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## Progress Report

### Development of Synchrotron X-Ray Micro-Spectroscopic Techniques and Application to Problems in Low Temperature Geochemistry

(DE-FG02-92ER14244)

This project is a collaboration between geoscientists experienced in the use of synchrotron radiation in earth materials research and low temperature geochemists and soil and environmental scientists. Our aim is to develop a single, synchrotron-based microprobe that can be used to determine the composition, structure, oxidation state, and bonding characteristics of earth materials with trace element sensitivity and  $\mu\text{m}$  spatial resolution. Geochemical applications include the nature of hydrothermal fluid inclusions, toxic metal and radioisotope speciation in low level wasteforms, uranium speciation in contaminated sediments, and aerosol climatology. The goal is to extend this research at the next generation of synchrotron radiation source currently under construction at Argonne National Laboratory, the Advanced Photon Source (APS).

The focus of the technical development effort has been the development of apparatus and techniques for the utilization of X-ray Fluorescence (XRF), Extended X-ray Absorption Fine Structure (EXAFS) and X-ray Absorption Near Edge Structure (XANES) spectroscopies in a microprobe mode. The present XRM uses white synchrotron radiation (3 to 30 keV) from a bending magnet for trace element analyses using the x-ray fluorescence technique. Two significant improvements to this device have been recently implemented. **Focusing Mirror:** An 8:1 ellipsoidal mirror was installed in the X26A beamline to focus the incident synchrotron radiation and thereby increase the flux on the sample by about a factor of 30. **Incident Beam Monochromator:** The monochromator has been successfully installed and commissioned in the X26A beamline upstream of the mirror to permit analyses with focused monochromatic radiation. The monochromator consists of a channel-cut silicon (111) crystal driven by a Klinger stepping motor translator. We have demonstrated the operating range of this instrument is 4 and 20 keV with 0.01 eV steps and produces a beam with a  $\sim 10^{-4}$  energy bandwidth. The primary purpose of the monochromator is for x-ray absorption spectroscopy (XAS) measurements but it is also used for selective excitation in trace element microanalysis. To date, we have conducted XANES studies on Ti, Cr, Fe, Ce and U, spanning the entire accessible energy range and including both K and L edge spectra. Practical detection limits for microXANES are 10-100 ppm for 100  $\mu\text{m}$  spots.

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The technical advances described above have led to important applications in low temperature geochemistry several of which are summarized here.

**Hydrothermal Fluid Inclusion Research** (Collaborators: D. Vanko, Georgia State University; R. Bodnar, Virginia Polytechnic Institute)

The chemical nature of hydrothermal fluids and their metal-carrying capacity are significant geochemical characteristics that speak to the chemical evolution of seawater as it permeates oceanic crust, and the ability of the fluid to transport and deposit massive sulfide deposits on the seafloor. Individual fluid inclusions can be analyzed within the host mineral using synchrotron radiation microanalytical techniques. Using a series of synthetic inclusions in the system  $\text{NaCl} + \text{H}_2\text{O} \pm \text{CaCl}_2 \pm \text{KCl}$ , the SXRF technique was used to determine ratios of K/Cl and Ca/Cl. Results show that such major element ratios may be determined without standard analyses with accuracies better than  $\pm 30\%$  on individual  $10 \mu\text{m}$  inclusions (Vanko et al 1992). Initial trace element studies of natural fluid inclusions have been done on specimens from extinct seafloor hydrothermal systems and from a major porphyry Mo deposit. Brine-filled inclusions, typically  $10 \mu\text{m}$ s across, and containing halite-saturated solutions, were shown to have fluids dominated by sodium, calcium, and potassium chloride. At higher energies, inclusions may contain Mn, Fe, Cu, Zn, Pb, Br, and Mo. These results are exciting because of the potential for quantifying the concentrations of major and minor elements in hydrothermal fluids from a multitude of geochemical settings.

**Toxic Metal and Radioisotope Speciation in Hazardous and/or Radioactive Wasteforms** (Collaborator: S. B. Clark, University of Georgia, Savannah River Ecology Laboratory)

Liquid waste streams contaminated with low levels of radioactive and/or hazardous metals are frequently disposed of by solidification in cement matrices. This process involves encapsulation of the metal bearing liquid in a porous cement medium. Ideally, the cement provides stabilization of the contaminants by controlling diffusion out of the matrix. However, understanding the chemical speciation of the metals in these matrices is essential to developing a cement formulation that provides controlled diffusion of the contaminants. Our efforts have been focused on determining the speciation of toxic metals and radionuclides in cement-based wasteforms representative of those generated at the Savannah River Site. To date, we have developed a XANES method to quantify the amount of  $\text{Cr}^{6+}$  in these wasteforms (Bajt et al., 1992). We have demonstrated that the intensity of the XANES pre-edge feature is directly

proportional to the quantity of chromate in the sample, and is independent on sample matrix. The results indicate that slag-based cements quantitatively reduce the chromate to  $\text{Cr}^{3+}$ , leading to enhanced retention of chromium in the wasteform.

