High-Field Magnetic Resonance Facility

The High-Field Magnetic Resonance Facility (HFMRF) brings a powerful synergy of creative scientific staff and unique instrumentation to bear on complex scientific problems. HFMRF is equipped with state-of-the-art nuclear magnetic resonance (NMR) and pulsed electron paramagnetic resonance (EPR) instruments, all of which play a role in determining molecular structures that are relevant to environmental remediation efforts, materials development for national energy needs, and biological health effects.

HFMRF offers unique tools and techniques designed in-house to enable novel research, including 1) *in situ* catalysis probes, 2) radionuclide NMR capabilities, 3) solid-state NMR cryogenic probes for direct observation of metals in macromolecules, 4) high-temperature probe technology, 5) laser-polarized gas for visualizing gas-filled spaces using magnetic resonance imaging (MRI), and 6) pulsed EPR techniques designed to follow conformational changes in membrane protein complexes containing metal clusters. In collaborative partnership with world-class scientists around the globe, we forge innovative approaches to some of the most pressing research needs in environmental molecular science and other national research priorities.

Staff and science consultants within this facility offer expertise in the areas of structural biology, solid-state materials characterization, and MRI techniques. Research activities include structure determination of large molecular assemblies such as protein-DNA (normal and damaged DNA) and protein-RNA complexes that model assemblies that may form as a cellular response to chemical or radiological insults; examination of conformational changes in membrane protein complexes involving metal clusters using pulsed EPR; NMR-based structural and functional genomics; multi-nuclear detection and catalyst and materials characterization using solid-state techniques; and non-invasive biological imaging, integrated magnetic resonance and confocal microscopy, and slow-spinning NMR to study cell systems.

Instrumentation & Capabilities

NMR and EPR

- 900-MHz NMR
- 800-MHz NMR
- 750-MHz NMR
- 600-MHz NMR (two systems)
- 500-MHz (two wide-bore systems and two narrow-bore systems)
- 400-MHz, wide-bore NMR
- 300-MHz, wide-bore NMR (two systems)
- Horizontal-bore 2-tesla NMR
- EPR spectrometer with electron nuclear double resonance (ENDOR)/electron-electron double resonance (ELDOR) capability

Additional Capabilities

- Combined optical and magnetic resonance microscope
- Low-temperature probes for metallo-protein chemistry and structure
- Virtual NMR capability enabling use and collaboration with EMSL scientists for remote users via secure shell over the internet

Since the EMSL opened in October 1997, HFMRF has been one of the highest-volume experimental user facilities in support of local and external user research programs. During 2005, HFMRF supported 142 projects in which 355 external scientists used the NMR spectrometers.

The research interests of staff and users include some of the most exciting areas in modern molecular biology and biochemistry:

- **Structural/Functional Genomics**. Determination of three-dimensional structures of DNA, RNA, proteins, and enzymes and their intermolecular associations. Particular interests and collaborations exist relative to protein fold classification and sequence-structure-fold relationships.

- **Biomolecular Complexes**. Understanding the molecular interactions of larger complexes of biomolecules (proteins, DNA, RNA, and mimetic membranes) that are key regulators in cell signaling and growth (e.g., DNA damage recognition and repair processes).

- **Biological Imaging**. Acquisition of imaging and corresponding chemical information in biological samples, with particular interest in development of combined magnetic resonance and optical spectroscopy techniques to observe and elucidate biological processes.

- **Solid State**. Low-gamma nuclei detection, ultra-low-temperature NMR for sensitivity enhancement, and slow-magic-angle-spinning (MAS) methodologies for nondestructive research of cells, tissues, small animals, and bacterial colonies.

- **Measurement Science and Instrumentation Development**. Development and application of novel and unique NMR instrumentation techniques for biological and environmental problems.

Capabilities

Varian INOVA 900. The Varian 900 (Figure 1) is an INOVA-based spectrometer utilizing an Oxford 21.1-tesla (T) magnet with a 63-mm room-temperature bore. This system is capable of high-resolution liquid- and solid-state NMR. There are four radio frequency (RF) channels with waveform generators and triple-axis pulsed-field gradients. The wide-line analog digital converters (ADCs) run at 5 MHz and the narrow ADCs have a maximum rate of 500 kHz. This console also has a solids variable-temperature (VT) control capability. We currently have a 5-mm HCN probe with X, Y, and Z axis gradients for liquids, a 5-mm orthogonal HX powder probe optimized for low-gamma nuclides, a 5-mm orthogonal H-N-Zn triple-tuned probe for powders, and a 3.2-mm low-gamma HX MAS probe (24 kHz spinning).

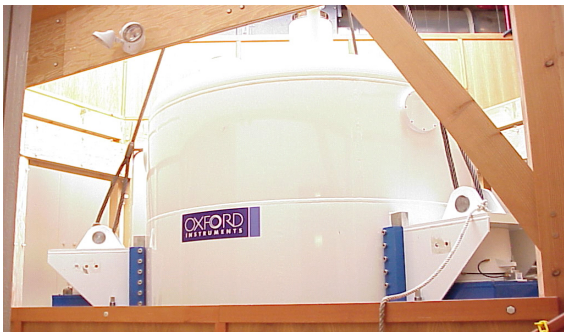

Figure 1. 900-MHz NMR spectrometer.

