



900-MHz NMR: Accelerating Scientific Discovery



Introduction

When the U.S. Department of Energy's (DOE's) Office of Biological and Environmental Research approved the development and purchase of the world's first 900-MHz NMR (nuclear magnetic resonance) spectrometer in 1992, the highest magnetic field available was 750 MHz. DOE's decision and the ultimate success of its 900-MHz NMR spectrometer, which recently saw its five-year anniversary of operation at EMSL, catalyzed the development of a new generation of ultrahigh-field NMR spectrometers worldwide.

Building new technology

Building the magnet for the 900-MHz NMR spectrometer brought engineering challenges. Can the superconducting wire that composes the NMR magnet handle the stresses imposed by the higher magnetic field? How should that wire be joined to make a continuous superconductor several hundred kilometers long? The complexity of the construction of the 900-MHz NMR spectrometer necessarily delayed delivery of the system until 2002; it became operational for users in 2004. In the meantime, the manufacturer provided EMSL with two 600-MHz NMR spectrometers, followed by a significant discount on an 800-MHz NMR spectrometer, helping EMSL keep its state-of-the-art instrument edge. Thus, through the 900-MHz NMR project, EMSL acquired a suite of spectrometers with varying field strengths (600 MHz, 800 MHz, and 900 MHz) to suit the myriad of needs of the EMSL user community.

Opening new doors

The never-before-available high resolution and high sensitivity of EMSL's 900-MHz NMR spectrometer enabled new kinds of experiments. The ultrahigh-field enhanced characterization of liquid-state biological samples; enabled better characterization of solid-state insoluble proteins; and for the first time, allowed measurements on a large class of nuclei in the periodic table that are described as quadrupolar in nature. Such nuclei are the building blocks for catalysts and biomaterials such as metalloproteins.

Influencing collaborators

In addition to advancing many fields of fundamental research, the high-quality data yielded by EMSL's 900-MHz NMR spectrometer unexpectedly influenced EMSL users to establish ultrahigh-field facilities at their home institutions. For example, following receipt of outstanding results from EMSL's 900-MHz NMR resources, former EMSL users from the University of Alberta and University of British Columbia helped to establish Canada's first 900-MHz NMR facility housed in Ottawa and modeled after EMSL.

Illustrating the scientific benefits

The pages that follow illustrate the benefits of DOE's and EMSL's investment in NMR resources, including the suite of spectrometers garnered through the 900-MHz NMR project and EMSL's collection of specialized sample



probes, many of which are custom designed and built in-house. This investment has fostered novel research in:

- Energy – making strides toward alternative energy sources
- Environmental research – understanding contaminant fate and transport and remediating unhealthy environments
- Catalysis – helping understand catalysts at the molecular level to then tailor them for specific functions
- Biology – relating the structure and function of proteins with critical cellular roles
- Capability development and integration – enhancing spectral interpretation with computational tools and enabling new NMR measurements via novel capabilities.

Advances in these research areas enabled by the 900-MHz project are evidenced by more than 130 publications, a significant number of which appeared in top-tier journals, including *Science*, *Nature*, and the *Proceedings of the National Academy of Sciences* (see pages 14-23). Contributors to those publications include many Distinguished Participants (see page 14).

Your research can benefit from EMSL's ultrahigh-field NMR resources. Submit a proposal to become an EMSL user at www.emsl.pnl.gov/access.

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Energy

Optimizing alternative energy sources

New details about a system with applications to hydrogen storage and lithium ion battery technologies were revealed using EMSL's suite of NMR instruments. Researchers used high-energy ball milling to create lattice defects in a lithium-nitrogen-hydrogen (Li-N-H) system consisting of lithium hydride (LiH) and lithium amide (LiNH₂), thus improving both the hydrogen uptake and release kinetics. 900-MHz NMR spectroscopy was applied to elucidate, for the first time, the electronic structure and the details of the chemistry of the activated L-N-H system. ⁶Li on the 900-MHz NMR spectrometer distinguished LiH and LiNH₂ molecules from sample impurities and revealed a spectral peak never before observed in the Li-N-H system. The new peak corresponds to the presence of lattice defects and thus enhanced hydrogen release from the Li-N-H system as observed using ¹H magic-angle spinning (MAS) NMR spectroscopy. Further, researchers found that ball milling at liquid nitrogen temperature causes hydrogen to be released from the system more quickly than ball milling at room temperature. Such studies help researchers optimize the manufacture of alternative energy sources.

Featured user publication

Hu JZ, Kwak JH, Yang Z, Osborn W, Markmaitree T, Shaw LL. 2008. "Investigation of Mechanical Activation on Li-N-H Systems Using ⁶Li MAS NMR at Ultra-High Field." *Journal of Power Sources* 182: 278-283.

