A68 Calcium and Phosphorus Detection Using Benchtop vs. Hand-Held X-Ray Fluorescence (XRF) Spectrometers

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After attending this presentation, attendees will be familiar with the different detection levels of calcium and phosphorus when using hand-held and benchtop XRF spectrometers.

This presentation will impact the forensic science community by confirming that XRF-generated Calcium to Phosphorus (Ca/P) ratios using either hand-held or benchtop XRF devices are a valid criteria for distinguishing between different material sources and by highlighting the need for users to evaluate Ca/P ratios in reference to those made under similar analytical conditions.

The analysis of skeletal remains (or potential skeletal remains) for chemical and elemental properties is becoming increasingly common in forensic anthropological investigations. XRF is one method of elemental analysis that has been used with increasing frequency, in part due to its ease of use and the fact that little to no sample preparation or destruction are required. XRF analyses have traditionally been performed in a laboratory setting using large benchtop XRF spectrometers. These machines offer a variety of benefits including the ability to control and vary the testing atmosphere. Recently, portable or hand-held XRF units have been developed, with the advantage of being able take the device into the field to perform preliminary or conclusive field tests *in situ* or even for use in a laboratory setting while minimizing the instrument footprint; however, these portable devices may pose potential analytical complications in forensic anthropological cases due to the fact that analyses are all performed in open air, which may affect phosphorus detection since elements of low atomic number are at risk of significant absorption by air. This presentation examines the detection of calcium and phosphorus using two different XRF instruments, a benchtop and a hand-held, and compares results.

Calcium and phosphorus content in osseous and dental material from human and non-human sources were measured by XRF to obtain Ca/P ratios. Samples were cut using a diamond wafering saw to reveal cross sections with flat, smooth surfaces for measurement and included human and non-human (cow, deer, and pig) bones as well as teeth from human, deer, and pig. Shell and coral samples (which are known to have high levels of Ca but little to no P) were also examined. A control sample consisting of calcium hydroxyapatite powder was also analyzed.

Samples were analyzed under three conditions using two XRF instruments. Specimens were analyzed in both air and vacuum atmospheres using a benchtop spectrometer and were analyzed in air using a field-portable XRF. The X-ray tubes of both instruments were operated in unfiltered conditions with an excitation voltage of 20kV. The collection time was maintained consistently at 100 seconds live time. Spectral analysis was performed using instrumental software, and calcium and phosphorus emissions were used to calculate Ca/P ratios using the counts detected in the region of each of the peaks summed over five channels.

Analysis of Variance (ANOVA) shows highly significant differences in Ca/P ratios obtained under the three analysis conditions for individual sample groups as well as for all bones combined. These differences are due in part to overall differences in the detection of both elements, but differences in the detection of phosphorus contribute most significantly. Detection levels for both elements were overall lowest using the hand-held instrument. Detection levels for both elements were greater when using the benchtop in air and greatest when analyses were performed on the benchtop in a vacuum.

Calcium and phosphorus are both measureable under all three analytic conditions included in this study; however, since the calcium and phosphorus signals are attenuated differently by air, comparing measurements made under different atmospheric conditions may be misleading. To use XRF-generated Ca/P ratios to evaluate skeletal versus non-skeletal origin, measurements should be made under consistent analytical conditions. That is, known skeletal and non-skeletal samples should be evaluated to establish the performance of an instrument and specific measurement conditions prior to attempting to evaluate an unknown. When measured under the same conditions, XRF-generated Ca/P ratios are valid criteria for distinguishing differences between material sources.

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