## High-Precision Isotope Acquisition with the NanoSIMS 50L

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The in situ measurement of isotope ratios at high precision has always been a goal of secondary ion mass spectrometery (SIMS). The NanoSIMS 50L is a third-generation ion microprobe developed by Cameca and modified in collaboration with the Carnegie Institution of Washington. This instrument encorporates a number of design and functional improvements over the stock NS50, including precise stepper motor control over all slits, apertures and stage movements, a larger magnet, and a modified multicollector (6 moveable and 1 fixed) capable of holding both Faraday cups and miniature multi-dynode electron multipliers. The instrument is capable of attaining a minimum beam diameter of <50 nanometers with Cs and <200 nanometers with oxygen (O-), a factor of 5-10 improvement over the IMS6F/7F/1280 generation of instruments. The CIW instrument is also the first NanoSIMS to be fitted with multiple Faradays and associated high-precision electrometers.

One year's experience with the NS50L has disposed of a number of incorrect perceptions with regard to its potential and capabilities for terrestrial geochemistry:

(1) "Beam currents are too low to be useful": With Cs, a routine primary beam diameter of 100 nm is obtained with 2-3pA of current, sufficient to yield >1MHz of <sup>32</sup>S from pyrite or <sup>16</sup>O from silicate glass at >6000 MRP. 250pA of current yields a 350 nm beam that gives an H<sub>2</sub>O detection limit of 50 ppm on olivine.

(2) "You'll never get enough signal for Faradays": A 2.5 nA Cs beam with a diameter of 700 nm yields 90 pA of  ${}^{32}S$  from pyrite at >6000 MRP, sufficient to analyze  ${}^{32}S{}^{-33}S{}^{-34}S$  on Faraday cups and  ${}^{36}S$  in EM @ >10,000 cps. Similar results have been obtained for oxygen from Al<sub>2</sub>O<sub>3</sub>.

(3) "The instrument is unproven at high precision": In multi-Faraday mode, spot-to-spot reproducibility is 0.4 permil (1sig) or better for 32-33-34S from pyrite, and for 28-29-30Si from a silicon wafer. Reproducibility as good as 0.15 permil (1sig) has been obtained for 10 analyses of separate grains of pyrite in different holes of a single sample holder. With multiple EMs, simultaneous measurements of Mg and Si isotopes in silicates achieves a reproducibility of ~1 permil (1sig) for all ratios from multiple craters. All of these measurements were obtained in raster craters at a spatial resolution of 5-10  $\mu$ m.

Other high-precision and high-resolution applications of the instrument will be presented at the meeting.

## Influence of reductive dissolution of iron oxides by S(II) on uranium mobility

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Transient redox conditions can influence the mobility of uranium by inducing changes in iron mineralogy with which uranium may be closely associated. Iron oxides play a critical role in uranium transport by providing reactive surfaces for sorption or by assisting in U(VI) reduction. Iron oxides reductively dissolve when exposed to S(II), produced by microbial sulfate reduction. This might lead to the mobilization of adsorbed U(VI). However, U(VI) might also remain solid-bound due to adsorption onto FeS or reduction by S(II) and precipitation as UO<sub>2</sub>. This study investigates the fate of sorbed U(VI) in response to reductive dissolution of iron oxides by S(II). In batch experiments, S(II) is incrementally added to a lepidocrocite suspension with preadsorbed U(VI). The S(II) concentration decreases with time due to oxidation by Fe(III), causing an increase in Fe (II) concentration. With every S(II) addition, an instantaneous increase in dissolved uranium concentration is observed. The concentration then gradually decreases for about one hour, but remains at a higher level than before sulfide amendment. This could be due to the competition between U(VI) and Fe(II) for surface sites, or to the transformation of lepidocrocite into FeS and lower affinity of FeS surfaces for U(VI) than lepidocrocite. Preliminary results regarding possible complexation of uranium with S(II) in solution, show that this process is negligible. XANES spectra imply a gradual but partial transformation of U(VI) into U(IV) during of the experiments. Results indicate that most likely the formation of FeS might be required for U reduction.