared. This evaluation will show that these samples can be analyzed using LIBS without changing the current means of sample preparation used in the mining industry. Pressed pellets were analyzed by ablating 9 locations per sample with a laser spot size of 200 $\mu$m. All data was analyzed using Applied Spectra's data analysis software package. A typical LIBS spectrum (190 – 1000 nm) from AMIS 171 is displayed in Figure 1.



Pressed Pellet: Post sampling; 9x3 and 3x3 laser-sampling grid.

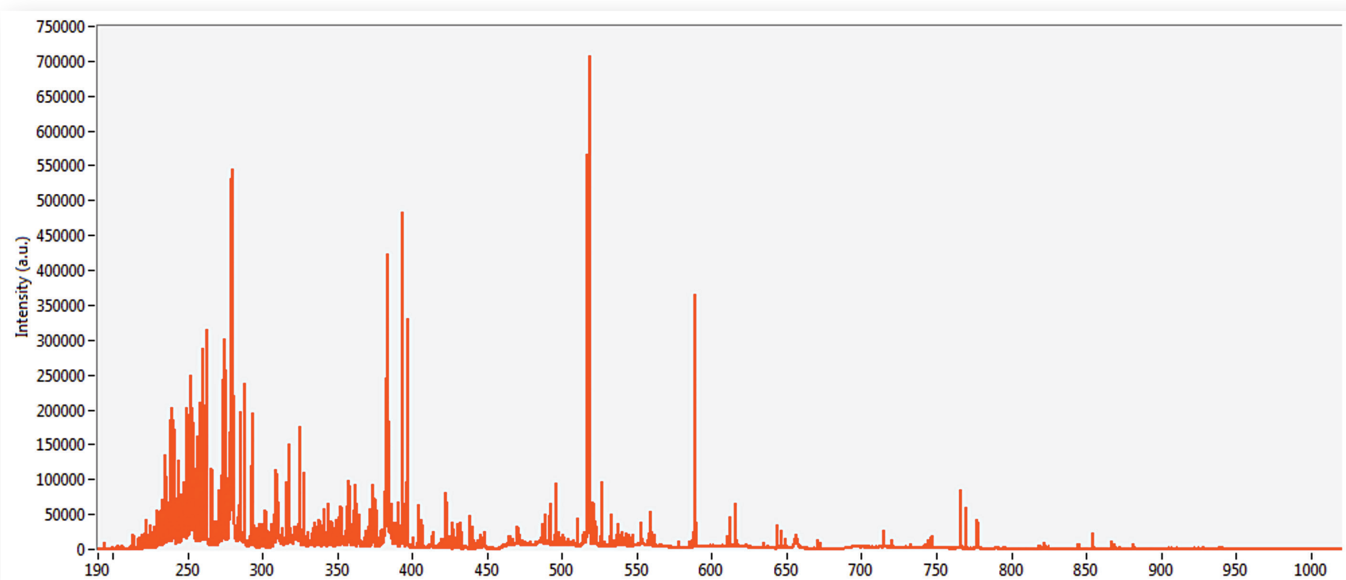



**Figure 1.** Typical LIBS spectrum of AMIS 171 acquired with the J200 LIBS instrument.

Using Applied Spectra's data analysis software package, Principal Component Analysis (PCA) can be performed on the data set to understand variabilities in sample composition (Figure 2). If there are differences in composition of the major and minor elements within the different samples, clear groupings of data points in the PCA plot are visible. By reviewing a PCA plot resulting from the analysis of LIBS data, a user can effectively understand the samples' composition variability and monitor the consistency of mining samples.

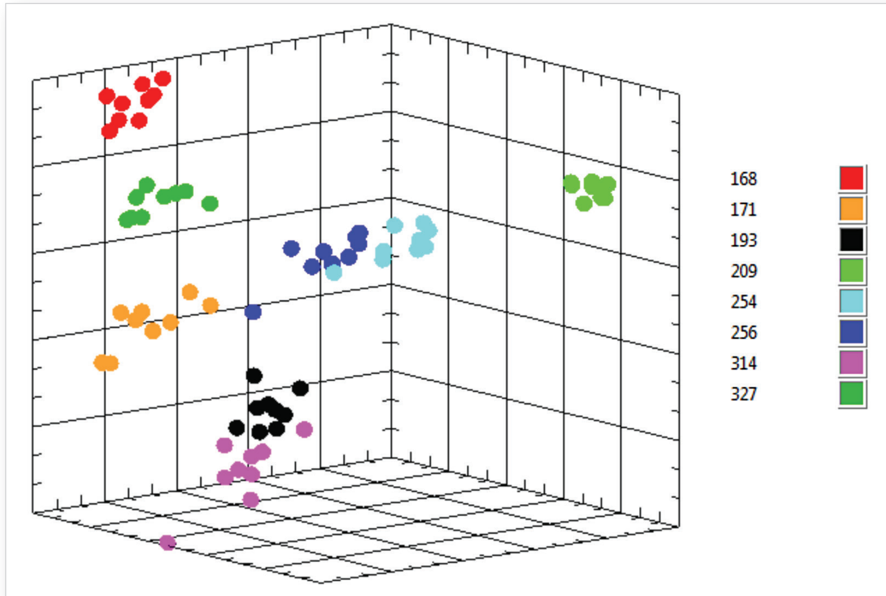



**Figure 2.** Principle Components Analysis (PCA) plot based on LIBS data of the mining sample sets analyzed.

Despite sample matrix differences that exist among samples within the same set, an accurate calibration model can be developed to predict the concentration of elements of interest, based on ASI's powerful multivariate calibration algorithm. The mining reference samples were used to create a multivariate calibration curve in Applied Spectra's data analysis software. Seven of the samples were used to create the calibration curve and one sample (AMIS 193) was used to determine the accuracy and precision of the method. The multivariate calibration responses for Al and Co are displayed in Figure 3. The concentration of Al ranged from 2.09 to 12.4% while Co ranged from 49 ppm to 820 ppm.