Related user publications

Hu JZ, JH Kwak, Z Yang, X Wan, LL Shaw. 2009. "Direct Observation of Ion Exchange in Mechanically Activated LiH+MgB₂ System using Ultra-High Field ⁶Li, ¹¹B and ²⁵Mg Magic Angle Spinning NMR Spectroscopy." *Physical Review Letters* (In Press).

Osborn W, T Markmaitree, LL Shaw, JZ Hu, JH Kwak, Z Yang. 2008. "Low Temperature Milling of the LiNH₂ + LiH Hydrogen Storage System." *International Journal of Hydrogen Energy* (In Press).

Creating a solid (hydrogen) path to energy

Establishing hydrogen as an alternative energy source relies on developing safe and efficient hydrogen storage materials, and EMSL's 900-MHz NMR spectrometer is advancing solid-state hydrogen storage research to this end. Promising solid-state hydrogen storage materials must be stable at room temperature and pressure as well as release hydrogen at low temperatures. Researchers built upon previous research using one such promising solid-state hydrogen storage candidate, ammonia borane (NH₃BH₃). Because NH₃BH₃ releases the contaminant borazine along with hydrogen, researchers modified NH₃BH₃ to yield lithium amidoborane (LiNH₂BH₃) and sodium amidoborane (NaNH₂BH₃). LiNH₂BH₃ and NaNH₂BH₃ were found to release considerable amounts of hydrogen just as NH₃BH₃ does, with the lithium-based compound releasing more than the sodium-based compound. Further, neither compound released borazine, and both compounds released hydrogen at even lower temperatures than NH₃BH₃. Using the 900-MHz NMR spectrometer equipped with a custom-made (designed and built in-house) high-temperature probe, researchers elucidated the structural details of the Li-based



LiNH₂BH₃ molecules (Li = yellow, N = blue, B = pink, and H = white).



EMSL's NMR resources enable researchers to characterize materials in their native environments. For example, reactions that occur at high temperatures can be measured with specialized, high-temperature probes. EMSL's NMR probe capabilities enable scientists to differentiate the spectral features of surface species from the bulk sample.

compound, helping them understand how to better design solid-state hydrogen storage materials.

Featured user publication

Xiong Z, CK Yong, G Wu, P Chen, WJ Shaw, AJ Karkamkar, T Autrey, MO Jones, SR Johnson, P Edwards, and WIF David. 2008. "High-Capacity Hydrogen Storage in Lithium and Sodium Amidoboranes." *Nature Materials* 7(2):138-141.

Related user publication

Xiong Z, YS Chua, G Wu, W Xu, P Chen, WJ Shaw, A Karkamkar, J Linehan, T Smurthwaite, and T Autrey. 2008. "Interaction of Lithium Hydride and Ammonia Borane in THF." *Chemical Communications* 43:5595-5597.

Tailoring hydrogen storage materials

Integrated experimental tools at EMSL including NMR spectroscopy are facilitating advances in hydrogen storage materials. Researchers used EMSL resources to characterize ammonia borane (NH_3BH_3) embedded in a carbon cryogel. Though promising as a hydrogen storage material, NH_3BH_3 in its pure form is incompatible with polymer electrolyte membrane fuel cells because it releases borazine, a volatile byproduct that will poison the fuel cell. In addition, it releases hydrogen at a temperature higher than the operating temperature of the fuel cell. But embedding NH_3BH_3 in a carbon cryogel support suppressed the release of borazine and improved the hydrogen release kinetics of NH_3BH_3 such that it released hydrogen at a lower temperature. Further, the combination of Fourier transform infrared spectroscopy and NMR spectroscopy revealed a new reaction product in the carbon cryogel-supported NH_3BH_3 compared to NH_3BH_3 alone. Detailing the kinetics of hydrogen storage material candidates will allow researchers to modify promising materials to achieve the highest performance possible.

Featured user publication

Sepehri S, A Feaver, WJ Shaw, CJ Howard, Q Zhang, T Autrey, and G Cao. 2007. "Spectroscopic Studies of Dehydrogenation of Ammonia Borane in Carbon Cryogel." *Journal of Physical Chemistry B* 111(51): 14285-14289.

Related user publication

Feaver AM, S Sepehri, PJ Shamberger, AC Stowe, T Autrey, and G Cao. 2007. "Coherent Carbon Cryogel-Ammonia Borane Nanocomposites for Improved Hydrogen Storage." *Journal of Physical Chemistry B* 111(26):7469-7472.

Environment

Predicting contaminant fate and transport

New details about how the radioactive contaminants strontium and cesium interact with their environment were obtained using EMSL's NMR capabilities. ^{90}Sr and ^{137}Cs compose the bulk of the radioactivity released at DOE's Hanford Site in Richland, Washington. To study the geochemical processes that influence strontium and cesium migration, researchers analyzed sediments from the Hanford Site using a multifaceted approach, which was enabled by EMSL's 900-MHz NMR spectrometer, custom probe technologies, and pulse sequence methods. The studies offered molecular-level insight about the uptake, distribution, and speciation of strontium and cesium—aspects of their chemistry and kinetics that directly affect their transport, sequestration, and remediation. Understanding the geochemistry of radioactive contaminants helps researchers predict their fate and transport as well as treat contaminated environments more effectively.

