B37 Analysis of Forensic Soil Samples Via High Performance Liquid Chromatography and Ion Chromatography

Christopher R. Bommarito, MSP, and Amanda D. Sturdevant, MS, Michigan State Police Forensic Science Division, 7320 North Canal Road, Lansing, MI 48913; and David W. Szymanski, MS, Department of Geological Sciences, Michigan State University, 206 Natural Science Building, East Lansing, MI 48824*

The goals of this presentation are to demonstrate the use of high performance liquid chromatography (HPLC) and ion chromatography (IC) to assess the qualitative and quantitative variation in these fractions of soil and to demonstrate spatial and temporal variation in soil and the importance of population sampling in soil analysis.

This presentation will impact the forensic community and/or humanity by showing how the use of these methods will decrease the number of type II errors in soil analysis and demonstrating the spatial and temporal variation will demonstrate the need for population sampling in soil analysis.

Traditional forensic soil comparisons are performed via physical and/or chemical examinations of color, texture, and mineral content, leaving any organic- or water-soluble fractions unexamined. This study uses high performance liquid chromatography (HPLC) and ion chromatography (IC) to assess the qualitative and quantitative variation in these fractions of soil.

Soil samples were collected over the course of three weeks from 120 locations in and around Lansing, MI and were designated as urban (n=40), suburban (n=40), or rural (n=40). Criteria used to categorize each areas included the local human population, the amount of vehicle and pedestrian traffic, the distance from residential or commercial structures, and the general use of the land as commercial (urban), residential (suburban), or agricultural (rural). Additional samples from six of these locations (two urban, two suburban, and two rural) were collected once a week for ten weeks for temporal analysis. Nine additional samples, equally spaced over a 1 m^2 grid, from these same six locations, were collected for spatial analyses.

All samples were collected using a #9 soil plug to a depth of about 1" five times and stored in brown paper bags. Each sample was placed in a glass petri dish, dried in a 60°C oven for two hours, and sieved through a 60/250 mesh/micron Tyler certified brass sieve. The fraction that passed through the sieve was stored in a vial for analysis. The Dionex HPLC system consisted of a P680 pump with an ASI-100 autosampler and UVD340U diode array detector. The columns used were a Phenomenex Widepore C18 guard column (4 mmL x 2 mmD) and an Alltech Widepore Econosphere C18 column (5 u particle size, 250 mm x 4.6 mm). Various combinations of extract concentrations, mobile phase compositions, and run times were tested to determine the optimal sample preparation method and system parameters to achieve sufficient peak resolution.

IC samples were run on a Dionex DX-120 ion chromatograph with a Dionex AS40 autosampler and electrochemical detector. Samples were analyzed on two different columns and by two separate methods. The first method utilized an IonPac AS9-HC (4 mm x 250 mm) column with a mobile phase of 9 mM Na_2CO_3 at a flow rate of 1.19 ml/min for 35 minutes and a 25 µl injection volume to detect and quantify nitrite, bromide, chlorate, nitrate, phosphate, and sulfate. The second method utilized an AS16 (4 mm x 250 mm) column with a mobile phase of 35 mM NaOH at a flow rate of 1.19 ml/min for 35 minutes and a 25 µl injection volume to detect and quantitate perchlorate, thiosulfate, and chlorate.

All samples were prepared for IC analysis using no less than 0.5 g of sieved soil in a 0.5 g/ml solution in reagent grade water. This solution was sonicated for ten minutes and filtered through a 0.45 µm syringe filter (Pall) into two 0.5 ml IC autosampler vials.

Qualitative and quantitative analysis of the resultant chromatograms separated the 120 samples into 10 groups by HPLC and 23 groups by IC groups.

This study shows that using HPLC and IC to analyze the organic- and water-soluble fractions of soil can successfully discriminate samples. Including quantitative analysis of the results eliminates some false inclusions by providing further differentiation of samples. To demonstrate that the variation observed via HPLC and IC analysis is an independent variable from the inorganic composition, ten samples that were differentiated by these methods were examined via X-Ray fluorescence. Some of the samples were broadly similar in elemental composition in a one to one comparison. Although this comparison was not performed with a population of known samples, the XRF data indicates that additional discrimination is possible when HPLC and IC analysis are added to traditional forensic soil analysis schemes.

The methods used in this study were able to detect both qualitative and quantitative variations in soil over a relatively small geographic area. This demonstration of soil heterogeneity underscores the importance of the collection of a representative known sample population when assessing a forensic soil comparison. Significant temporal variation was also demonstrated over the course of ten weeks of sampling; however, samples were found to be consistent over shorter periods of time.

Baseline levels of inorganic anions were determined via IC; these levels may be useful in assessing the significance of anions detected in soil from cases involving low explosives.

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Soil Analysis, High Performance Liquid Chromatography, Ion Chromatography

 ** Presenting Author*