B140 Soil Analysis Using Automated Scanning Electron Microscopy

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Upon completion of this presentation, attendees will have been introduced to the use of automated scanning electron microscopy for forensic soil comparisons and provided data from a blind study that validates the technique for use in the crime laboratory.

This presentation will impact the forensic science community by describing a practical means for crime laboratories to use soil as trace evidence.

The most common approach for characterizing soil is to consider its color; relative amounts of gravel, sand, and silt; the mineral composition including heavy minerals; biological materials like pollen and phytoliths; and building materials. The minerals are identified using polarized light microscopy (PLM) and the number of each mineral type is tallied.

The identification of individual mineral grains requires a skilled specialist and the resulting tally is based on the examiner's subjective observations. Therefore, more practical methods of identifying and counting the soil mineral grains might be useful to crime laboratories. The recent development of automated scanning electron microscopes employing energy dispersive x-ray spectrometry (SEM/EDS) could offer practical advantages for soil analysis. Many crime laboratories are using these instruments for gunshot residue (GSR) analysis; thus these instruments may already be available for soil analysis.

McCrone Associates currently uses automated SEM/EDS to search for specific particles of interest such as the search for GSR. The other use of automated SEM/EDS is to briefly survey and catalog all particles by stopping and analyzing all particles on the stub without regard to their composition. The analysis produces EDS spectra (elemental composition) and images (morphological features) from each particle in turn. Thousands of particles can be quickly analyzed unattended (approximately ten grains per minute), producing a count of the minerals in the sample.

Soil samples, previously collected from three rural counties in Michigan, were selected at random for a blind study. The analysts were never informed of the source of any of the samples and which were duplicates. The blind trials used soil from fourteen sites, separated into two duplicates from each site; three different grain sizes and two replicate samples of each size were prepared by reverse sieving. Approximately 2000 grains from each sample preparation were analyzed. Finally, after all analyses were completed, the soil unit from which each sample was collected was disclosed in order to evaluate the data. Each of the original samples would likely be different from the others, but the duplicates should be similar.

Soil is prepared for automated SEM/EDS analysis by reverse sieving. After sieving and cleaning a chosen grain size fraction, the grains are passed back through the same sieve and affixed to a 10 mm aluminum stub topped with conductive double-sided carbon sticky tabs. For example, grains that passed a No. 100 mesh sieve and collected on a No. 120 mesh sieve were passed back through the No. 100 mesh sieve onto the stub. The preparation results in a single layer of particles, between 125 and 150 micrometers, with none touching another in the pattern of the mesh.

An ASPEX 3025 Low Vacuum (LV) Personal Scanning Electron Microscope (PSEM) equipped with an Energy Dispersive X-ray Spectrometer (EDS) for elemental analysis was used to analyze the soil grains. Using EDS, only the mineral's elemental composition is available to differentiate between mineral classes. For example, microcline and orthoclase, which have a different crystal structure but the same chemical stoichiometry ($(K,Na)AlSi_3O_8$), cannot be differentiated except by optical crystallography. On the other hand, EDS can easily distinguish between some minerals such as rounded quartz and untwined plagioclase feldspars (oligoclase) that are not rapidly distinguished in grain mounts by PLM, without observing interference figures. The output of the EDS software is a normalized k-ratio, which is roughly equivalent to elemental weight percent. An Excel library of expected values based on elemental weight percent for more than a hundred common minerals was generated using published formulae supplemented by the analysis of known minerals from which a classification was defined. EDS data from each grain were imported into the spread sheet and the mineral identified by comparing the EDS data with the table of expected values.

Automated SEM/EDS analysis in a blind study found that soils from the same site could be matched and most soils from other sites were different in their mineral tallies. Automated SEM/EDS should be considered preliminary to PLM; morphological analysis, whether by PLM or SEM, is essential to identify anthropogenic and biogenic materials that cannot be distinguished by EDS alone. Additional studies to determine whether automated SEM/EDS is useful for comparison of soils from regions other than the American Midwest and to measure the discriminating power of the method on samples from similar soil units are recommended.

Soil, SEM/EDS, ASPEX