Featured user publication

Chorover J, S Choi, P Rotenberg, RJ Serne, N Rivera, CR Strepka, A Thompson, KT Mueller, and PA O'Day. 2008. "Silicon Control of Strontium and Cesium Partitioning in Hydroxide-Weathered Sediments." *Geochimica et Cosmochimica Acta* 72(8):2024-2047.

Related user publications

Bowers GM, MC Davis, R Ravella, S Komarneni, and KT Mueller. 2007. "NMR Studies of Heat Induced Transitions in Structure and Cation Binding Environments in a Strontium Saturated Swelling Mica." *Applied Magnetic Resonance* 32:595-612.

Bowers GM, AS Lipton, and KT Mueller. 2006. "High-Field QCPMG NMR of Strontium Nuclei in Natural Minerals." *Solid State NMR* 29:95-103.

Bowers GM, R Ravella, S Komarneni, and KT Mueller. 2006. "NMR Study of Strontium Binding by a Micaceous Mineral." *Journal of Physical Chemistry B* 110(14):7159-7164.

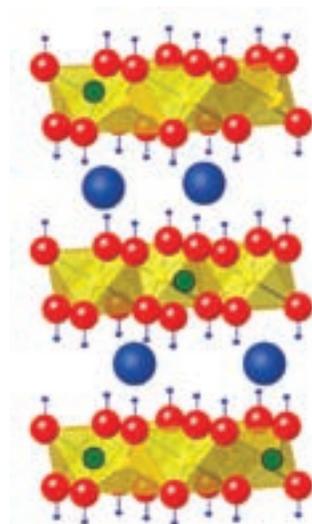
Crosson GS, S Choi, J Chorover, MK Amistadi, PA O'Day, and KT Mueller. 2006. "Solid-State NMR Identification and Quantification of Newly Formed Aluminosilicate Phases in Weathered Kaolinite Systems." *Journal of Physical Chemistry B* 110(2):723-732.

Tailoring remediation materials

Layered double hydroxides (LDHs) are versatile materials with many applications, including environmental remediation. Structurally, they are a framework of cation-containing metal hydroxide sheets separated by anions and water molecules.

Functionally, anions in the LDH interlayer are exchanged for anions in another chemical or material; for example, LDHs can sequester the toxic chromate from contaminated environments. Ultimately, the anion exchange ability and selectivity of LDHs are dictated by the number and arrangements of the different cations in the sheets of the structure. To gain insight into cation distribution in LDHs, researchers used EMSL's 900-MHz NMR spectrometer and an EMSL-built ^{25}Mg MAS NMR probe to study three analogs of a naturally occurring LDH, hydrocalcite. Relating LDH structure and function will enable new approaches to tailor LDHs that could lead to

LDHs consist of hydroxide sheets containing cations (green and yellow) separated by anions (blue) and structural water molecules.





The high resolution and high sensitivity of EMSL's 900-MHz NMR capability greatly increases researchers' efficiency. EMSL user Karl Mueller stated in a *DOE Pulse* article, "Some of the data we can get in three days at EMSL would have taken more than two years to acquire with the equipment at Penn State." The ability to quickly characterize difficult-to-measure nuclei, such as the strontium systems studied by Mueller, has set the stage for future research with strong environmental impact.

refined materials for established applications as well as new materials for unexplored applications.

Featured user publication

Sideris PJ, UG Nielsen, Z Gan, and CP Grey. 2008. "Mg/Al Ordering in Layered Double Hydroxides Revealed by Multinuclear NMR Spectroscopy." *Science* 321(5885):113-117.

Demystifying denitrification

Researchers used EMSL's NMR resources to yield the first detailed structure of an accessory protein needed for the environmentally relevant process of denitrification. Via denitrification, bacteria reduce nitrate and nitrite to nitrogen gas, thus cycling nitrogen derived from biomass back to the atmosphere. The metallo-enzyme nitrous oxide reductase (N₂OR) performs the last step of denitrification, and it relies upon the copper-binding protein NosL for assembly. The sequence of NosL could not be used to gain insight into its cellular role because it is unlike other proteins with known function. So, NMR spectroscopy was employed to discern its structure-function relationship, thus establishing the groundwork for understanding its role in denitrification at the molecular level. Importantly, NMR revealed that NosL has a unique structure with a new configuration for binding and transporting metal atoms in cells. It likely carries copper atoms through the cell to N₂OR. Denitrification protein studies may be applied to design strategies to deal with N₂O emissions in the atmosphere and excess nitrogen in groundwater, soils, and wetlands contaminated with fertilizer runoff.

