A Comparison of Methods for Analyses of Soil Trace Metals in a Mining Impacted Agricultural Watershed[1]

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Abstract: Field portable X-ray fluorescence (XRF) has become an increasingly popular technology for *in-situ* detection of trace metals. This technology allows for rapid screening of environmental contaminants when compared to other techniques, like inductively coupled plasma-optical emission spectrometry (ICP-OES) or mass spectrometry (ICP-MS). The accuracy of *in-situ* XRF analyses has been questioned due to possible interference from elevated soil moisture and organic content. In this study, three metals analysis protocols were compared for surface soil samples. Soil samples were collected near the Tar Creek Superfund Site in northeastern Oklahoma. A field portable XRF spectrometer was used *in-situ* for analysis of metals concentrations in small field plots cleared of vegetation and debris. Collected samples were homogenized, pulverized, air dried, and sieved to < 250 um fraction in the laboratory and re-tested using the field portable XRF. Samples were also analyzed via microwave-assisted hot HNO_3 digestion followed by ICP-OES analyses. Moisture content and loss-on-ignition (as a surrogate for organic matter) were determined for each sample. Soil moisture exceeding 10% in the field was found to decrease the accuracy of XRF metals concentrations readings. Elevated moisture contents caused underreporting of field XRF readings when compared to the laboratory XRF readings. Relationships between laboratory XRF and ICP-OES concentrations for lead ($r^2 = 0.96$) and zinc ($r^2 = 0.91$) were strong. No statistical relationship between soil organic content and XRF accuracy was established. The relationship for ICP-OES concentrations for cadmium and zinc resulted in an r^2 of 0.93 which allowed for prediction of cadmium concentrations for samples not analyzed by the ICP-OES. This study recommends that when analyzing samples with the field portable XRF, samples should be homogenized, air dried, sieved and analyzed in the laboratory, rather than *in situ*, to yield the most accurate results.[3]

Additional Key Words: XRF, ICP-OES, moisture content, loss-on-ignition

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[3] Work reported here was conducted near 36° 54’ 41.85” N; 94° 55’ 53.56” W.