A180 Identification of Small Mineral Grains Separated From Soil Samples Using MicroRaman Spectroscopy

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After attending this presentation, attendees will become aware of the possibility of a simple preparation of mineral grains from small soil samples suitable to obtain Raman spectra and the potential of using such spectra in the examination and comparison of forensic soil evidence.

This presentation will impact the forensic science community by discussing how soil mineral grain comparison using Raman spectroscopy should become a simple and useful part of forensic soil examination and comparison.

Analysis and comparison of soils can be greatly aided by identification of mineral grains present in the soil. However, mineral identification using polarized light microscopic methods, as currently done, requires considerable specialized training and skill and can be time consuming. Many forensic laboratories do not have an examiner with such training. There exist on the internet several quite large databases of Raman spectra of minerals. Instrumental analysis followed by the use of a suitable Raman database[1] should provide a less time consuming and more easily implemented method of soil mineral analysis.

Reffner et al.[2] have recently published a small database of known mineral spectra using attenuated total reflectance (ATR) to obtain infrared spectra by crushing mineral grains against the diamond ATR crystal. Raman spectroscopy has the advantage of allowing spectral data to be captured in the 600 to 50 cm^{-1} region, where many minerals have strong absorption; this region is not available with most infrared detectors. Since it has been found that many dark colored mineral grains give very weak Raman absorption, infrared might be very useful for improving the identification of such mineral grains.

The researchers have developed and tried out a simple method for recovering two different sized fractions of sand-sized mineral grains. If a small (generally two grams or less) sample of soil is deflocculated by stirring the soil overnight in a 4% solution of sodium metaphosphate followed by agitation and rinsing first with fresh deflocculating solution and then several times with distilled water, material suitable for size separation is obtained. Three sieves were chosen to do this separation, numbers 60 (250 µm), 120 (125 µm), and 170 (90 µm). The material is wet sieved through this set of sieves with distilled water and then alcohol and the materials in the 120 and 170 sieves are dried in a warm oven. This produces two fractions of small mineral grains that are quite clean and are suitable for direct Raman analysis. Excellent Raman spectra have been obtained on many of these grains that are suitable for identification using a large Raman mineral database (RRUFF) compiled jointly by Caltech and the University of Arizona.[1]

Approximately 15 soil samples were collected over about a five mile stretch of Connecticut route 34 by sampling from intersections with easily available soil. Additional suburban soil samples were collected from several different locations also in Connecticut. These samples were processed as described above to produce two sand grain-sized mineral fractions each. Samples were examined by spreading a small amount of mineral grains on a cleaned piece of aluminum sheet cut to microscope slide size. The aluminum slide had a small grid scratched onto the surface to allow a systematic search of the sample. Mineral grains were then examined to produce Raman spectra that were then searched against the RRUFF Database. In several cases grains were found with what appeared to be good quality spectra, but they did not match any of the minerals in the Database. Grains of five to ten different types, some identified as a particular mineral or as from a mineral family, were found in each sample. Those that did not match any of the spectra in the Database but provided distinctive Raman spectra were likely grains with a mixture of two or more different minerals.

Over twenty soil samples were examined to determine if this technique can provide useful data toward differentiating these samples. The data developed data in this way was found to be consistent and reproducible.

References:

1. B. A. Weinger, J. A. Reffner, P. R. DeForest; J Forensic Science; Vol. 54; No. 4; pp851-856; 2009
2. http:minerals.caltech.edu/FILES/raman/

Raman Spectroscopy, Minerals in Soil, Soil Comparison

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