Full Chemical Composition Characterization: The Promise of Microcalorimeter Detectors for X-ray Spectroscopy

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Microcalorimeter detectors are still a novelty in X-ray spectrometry, although they have been around for several decades. While microcal detectors are studied and developed in laboratories they have not yet been implemented in commercial X-ray spectrometers. This presentation will focus on the use of a commercial TES (transition edge sensor) array microcal detector demonstrating the potential of microcal detectors for full chemical compositional analyses. The primary advantage of the microcal detector is being able to determine the oxidation state of the analyte element of interest. This determination has been the province of synchrotron measurements for many years due to the high spectral resolution of the synchrotron probes. The use of synchrotrons for oxidation state and elemental binding can be considered nearly routine, the access is limited to once or twice a year, hence making routine implementation of XANES and EXAFS measurements not feasible. The ability to measure oxidation state of an analyte as well as determining the elements bound to the analyte in the laboratory offer a powerful potential capability in materials characterization. Three different elemental analytes will be presented including iron, uranium and carbon. Different molecular forms of these elements have been measured using a microcal detector on a commercial scanning electron microscope. The measured spectra were processed to identify the analyte peak position which is a measure of the oxidation state. In addition, the analyte peak shape contains information on the binding of the analyte to other elements. With appropriate molecular modeling it is feasible to obtain the molecular formula of the material being probed. Figure 1 illustrates the iron La peak in two different iron compounds with the same +3 oxidation state. While not yet developed, the ability to elucidate the chemical nature of the iron species is clearly apparent in the overlay of these two spectra. As development of the microcal detector to lower resolution and higher array size, these new capabilities for materials characterization hold tremendous promise.

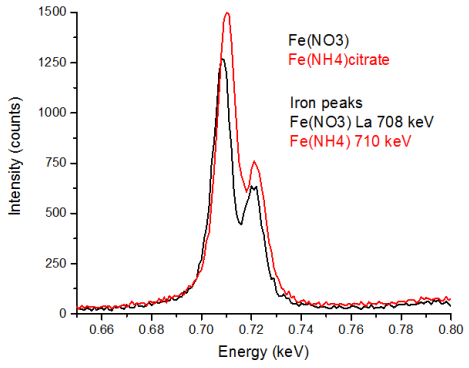

Figure 1. Overlay of Fe Lα and Lβ peaks around 700 eV for two different iron compounds in +3 oxidation state.