EMSL

Capabilities Series

Spectroscopy and Diffraction

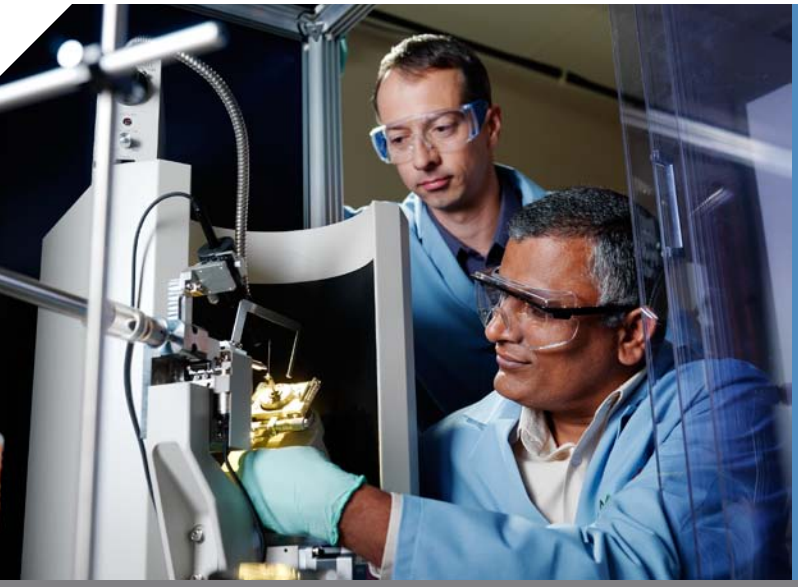
EMSL houses a suite of spectroscopy and diffraction instruments that allow users to study the structure and composition of solid, liquid, and gas samples with molecular resolution. EMSL's spectroscopy and diffraction technology contributes to an unparalleled collection of capabilities designed to support an integrated experimental and computational approach to research challenges. These instruments and expertise enable novel, fundamental research in EMSL's Science Themes of Biological Interactions and Dynamics, Geochemistry/Biogeochemistry and Subsurface Science, and Science of Interfacial Phenomena. Specific research topics include:

- **Material science and catalysis** – providing molecular-level characterizations of materials like catalysts and iron-doped glasses and tailoring these materials for specific functions
- **Environmental remediation** – understanding factors that impact contaminant fate and transport, such as the presence of iron oxides and microbial communities, for the cleanup of subsurface environments
- **Energy** – making strides toward alternative and renewable energy sources as well as the capture and sequestration of greenhouse gases
- **Biology** – relating the structure and function of proteins with critical cellular roles
- **Fundamental science development** – moving research forward by enhancing experimental techniques through integration with computational tools, and validating models through experiment.

CAPABILITY DETAIL

EMSL offers scientific users:

- **Electron spectroscopy** – Auger, high spatial, and energy resolution X-ray photoelectron spectroscopy
- **Electron backscatter diffraction** – electron diffraction (combined with focused ion beam slicing) for three-dimensional phase reconstruction of microstructures
- **Ion/molecular beam spectroscopy** – energetic ion beam (~ MeV) analyses of complex materials, including thin films, radiation effects, aerosols, and molecular beam scattering (~10 eV – 3000 eV) studies of surfaces
- **Mössbauer spectroscopy** – several systems with variable temperature capability for conversion electron Mössbauer spectroscopy
- **Optical spectroscopy** – advanced cryogenic, time-resolved fluorescence spectroscopy, second harmonic/sum frequency generation, stopped-flow absorbance, Fourier transform infrared spectroscopy, Fourier transform Raman spectroscopy, and confocal Raman microscopy tools for biology, radiochemistry, and catalysis studies
- **X-ray diffractometers** – multiple systems such as computed tomography, spatially resolved microbeam, and specialized thin film diffractometers for analysis of powder, epitaxial thin film, geologic cores, and other challenging sample types
- **Nuclear Magnetic Resonance** – a suite of 10 NMR spectrometers up to 900 MHz with specialized probes for solid, liquid, and thin film studies.



WHY SPECTROSCOPY AND DIFFRACTION AT EMSL?

- EMSL offers a wide variety of cutting-edge spectroscopy and diffraction tools for *in situ* molecular-level characterization of chemical speciation and structure on surfaces and interfaces.
- EMSL's complementary spectroscopy and diffraction tools, techniques, and staff expertise come together to allow researchers to collect holistic, systems-wide observations.
- Researchers are encouraged to integrate spectroscopy and diffraction instruments with EMSL's computational tools—an interaction that brings data to life with advanced modeling, and validates or improves models with enhanced data.

ABOUT EMSL

EMSL, a U.S. Department of Energy national scientific user facility located at Pacific Northwest National Laboratory, provides integrated experimental and computational resources for discovery and technological innovation in the environmental molecular sciences to support the needs of DOE and the nation.