Featured user publication

Taubner LM, MA McGuirl, DM Dooley, and V Copie. 2006. "Structural Studies of Apo NosL, an Accessory Protein of the Nitrous Oxide Reductase System: Insights from Structural Homology with MerB, a Mercury Resistance Protein." *Biochemistry* 45(40):12240-12252.

Catalysis

Building better molybdenum catalysts

New molybdenum species and the active centers for catalytic reactions in molybdenum-based catalysts have been identified using EMSL's 900-MHz NMR spectrometer. Molybdenum, which is important for catalytic reactions, such as converting methane to value-added chemicals, was not easily studied before the advent of ultrahigh-field NMR spectroscopy. Scientists used the 900-MHz NMR spectrometer to study for the first time how a class of molybdenum-based catalysts called Mo/HZSM-5 facilitate the transition of methane to aromatic compounds, a process known as methane dehydroaromatization (MDA). The research team examined four MDA catalysts composed of different amounts of molybdenum before and after reacting with methane. Prior to the reaction, ^{95}Mo spectra from the 900-MHz NMR revealed different molybdenum species on the catalysts, including a new type of species. By comparing spectra before and after the reactions, scientists identified the active center for the MDA reaction. Electronic structure observations such as these are a solid step toward building tailored molybdenum-based catalysts, such as those that could efficiently convert methane to the alternative energy source, hydrogen.

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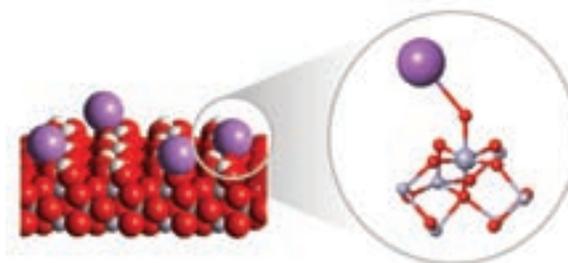
Zheng Z, D Ma, X Bao, JZ Hu, JH Kwak, Y Wang, and CHF Peden. 2008. "Direct Observation of the Active Center for Methane Dehydroaromatization Using an Ultra-High Field ^{95}Mo NMR Spectroscopy." *Journal of the American Chemical Society* 130(12):3722-3723.

Related user publication

Hu JZ, JH Kwak, Y Wang, CHF Peden, Z Heng, D Ma, and X Bao. 2009. "Studies of the Active Sites for Methane Dehydroaromatization Using Ultrahigh-Field Solid-State ^{95}Mo NMR Spectroscopy." *Journal of Physical Chemistry C* 113(7): 2936-2942.

Preventing pollution

Researchers used ^{27}Al MAS spectroscopy on the 900-MHz NMR spectrometer to conduct detailed studies of a promising system for preventing pollution from diesel engines: the γ -alumina support, $\gamma\text{-Al}_2\text{O}_3$, and the catalyst, barium oxide (BaO). γ -alumina materials are important because they act as catalysts and as a support that binds with other materials to form new catalysts. In the emissions control system described here, $\gamma\text{-Al}_2\text{O}_3$ supports BaO, and BaO binds to NO_x vehicle emissions products. The chemical properties of γ -alumina materials are such that it is difficult to study them with traditional surface techniques. But EMSL's 900-MHz NMR spectrometer allowed researchers to generate the highest-resolution spectra ever obtained for $\gamma\text{-Al}_2\text{O}_3$ and to observe, for the first time and quantitatively, how the support interacts with a catalyst at the atomic scale. The data from EMSL's 900-MHz NMR system indicate that penta-coordinated aluminum atoms sit on the surface of $\gamma\text{-Al}_2\text{O}_3$ and that BaO binds to this specific atomic arrangement. Understanding catalysts at this level of detail is a solid step toward the ultimate goal of tailoring them for specific functions.



BaO (Ba = purple and O = red) binds to penta-coordinated aluminum atoms (grey) on the surface of $\gamma\text{-Al}_2\text{O}_3$.

Featured user publication

Kwak JH, JZ Hu, DH Kim, J Szanyi, and CHF Peden. 2007. "Penta-Coordinated Al^{3+} Ions as Preferential Nucleation Sites for BaO on $\gamma\text{-Al}_2\text{O}_3$: An Ultra-High-Magnetic Field ^{27}Al MAS NMR Study." *Journal of Catalysis* 251:189-194.



Ultrahigh-field NMR spectroscopy and EMSL's strong focus on locally designed and built probe technologies allow researchers to explore new territory, such as studying molybdenum nuclei and distinguishing between surface and bulk aluminum atoms. Such capabilities, when added to the spectrometer portfolio, enable significant scientific advancements in catalysis research.