**Air Mass Aerosol Climatology** (Collaborators: R. Grant and D. Schulze, Purdue University)

The size and composition of ambient aerosols in the troposphere are critical in determining the interaction of aerosols and the environment. Trace elements in the atmosphere are important in local and regional toxicity assessments. Results of x-ray microprobe analysis of eight size-fractionated aerosol samples chosen to represent air masses coming rapidly out of Canada (cP or continental polar air masses), off the Atlantic coast (mP or maritime polar air masses), and up from the Gulf of Mexico (mT or maritime tropical air masses) as well as air having resided over the Midwest USA for greater than 4 days show some intriguing trends (Grant et al. 1992). As expected, the Midwest coarse fraction aerosol (between 20 and 7 micron diameter) had high levels of many metals including Ti, Mn, Fe, Ni, Cu, Zn, Pb, and Sr at high levels. The high levels of Cu and Zn, however, were not found in the cP or mT aerosol indicating Midwestern sources of the coarse Cu and Zn- possibly smelters. The Midwest fine fraction aerosol (less than 1.1  $\mu\text{m}$  diameter) showed high levels of Fe and Cu and moderate levels of Zn. While Fe was commonly found in the cP aerosol fine fraction, Ti, Fe, and Cu were more prevalent in the mT fine fraction aerosol. The coincident elemental compositions of individual particles and/or clusters of particles from these aerosol samples were determined using two-dimensional (2D) scans (10  $\mu\text{m}$  resolution) of the sample surface. The coincidence of different elements in the same area of the filter can show causative or coincidental association of the elements by an analysis of their co-occurrence frequency. Preliminary results of 2D scans of a Midwest aerosol show the utility of this analytical technique. In the Midwest aerosol, the coarse fraction of Ca, Mn, Ti, and Fe rich areas of the filter were commonly coincident suggesting the particle was of mineral earth origin. The coarse fraction aerosol also showed Cu highly associated with these apparent mineral earth particles while other Cu rich areas (approx. 30  $\mu\text{m}$  diameter) were not associated with the mineral earth particle. Apparently, the coarse fraction Cu exists in the aerosol as both relatively pure solid particles as well as possibly being adsorbed or condensed from the vapor state onto mineral earth particles. 2D scans of the fine fraction aerosol from the same air mass showed highly associated Ca, Fe, Ti, and Cu rich areas and different highly associated Mn and Zn rich areas.

## **Uranium Speciation in Contaminated Sediments (Collaborator: P. Bertsch, University of Georgia, Savannah River Ecology Laboratory)**

Releases of U and other metals, namely Cu, Cr, and Ni, from a nuclear reactor fuel and target fabrication facility on the Department of Energy's Savannah River Site (SRS), near Aiken, South Carolina to surrounding soils and streams have occurred since the start of the facility in the early 1950s. Specific information on the chemical speciation of U in these sediments is required for predicting its potential mobility and fate as well as for providing information essential to developing effective, yet cost effective remediation strategies. Our preliminary experiments have demonstrated that XANES measurements are possible on the contaminated sediments of interest and that measurable differences in the fingerprint region around the absorption edge as a function of chemical pretreatment are observed. Additionally, we have examined grains in  $>50 \mu\text{m}$   $<200 \mu\text{m}$  isolates from U contaminated soils collected at the Fernald nuclear processing facility near Cincinnati, OH, XANES and have found evidence for microregions within the sand fraction that are enriched in  $\text{U}^{6+}$ , and others that are highly enriched in  $\text{U}^{4+}$ , as well as others that have variable  $\text{U}^{4+}/\text{U}^{6+}$  ratios, albeit predominantly  $\text{U}^{6+}$ . The grains having predominantly  $\text{U}^{4+}$  also have high Cu concentrations and relatively low Fe, whereas the grains that are typically predominated by  $\text{U}^{6+}$  tend to have high Fe concentrations.

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## Stephen Roy Sutton

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