EMSL's distinctive focus on integrating computational and experimental capabilities as well as collaborating among disciplines yields a strong, synergistic scientific environment. Bringing together experts and an unparalleled collection of state-of-the-art instruments under one roof, EMSL has helped thousands of researchers use a multidisciplinary, collaborative approach to solve some of the most important and complex national scientific challenges in energy and environmental sciences.

To learn more about EMSL, the science conducted at EMSL, as well as the instruments and expertise available to users, visit www.emsl.pnl.gov.

BECOME AN EMSL USER

Researchers are invited to access the world-class capabilities and collaborate with the internationally recognized experts at EMSL via its peer-reviewed proposal process. To submit a proposal, follow the five steps outlined on the EMSL website (www.emsl.pnl.gov) under User Access. Current and potential EMSL users are encouraged to respond to Calls for Proposals, which are announced each spring. However, unique research proposals that fall outside the Calls for Proposal focus may be submitted at any time.

Applicants are encouraged to submit proposals for use of EMSL's capabilities with an emphasis on integrating computational and experimental tools. In general, most users whose open research proposals are accepted may use EMSL resources free of charge. Open research is loosely defined as science and engineering research for which the resulting information is published and shared broadly within the scientific community.

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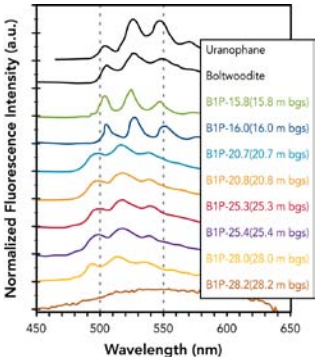
SPEC/DIFF RESEARCH HIGHLIGHTS

Tracking Uranium's Travels

Using time-resolved, laser-induced fluorescence spectroscopy (TRLIFS), EMSL users conducted macroscopic and spectroscopic investigations on contaminated Hanford Site vadose zone sediments. They discovered several different uranium surface phases in the contaminated soil, indicating that the uranium release mechanism could be very complicated. This signaled the need for a detailed sediment characterization to estimate uranium fate and transport in the vadose zone. Capabilities like TRLIFS provide experimental data that reveal how environmental contaminants behave at a fundamental level. When combined with further modeling tools, these data allow researchers to predict contaminant behavior, opening the door to new cleanup strategies.

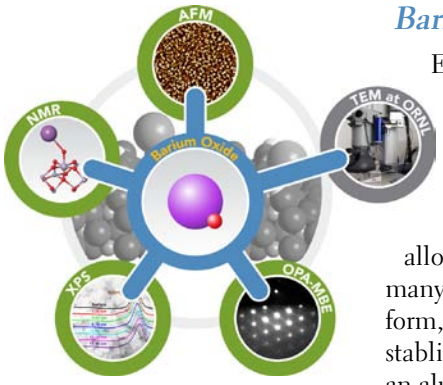


Uranyl fluorescence spectra vary from boltwoodite/uranophane-type secondary precipitates to surface-adsorbed U(VI) phases with blue-shifted, less-resolved spectral features as the sediment depth increases.

EMSL Users: Pacific Northwest National Laboratory, Brookhaven National Laboratory

Um *et al*. 2009. *Environmental Science & Technology* 43(12):4280-4286.

Barium Oxide from Every Angle



Researchers applied several spectroscopy and diffraction capabilities at EMSL—combined with NMR and electron microscopy—to study a BaO catalyst system.

EMSL's spectroscopy and diffraction tools were integrated with 900-MHz nuclear magnetic resonance and electron microscopy to study barium oxide (BaO), a catalyst that grabs and stores nitrogen oxide (NO_x) to help control emissions. EMSL capabilities allowed researchers to study BaO catalysts from many angles, including how BaO nanoparticles form, BaO nanoparticle characteristics on an yttria-stablized zirconia support, and BaO binding on an aluminum oxide (Al_2O_3) support. This rounded characterization was made possible by the full spectrum of traditional and non-traditional surface science capabilities under the EMSL roof.

These included Oxygen-plasma-assisted molecular beam epitaxy for high-quality BaO thin-film growth, high-resolution infrared spectroscopy measurements to identify vibrational modes, *ex situ* high-resolution x-ray diffraction for structural analysis, and high-resolution x-ray photoelectron spectroscopy to characterize chemical states of elements. Deep, systems-scale study of catalysts leads to better pollution prevention.

EMSL Users: Pacific Northwest National Laboratory, Oak Ridge National Laboratory, Hanbat National University, and Nanjing Normal University

Kwak *et al*. 2007. *Journal of Catalysis* 251:189-194.

Nachimuthu *et al*. 2009. *Journal of Physical Chemistry* C 113 (32):14324-14328.

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