Related user publication

Kwak JH, JZ Hu, A Lukaski, DH Kim, J Szanyi, and CHF Peden. 2008. "The Role of Penta-Coordinated Al^{3+} Ions in the High Temperature Phase Transformation of $\gamma-Al_2O_3$," *Journal of Physical Chemistry C* 112:9486-9492.

Converting crude oil into gasoline

The first steps toward further improving the efficiency of zeolite catalysts that convert crude oil to gasoline and other petroleum products were enabled by high-field NMR spectroscopy at EMSL. Zeolites have very open frameworks, containing molecular-level systems of cavities and channels that control the uptake of organic molecules in terms of shape and size selectivity. Though zeolite catalysts are active alone, they perform even more efficiently as nanozeolite/mesoporous aluminosilicate composites made by applying a zeolitic nanocluster coating to aluminosilicate materials. Because instruments of lesser resolution were ineffective for better understanding the structure of these composites, researchers used high-field ^{27}Al MAS and multiple quantum MAS (MQMAS) NMR at EMSL for characterization. These sophisticated NMR techniques were used to detect the zeolitic nanocrystals on and to study the aluminum environments in composite materials. Understanding the molecular-level structure of these materials important to the oil refining industry is critical for implementing future structural improvements to yield greater catalytic efficiency.

Featured user publication

Do T, A Nossov, M Springuel-Huet, CM Schneider, JL Bretherton, CA Fyfe, and S Kaliaguine. 2004. "Zeolite Nanoclusters Coated onto the Mesopore Walls of SBA-15." *Journal of the American Chemical Society* 126(44):14324-14325.

Related user publications

Fyfe CA, JL Bretherton, and LY Lam. 2001. "Solid-State NMR Detection, Characterization, and Quantification of the Multiple Aluminum Environments in US-Y Catalysts by Al-27 MAS and MQMAS Experiments at Very High Field." *Journal of the American Chemical Society* 123(22):5285-5291.

Fyfe CA, JL Bretherton, and LY Lam. 2000. "Detection of the 'Invisible Aluminium' and Characterisation of the Multiple Aluminium Environments in Zeolite USY by High-Field Solid-State NMR." *Chemical Communications* 17:1575-1576.

Biology

Studying medically relevant protein complexes

EMSL's high-field NMR resources are helping researchers understand the assembly of multi-protein (as many as five) complexes involving the tumor suppressor protein linked to breast cancer, BRCA1. BRCA1 plays a role in ubiquitin (Ub) transfer, a critical cell-signaling pathway whereby the protein Ub is covalently attached to other proteins. The ubiquitination process is dependent on three kinds of enzymes, denoted E1, E2, and E3. BRCA1 is a member of the E3 family, and its enzymatic activity is increased upon binding to another protein, BARD 1. The BRCA1-BARD1 complex in turn interacts with the E2 family member UbcH5, which has the unusual property of interacting with Ub covalently at its active site and *noncovalently* at a surface far from its catalytic center. Using a combination of 900-MHz NMR spectroscopy, specialized “edited-filtered” pulse-sequence techniques, and cryoprobe technology, researchers determined the structure of the UbcH5-Ub noncovalent complex. They found that the noncovalent interaction leads to activated UbcH5-Ub self-assembly, which in turn is essential for synthesis of BRCA1-directed poly-Ub chains using UbcH5. NMR-enabled experiments such as these offer invaluable insight into the molecular mechanisms of medically relevant proteins.

Featured user publication

Brzovic P, AV Lissounov, D Christensen, DW Hoyt, and RE Klevit. 2006. “A UbcH5/Ubiquitin Noncovalent Complex is Required for Processive BRCA1-Directed Ubiquitination.” *Molecular Cell* 21(6):873-880.

Related user publications

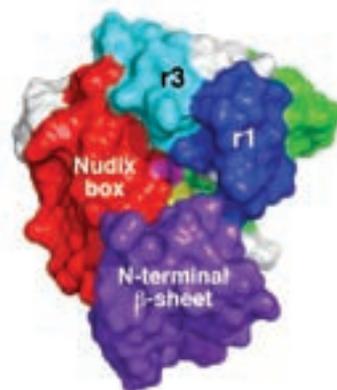
Fox DN, I Le Trong, P Rajagopal, P Brzovic, RE Stenkamp, and RE Klevit. 2008. “Crystal Structure of the BARD1 Ankyrin Repeat Domain and its Functional Consequences.” *Journal of Biological Chemistry* 283(30):21179-21186.

Brzovic P, JR Keeffe, H Nishikawa, K Miyamoto, D Fox, M Fukuda, T Ohta, and RE Klevit. 2003. “Binding and Recognition in the Assembly of an Active BRCA1/BARD1 Ubiquitin-Ligase Complex.” *Proceedings of the National Academy of Sciences of the United States of America* 100(10):5646-5651.

