New, smaller X-ray spectrometers developed

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X-ray absorption spectroscopy (XAS) and X-ray emission spectroscopy (XES) are useful tools for characterizing numerous properties of a given material. They can give elemental composition, metal oxidation states, spin state, orbital occupancy and more. A drawback is that these types of measurements have historically needed synchrotron light sources to generate high flux X-ray beams and high-resolution monochromators to provide a monoenergetic excitation source. Synchrotrons are building-sized facilities (often operated in the United States by the DOE) and have the scheduling complexities and logistical issues that accompany any large-scale research facility. There are added challenges for scientists wishing to analyze radioactive samples such as plutonium or other actinides.

These limitations are now mitigated by a small, “portable” X-ray emission and absorption spectrometers developed by scientists at Los Alamos National Laboratory and the University of Washington. Two of these new X-ray spectrometers were commissioned in 2018. These instruments provide information about the oxidation state and local electronic structure without the need to transport samples off-site to a synchrotron. Moreover, they can (and have) been used to analyze samples containing plutonium as well as more conventional samples, like indium phosphide quantum dots.

An XES instrument works particularly well in the tender X-ray regime (2–5 keV) and a hard X-ray XES/XAS instrument that operates in the 5–19 KeV range. The tender X-ray XES instrument has been used in numerous piolet studies, such as characterizing oxidation states of phosphorous in indium phosphide quantum dots and orbital mixing in uranium containing molecules. This instrument’s small size also provides the opportunity for integration into controlled-gas glove box systems for analysis of air-sensitive materials. The high-energy XAS/XES instrument has been used to characterize oxidation states and electronic configurations for elements ranging from V to Pu. The sensitivity for the XES measurements is impressive and the instrument is currently being used to characterize speciation of microscopic impurities in numerous bulk materials.

Both instruments operated efficiently with an unfocused X-ray source and consequently a large beamspot on the sample, which decreases cost and increases ease of use. The development of these two instruments was enabled by recent advances in commercially available specialized crystal analyzers and position-sensitive detectors and was sponsored by the Department of Energy, Basic Energy Sciences.

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