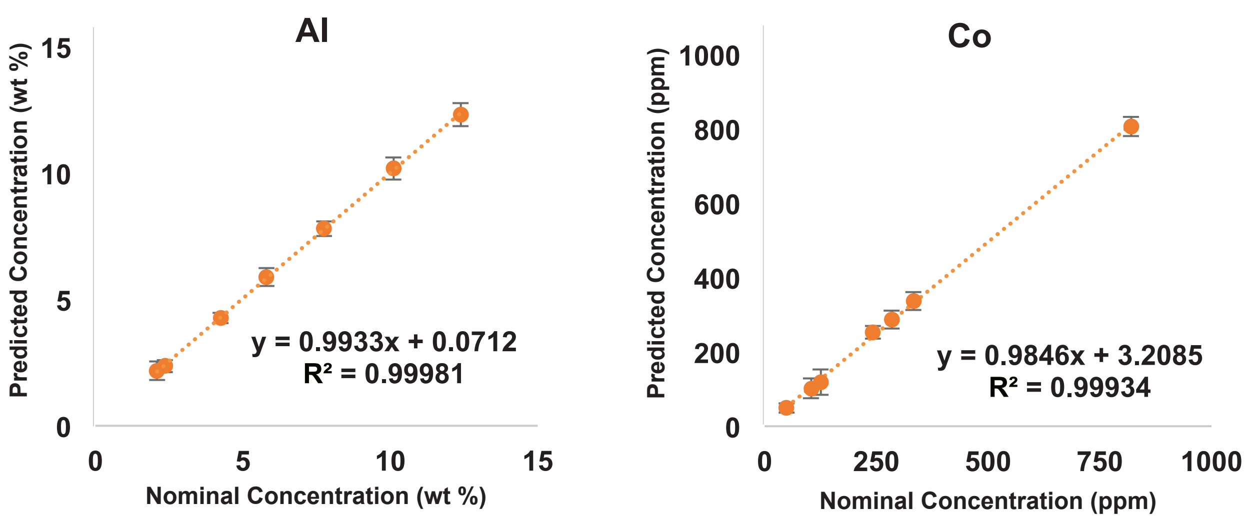



**Figure 3.** Multivariate calibration curves for Al (2.09 – 12.4 wt. %) and Co (49 – 820 ppm).

**Table 1.** Accuracy and precision for AMIS 193 African mining certified reference sample.

| | Reference Values | | | Measured Values | | |
|---|---|---|---|---|---|---|
| **Elements** | **Avg.** | **Range** | **Units** | **Avg.** | **RSD** | **% BIAS** |
| **Al** | 7.77 | 7.17 - 8.37 | wt % | 7.21 | 3.1 | -9.8 |
| **Cr** | 1.26 | 1.11 - 1.41 | wt % | 1.27 | 7.0 | 1.0 |
| **Mg** | 16.94 | 16.3 - 17.6 | wt % | 16.7 | 3.6 | -1.4 |
| **Ti** | 0.202 | 0.181 - 0.223 | wt % | 0.206 | 2.7 | 2.1 |
| **Ca** | 4.82 | 4.64 - 5.00 | wt % | 4.65 | 5.7 | -3.5 |
| **Si** | 45.7 | 44.7 - 46.7 | wt % | 46.7 | 2.1 | 2.2 |
| **Co** | 358 | 292 - 424 | ppm | 361 | 4.1 | 0.9 |
| **Cu** | 8416 | 7552 - 9280 | ppm | 8261 | 3.2 | -1.8 |
| **Ni** | 13132 | 11152 - 15112 | ppm | 11175 | 3.6 | -14.9* |

* % Bias > 10 %, but measured value is within reported range from certificate of analysis.

Table 1 shows the accuracy and precision for the calibration curves built using the AMIS mining reference standard. All results were found to have < 10% Bias, except for Ni which was found to be -14.9 %. However, the measured value for Ni (11175 ppm) was within in the reported range of 11152 – 15112 ppm for Ni. The precision for the elements evaluated in this study were all ≤ 7.0 % RSD.

## Conclusion

LIBS provides the ability to analyze mining samples for multiple elements across a concentration range of ppm to wt. %. In this application note, it was shown that the samples in the pressed pellet form, a sample matrix similar to what is common for XRF measurements, were analyzed using LIBS with good accuracy and precision. Since LIBS has the capabilty to measure important lighter elements such as Li, B, C, H, O, N, Al, Ca, Mg, and Si more effectively than XRF, LIBS is a great complementary technique for the mining industry. Using the same type of sample preparation already implemented in the mining industry, such as the before mentioned pressed pellets, Applied Spectra's J200 LIBS is an integral technique that can provide elemental information for H, Li, Be, B, C, N, O, and F, which can be difficult or impossible to acertain with other methods of analysis.

**Applied Spectra, Inc.**
46665 Fremont Blvd.
Fremont, CA 94538
Telephone: + 1 510.657.7679
Fax: + 1 510.657.7074
Sales Support: + 1 510.657.7679 x410