Brzovic PS, P Rajagopal, DW Hoyt, MC King, and RE Klevit. 2001. “Structure of a BRCA1/BARD1 Complex: A Heterodimeric RING-RING Interaction.” *Nature Structural Biology* 8(10):833-837.

Gaining insight into immunity

NMR studies conducted at EMSL have revealed new insights related to the unique capacity for the bacterium *Deinococcus radiodurans* to withstand desiccation, relatively high doses of ionizing radiation, and chemical agents that damage DNA. The biological mechanism for *D. radiodurans*' radiation resistance may be related to a large family of housekeeping proteins found in organisms from viruses to humans called Nudix hydrolases. EMSL's 900-MHz NMR spectrometer was used to identify specific locations within the molecular structure for the Nudix hydrolase, DR_0079. These studies revealed that DR_0079 prefers an unusual substrate. To illustrate, the Nudix protein MutT protects cells by converting dangerous promutagenic nucleoside triphosphates into safe nucleoside monophosphates—the former can be incorporated into DNA and RNA leading to cancer, and the latter cannot. But DR_0079 was



Surface structure of DR_0079 (205F) highlighting the Nudix box (red) and regions of the protein associated with substrate binding.



EMSL's investment in probe technologies, such as cryogenically cooled probes, coupled with ultrahigh-field NMR capabilities yield the high sensitivity and high resolution necessary to characterize large, medically relevant complexes. Further, custom-built ultra-low temperature (10K) solid-state NMR capabilities allow researchers to directly observe NMR resonances of metals, such as zinc and magnesium in metalloproteins. Under ambient conditions these experiments would be arduous, if not impossible.

found to convert nucleoside *diphosphates* instead of nucleoside triphosphates into nucleoside monophosphates. Understanding radiation resistance at the molecular level may lead to novel bioremediation methods and strategies that protect humans from the deleterious effects of ionizing radiation.

Featured user publication

Buchko GW, O Litvinova, H Robinson, AF Yakunin, and MA Kennedy. 2008. "Functional and Structural Characterization of DR_0079 from *Deinococcus radiodurans*, a Novel Nudix Hydrolase with a Preference for Cytosine (deoxy)Ribonucleoside 5'-di- and Triphosphates." *Biochemistry* 47:6571-6582.

Related user publications

Buchko GW, K McAteer, SS Wallace, and MA Kennedy. 2005. "Solution-State NMR Investigation of DNA Binding Interactions in *Escherichia coli* Formamidopyrimidine-DNA Glycosylase (Fpg): A Dynamic Description of the DNA/Protein Interface." *DNA Repair* 4(3):327-339.

Buchko GW, S Ni, SR Holbrook, and MA Kennedy. 2003. "H-1, C-13, and N-15 NMR Assignments of the Hypothetical Nudix Protein DR0079 from the Extremely Radiation-Resistant Bacterium *Deinococcus radiodurans*." *Journal of Biomolecular NMR* 25(2):169-170.

Understanding immune function

Chemokines play a major role in immune response and inflammation-related diseases such as asthma and arthritis. All known pox and herpes viruses encode proteins that inhibit chemokines, enabling them to influence immune function in their host. Researchers used EMSL's high-field NMR resources to determine the first known structure of a complex composed of a human chemokine belonging to a subfamily referred to as the CC family and a viral protein that inhibits it, vCCI. NMR data revealed that the inhibitor and chemokine form a complex in a 1:1 stoichiometry and that vCCI interferes with regions in the chemokine important to receptor binding and immune response. Structural elucidation of the complex also revealed key interactions that form the basis for the affinity and selectivity of vCCI toward certain CC chemokines. Insights such as these into the structural interactions of human and viral proteins are critical to developing therapeutic agents that modulate immune response.

Featured user publication

Zhang L, M DeRider, MA McCornack, C Jao, NG Isern, T Ness, R Moyer, and PJ Liwang. 2006. "Solution Structure of the Complex Between Poxvirus-Encoded CC Chemokine Inhibitor vCCI and Human MIP-1 β ." *Proceedings of the National Academy of Sciences of the United States of America* 103(38):13985-13990.

