Improved Benchtop XAFS and XES for Materials Chemistry, Actinide Studies, and Electrochemical Research

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Advanced x-ray spectroscopies allow the direct and element-specific interrogation of local electronic structure, yet their scientific impact is necessarily constrained by access limitations. This is particularly evident for x-ray absorption fine structure (XAFS) and x-ray emission spectroscopy (XES) techniques. Here, synchrotron x-ray facilities help push the forefront of science, yet often provide sole access to these techniques and necessarily exclude projects that require routine analytical characterization or rapid feedback for prototyping applications or industrial process control.

Over the last four years, our group at the University of Washington has been developing several new families of lab-based instruments to expand the accessibility of advanced x-ray spectroscopies.[1-5] We now report on the construction of a new lab-based spectrometer employing four primary design advances. First, the source and detector stages are rotated relative to the direction of travel of a spherically-bent crystal analyzer (SBCA). This choice allows for minimal motion of the SBCA and extended angular range, resulting in decreased air attenuation and the possibility for extended XAFS (EXAFS) studies, respectively. Second, mechanical coupling between the source and SBCA stage enables passive tracking for improved stability. Third, the design integrates a 100 W x-ray tube source into the previous synchronous scanning methodology. This improvement upon the original 10 W source increases sensitivity and decreases measurement time. Finally, the instrument abandons the two-axis tilt procedure in favor of a tilt-free optic alignment advocated elsewhere.[4] This final design decision expedites optic exchange and is especially helpful for weak XES signals.

From the standpoint of instrument performance and materials inquiry, various spectra are presented establishing our current capabilities as well-suited for materials chemistry research. XANES and EXAFS comparisons of metal foils and simple chemistries demonstrate excellent agreement between the lab-based instrumentation and its synchrotron counterpart. Sensitivity to a metal’s ligand environment is established via valence-to-core XES results from a suite of transition metal oxides. The applicable energy range and versatility of the instrument is confirmed by spectra of lanthanide and actinide compounds. Finally, measurements of energy storage materials are presented as an example of the potential real-world impact of lab-based spectrometers.

Here, we present a survey of the above instrument development and its relevance to numerous problems, including battery electrode materials, battery electrolytes, transition-metal based nanoparticle compounds, and lanthanide and actinide chemistry. The present work advocates for laboratory-based instrumentation as a promising avenue for high-access and high-throughput x-ray absorption spectroscopy analysis suitable for both academic and industrial research programs.

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