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Elemental Analysis and Comparison of Bulk Soil Using LA-ICP-MS and LIBS methods

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Elemental analysis methods utilizing Laser ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS) and laser-induced breakdown spectroscopy (LIBS) were developed and used in the characterization of soil samples from the US and Canada as part of a comprehensive forensic evaluation of soils. A LA-ICP-MS method was recently optimized for analysis and comparison between different soil samples in an environmental forensic application [1,2] and LIBS has recently attracted the interest of analytical chemists and forensic laboratories as a simpler, lower cost alternative to the more established analytical methods. In developing a LIBS method, there are many parameters to consider, including laser wavelength, spectral resolution, sensitivity, and matrix effects. The first LIBS method using a 266 nm laser for forensic soil analysis has also been recently reported by our group [3]. The results of an inter-laboratory comparison involving thirteen (13) laboratories conducting bulk elemental analysis by various methods are also reported. The aims of the inter-laboratory tests were: a) to evaluate the inter-laboratory performance of three methods (LA-ICP-MS, μXRF and LIBS) in terms of accuracy (bias), precision (relative standard deviation, RSD) and sensitivity using standard reference materials (SRMs); b) to evaluate the newly released NIST SRM 2710a, which supersedes 2710; and c) to evaluate the utility of LIBS as an alternative technique to LA-ICP-MS and μXRF for bulk analysis of soils. Each sample and standard was homogenized in a high-speed ball mill and pressed into pellets. Participants were instructed to measure the following elements: 7Li, 25Mg, 27Al, 42Ca, 45Sc, 47,49Ti, 51V, 55Mn, 88Sr, 137Ba, 206,207,208 Pb (LA-ICP-MS); Ti, Cr, Mn, Fe, Cu, Sr, Zr, Pb (μXRF); Ba, Cr, Cu, Fe, Li, Mg, Mn, Pb, Sr, Ti, Zr (LIBS). For both LIBS and μXRF, the choice of appropriate spectral lines was determined by the user, optimizing for linearity, sensitivity and precision.

Results for both LA-ICP-MS and μXRF were generally consistent for most elements, resulting in good intra-laboratory precision (< 8 % RSD for LA-ICP-MS; < 20 % RSD for μXRF) and low bias (< 10% for LA-ICP-MS; < 35 % for μXRF), which are important characteristics for forensic comparison of soils. Linear calibration curves were also obtained for both μXRF and LIBS. Results for LIBS showed good precision (< 15 %) and bias (< 15 %) for most elements. Limits of detection for trace and minor elements were in the 0.01 – 1 ppm range for LA-ICP-MS and 1 to 200 ppm for LIBS.

Finally, the results of a study comparing the bulk elemental composition from soil collected in different locations in Florida and in Canada for the purposes of providing forensic information as part of a broader forensic examination of soil samples are also reported.

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