Capability Development and Integration

Characterizing titanium-based materials

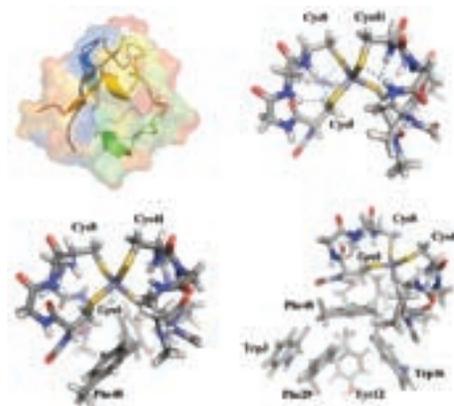
New experimental NMR techniques have allowed researchers, for the first time, to interpret the spectra of titanium-based materials with applications to radioactive waste immobilization and photovoltaic development. Before the advent of ultrahigh-field NMR, it was difficult to untangle the overlapping resonances of two naturally occurring isotopes, ^{47}Ti and ^{49}Ti , in titanium-based materials. However, combining EMSL's 900-MHz NMR spectrometer, probes custom-built at EMSL, and a novel pulse-sequence technique enabled researchers to collect and interpret complete spectra of the two isotopes in each of two titanium dioxide polymorphs, anatase and rutile. Complete characterizations of titanium-based materials are critical for structural studies of titanium-based ceramics and glasses that may be used for radioactive waste vitrification as well as of titanium-based materials with applications to alternative energy production.

Featured user publication

Larsen FH, I Farnan, and AS Lipton. 2006. "Separation of ^{47}Ti and ^{49}Ti Solid-State NMR Lineshapes by Static QCPMG Experiments at Multiple Fields." *Journal of Magnetic Resonance* 178(2):228-236.

Understanding metalloproteins

Researchers have gained new knowledge about metalloproteins—a class of proteins critical to many biological functions—by integrating EMSL's computational chemistry tool NWChem and its unique low-temperature, solid-state NMR capabilities at high magnetic fields. Coupling NWChem and NMR spectroscopy yielded a molecular model that describes the electronic environment of the metalloprotein motif, zinc coordinated to four cysteines, in the context of its structure and function. Once thought to only play a structural role, the zinc-cysteine motif is now known to play a reactive role as well; for example, it facilitates DNA repair. The team's method was tested using a protein important to the regulatory biological mechanism of electron transfer that was modified to contain zinc: zinc-substituted rubredoxin derived from the thermophile *Pyrococcus furiosus*. In future studies, similar sites with reactive roles will be compared and the molecular models refined to describe them. These integrated experiments are helping researchers discern what differentiates structural and reactive metalloprotein sites, allowing a deeper understanding of DNA repair.



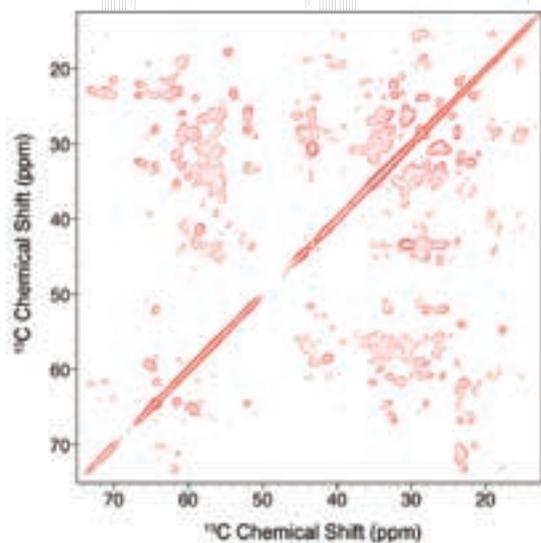
Structure of the zinc- and cysteine-containing region of *Pyrococcus furiosus* rubredoxin determined with computational chemistry models.

Featured user publication

Lipton AS, RW Heck, GR Staeheli, M Valiev, WA De Jong, and PD Ellis. 2008. "A QM/MM Approach to Interpreting Zn-67 Solid-State NMR Data in Zinc Proteins." *Journal of the American Chemical Society* 130(19):6224-6230.

Related user publications

Lipton AS, MM Morlok, G Parkin, and PD Ellis. 2008. " ^{67}Zn Solid State NMR Spectroscopy of $\{[\text{Tp}^{\text{But}},\text{Me}]\text{Zn}(\text{OH})_2\}[\text{HOB}(\text{C}_6\text{F}_5)_3]_3$: The Importance of the Anion, $[\text{HOB}(\text{C}_6\text{F}_5)_3]_3^-$." *Inorganic Chemistry* 47(12):5184-5189.



Novel, integrated NMR capabilities at EMSL have enabled a broad range of new research areas. For example, computational capabilities facilitate the interpretation of complex experimental data sets with a myriad of applications. A Varian BioMAS™ probe coupled to the 900-MHz NMR spectrometer is specially designed to yield the highest resolution spectra available for bio-solid studies of membrane proteins and insoluble proteins, which are intractable for solution-state NMR.

Lipton AS, RW Heck, M Hernick, CA Fierke, and PD Ellis. 2008. "Residue Ionization in LpxC Directly Observed by ^{67}Zn NMR Spectroscopy." *Journal of the American Chemical Society* 130(38):12671-12679.

