

B176 Elemental Analysis of Presumptive Clandestine Laboratory Evidence Using Laser-Induced Breakdown Spectroscopy (LIBS)

Annessa L. Burnett, BS*, 1200 W Marshall, Apt 205, Richmond, VA 23220; Douglas DeGaetano, MS, Commonwealth of VA, Dept of Forensic Science, 700 N 5th Street, Richmond, VA 23219; and Rebecca Wagner, PhD, Virginia Department of Forensic Science, 700 N 5th Street, Richmond, VA 23219

The goals of this presentation are to introduce the fundamentals of LIBS, define what is needed for trace element identification, and explain how it can be incorporated into the elemental analysis of clandestine laboratory evidence.

This presentation will impact the forensic science community by explaining the advantages of continuing to integrate LIBS in trace elemental analyses and explaining the potential use of the LIBS instrument in forensic cases.

Clandestine laboratories have been a rapidly growing problem throughout the United States. The ease of obtaining the ingredients to manufacture methamphetamine is a growing concern; therefore, analytical methods for the detection of these ingredients are critical. Numerous elemental analysis techniques currently exist in forensic science; however, LIBS is a rapidly expanding technique. LIBS uses a low-energy laser pulse in conjunction with lenses to generate a plasma when the laser beam interacts with the sample surface. A spectrum is generated that displays atomic and ionic spectral emission lines unique to the elements present. This spectrum is essentially a "fingerprint" of the respective elements present in the sample. The main objective of this research was to develop and optimize a method to identify elements commonly found in clandestine laboratory evidence. This was accomplished by examining the changes in the data caused by varying parameters of the instrument including: spot size, number of shots, spectrometer delay, repetition rate, laser intensity, accumulation of data, and gas flow. Helium and argon were used to optimize elemental excitation. The elements included in this study are commonly encountered in the methamphetamine manufacturing process including: sodium, sulfur, chlorine, red phosphorus, iodine, and lithium. An advantage of LIBS is that it can detect lighter elements, such as lithium, which is not possible with scanning electron microscopy-energy dispersive spectrometry.

The samples were evaluated during method development and optimized for each instrumental parameter. The data were reviewed for peak shape, peak abundance, and the presence of atomic and ionic spectral lines. Sample preparation techniques were also compared for each sample including: embedding in nail polish, using double-sided adhesive tape, and the use of adhesive dots. Three replicates for each sample preparation technique were examined in triplicate to observe homogeneity of the sample preparation technique as well as matrix effects. Once the optimal sample preparation technique was established, a large number of samples for each element were analyzed in triplicate to evaluate reproducibility and determine a potential threshold for elemental identification. The optimized methods for each element as well as identification threshold levels were also applied to mock case samples.

The neat samples were successfully optimized for each instrumental parameter. The intensity of the spectral lines for sodium, red phosphorus, and lithium were detected in an ambient atmosphere, while helium was needed to increase the spectral line intensities for sulfur, chlorine, and iodine samples. Some of the elements of interest displayed spectral line overlaps, and in some cases, the adhesive/ sample preparation technique demonstrated interferences. A minimum of three to four spectral lines were used to determine if an element was present. Overall, LIBS has the potential to be a powerful technique for forensic trace elemental analysis.

LIBS, Methamphetamine, Clandestine Laboratories

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