



Oxidation State Measurements of the Uranium M-alpha Peak Using a Microcalorimeter Array Coupled to a Scanning Electron Microscope

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We report the measurement of X-ray fluorescence (XRF) peak shifts associated with the M-alpha peak at nominally 3.171 keV indicative of the oxidation state of the uranium for four different samples covering three different oxidation states without the use of a synchrotron source. The oxidation state of an element defines the chemistry and molecular bonding of that element. Typical X-ray measurements of an elemental oxidation state occur within the confines of a synchrotron source. It is the high spectral resolving power of the synchrotron which provides the fine spectral selectivity required to excite the elemental energy levels generating X-ray absorption features indicative of the oxidation state. In general, the X-ray absorption measurements are characteristically referred to X-ray absorption near edge structure (XANES) which defines the characteristic oxidation state of the element under study. A high resolution microcalorimeter coupled with a scanning electron microscope has provided a transformative approach to oxidation state measurements. An eight-pixel microcalorimeter array with a resolution of about 5 eV was used to detect the X-ray fluorescence emission of the four different specimens using an electron beam excitation potential of 7 keV and an applied filament current of 50 micro-Amperes. This excitation was sufficient to generate M-alpha XRF emission from pure uranium compounds with oxidation states of 0, +4 and +6. In each case the M-alpha XRF line was measured for

500 s. The M-alpha spectral peaks from each specimen were fitted with a Gaussian and the peak centroid of the fit recorded. The shift of the peak center measured for 0 and +4 was ~ 1 eV and for 0 and +6 ~ 3 eV. Multiple measurements verified the reproducibility of the spectra. These results demonstrate the feasibility of oxidation state measurements without the use of a synchrotron source and conducting such measurements in a regular laboratory.