Lipton AS, RW Heck, SV Primak, DR McNeill, DM Wilson III, and PD Ellis. 2008. "Characterization of Mg^{2+} Binding to the DNA Repair Protein Apurinic/Apyrimidic Endonuclease I via Solid-State ^{25}Mg NMR Spectroscopy." *Journal of the American Chemical Society* 130(29):9332-9341.

Lipton AS and PD Ellis. 2007. "Modeling the Metal Center of Cys_4 Zinc Proteins." *Journal of the American Chemical Society* 129(29): 9192-9200.

Measuring molybdenum in catalysts

EMSL's NMR resources have opened new doors for molybdenum research, including studies about its potential applications in catalysis. Although the nature of molybdenum nuclei make molybdenum complexes ideally suited for studies using solid-state NMR, acquiring NMR spectra for molybdenum has presented a challenge. Molybdenum data were traditionally difficult to obtain because molybdenum nuclei potentially have broad resonances and the inherent sensitivity of available technologies was low. But the high sensitivity of EMSL's 900-MHz NMR spectrometer in conjunction with sophisticated sensitivity-enhancement techniques was used to overcome these barriers and has afforded researchers a wealth of information about the molecular and electronic structure of molybdenum without what has traditionally been a heroic effort. Further, computational chemistry calculations on molybdenum complexes were in agreement with experimental results. Because molybdenum is a metal that withstands exposure to quite high temperatures, understanding its structure and reactivity is of particular interest to the catalysis community.

Featured user publication

Forgeron MAM and RE Wasylshen. 2006. "A Solid-State ^{95}Mo NMR and Computational Investigation of Dodecahedral and Square Antiprismatic Octacyanomolybdate(IV) Anions: Is the Point-Charge Approximation an Accurate Probe of Local Symmetry?" *Journal of the American Chemical Society* 128(24):7817-7827.

Distinguished Participants

Arrowsmith, Cheryl – University of Toronto
Baker, David – University of Washington
David, William I – Rutherford Appleton Laboratory, United Kingdom
Dixon, David A – University of Alabama, Tuscaloosa
Fierke, Carol A – University of Michigan
Fyfe, Colin A – University of British Columbia
Grey, Clare P – State University of New York at Stony Brook
Kirkpatrick, R James – Michigan State University
Klevit, Rachel E – University of Washington
Montelione, Gaetano – Rutgers University
Opella, Stanley J – University of California, San Diego
Pakrasi, Himadri B – Washington University in St. Louis
Parkin, Gerard – Columbia University
Reeves, Raymond C – Washington State University
Rienstra, Chad M – University of Illinois at Urbana-Champaign
Shaw, Leon L – University of Connecticut
Squier, Thomas C – Pacific Northwest National Laboratory
Szyperski, Thomas – State University of New York at Buffalo
Terwilliger, Thomas C – Los Alamos National Laboratory
Varani, Gabriele – University of Washington
Wallace, Susan S – University of Vermont
Wasylishen, Roderick E – University of Alberta
Wilson, David M – National Institute of Aging

EMSL Publications Resulting from DOE's 900-MHz NMR Project

More than 130 publications have resulted from DOE's 900-MHz NMR project, including those in *Science*, *Nature*, and the *Proceedings of the National Academy of Sciences*. These publications reflect use of EMSL's 900-MHz, 800-MHz, or 600-MHz NMR spectrometers, which were all obtained as part of the 900-MHz NMR project. Five publications also reflect use of a 900-MHz spectrometer made available for testing by the 900-MHz NMR project manufacturer as part of their development of the 900-MHz NMR spectrometer technology. Of the more than 130 publications, almost 40 journal articles reflect use of 900-MHz NMR spectroscopy (these publications are denoted with an *).

ENERGY

2009

Hu JZ, JH Kwak, Z Yang, X Wan, and LL Shaw. 2009. "Direct Observation of Ion Exchange in Mechanically Activated LiH+MgB₂ System Using Ultra-High Field ⁶Li, ¹¹B and ²⁵Mg Magic Angle Spinning NMR Spectroscopy." *Physical Review Letters* (In Press).*

Osborn W, T Markmaitree, LL Shaw, JZ Hu, JH Kwak, and Z Yang. 2009. "Low Temperature Milling of the LiNH₂ + LiH Hydrogen Storage System." *International Journal of Hydrogen Energy* (In Press).*

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CATALYSIS

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Hu JZ, JH Kwak, Y Wang, CHF Peden, H Zheng, D Ma, and X Bao. 2009. "Studies of the Active Sites for Methane Dehydroaromatization Using Ultra High Field Solid State ^{95}Mo NMR Spectroscopy." *Journal of Physical Chemistry C* 113 (7):2936-2942.*

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Fyfe CA, JL Bretherton, and LY Lam. 2000. "Detection of the 'Invisible Aluminium' and Characterisation of the Multiple Aluminium Environments in Zeolite USY by High-Field Solid-State NMR." *Chemical Communications* (17):1575-1576.

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