Varian INOVA 800. The Varian 800 (Figure 2) is an INOVA-based spectrometer utilizing an Oxford 18.8-T magnet with a 63-mm, room-temperature bore. This system is capable of high-resolution liquid- and solid-state NMR. There are four RF channels with waveform generators and pulsed-field gradients. The wide-line ADCs run at 5 MHz, and the narrow-line ADCs run at a maximum rate of 500 kHz. This console also has a solids VT control capability. Available probes include two 5-mm HCN probes with Z gradient for liquids, a 4-mm HXY MAS probe (25-kHz spinning, VT-capable), a 5-mm HX orthogonal powder probe optimized for low-gamma (38 to 65 MHz) nuclides, and a 5-mm HX static low-temperature probe (3.8 to 300 K). A 5-mm HX MAS probe (12-kHz spinning) is under construction.

Figure 2. Varian INOVA 800-MHz NMR spectrometer.

Varian INOVA 750. The Varian 750 (Figure 3) is an INOVA-based spectrometer utilizing an Oxford 17.6-T magnet with a 51-mm, room-temperature bore. This system is capable of high-resolution liquid- and solid-state NMR. There are four RF channels with waveform generators and pulsed-field gradients. The narrow ADCs have a maximum rate of 500 kHz. We currently have two 5-mm HCN probes (Z gradient), a 5-mm HCP probe (Z gradient), a 5-mm HX MAS probe (X tuning range is 321 to 130 MHz; the spinning speed is rated to 12 kHz), and two 5-mm HX MAS probes (15-kHz spinning) with X tuning ranges of 60 to 120 MHz and 30 to 50 MHz.

Figure 3. Varian INOVA 750-MHz NMR spectrometer.

Varian INOVA 600. The Varian 600 (Figure 4) is an INOVA-based spectrometer utilizing an Oxford 14.1-T magnet with a 51-mm, room-temperature bore. This system is capable of high-resolution liquid-state NMR. There are four RF channels with waveform generators and pulsed-field gradients. The narrow-line ADCs run at a maximum rate of 500 kHz. We currently have a 5-mm HCN probe with Z gradient and a 5-mm HX probe (X tuning range is 242 to 60 MHz). Our first 5-mm HCN cryogenic probe was installed on this system in February of 2004.

Figure 4. Varian INOVA 600- MHz NMR spectrometer with Cryoprobe.

Varian Unity 600. The Varian 600 (Figure 5) is an INOVA-based spectrometer utilizing an Oxford 14.1-T magnet with a 51-mm, room-temperature bore. This system is capable of high-resolution liquid-state NMR. There are three RF channels with waveform generators and pulsed-field gradients. The narrow-line ADCs run at a maximum rate of 500 kHz. We currently have a 5-mm HCN probe with Z gradient and a 5-mm HX probe (X tuning range is 242 to 60 MHz). Installation of a 5-mm pentaprobe is in progress.

Figure 5. Varian INOVA 600-MHz NMR spectrometer.

Varian Unity+ 500 Wide Bore. The Varian 500 Wide Bore (Figure 6) is a Unity+-based spectrometer utilizing an Oxford 11.7-T magnet with an 89-mm room-temperature bore. This system is capable of solid-state NMR, micro-imaging, and small-animal MRI. There are three RF channels with waveform generators. The wide-line ADCs run at 5 MHz. We currently have a 7-mm HX MAS probe (10-kHz spinning), an HX single-crystal probe, a ^{1}H CRAMPS probe, a micro-coil imaging probe, a 40-mm imaging probe, and a static HX low-temperature probe (2 to 300 K).

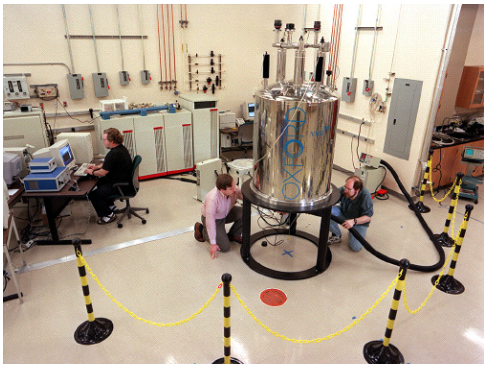

Figure 6. Varian Unity+ 500-MHz wide-bore NMR spectrometer.

Bruker Avance 500 Wide Bore. The Bruker Avance 500 Wide Bore (Figure 7) is a micro-imaging system using an 89-mm vertical room-temperature bore. The system is capable of imaging mice and also has high-resolution liquid magnetic resonance capabilities with a Bruker 10-mm QNP probe. This liquid probe has a ^{1}H outer coil and an inner coil that is switchable among ^{13}C, ^{31}P, and ^{19}F with no gradients. The system is equipped with a combined confocal and magnetic resonance microscope capable of monitoring single layers of eukaryotic cells in a perfusion system simultaneously with both modalities.

Figure 7. Bruker Avance 500-MHz NMR spectrometer.

Varian/Chemagnetics Infinity 500. The Chemagnetics 500 (Figure 8) is an Infinity-based spectrometer utilizing an Oxford 11.7-T magnet with a 51-mm, room-temperature bore. This system is capable of high-resolution liquid- and solid-state NMR. It has three RF channels and is equipped with both 16- and 14-bit ADCs. The solution state probes for this instrument include a 5-mm HCN gradient probe, a 5-mm DB gradient probe (X tuning range is 208.1 to 49.5 MHz), and a 10-mm HX probe (X tuning range is 218.6 to 21.2 MHz). There are two solid-state probes, a 5-mm HX MAS probe (X tuning range is 206.6 to 47 MHz; the spinning speed is rated to 12 kHz) and a 6-mm HX MAS probe (X tuning range is 218.6 to 48.7 MHz, H/F tuning range is 510.6 to 459 MHz; the spinning speed is rated to 9 kHz).

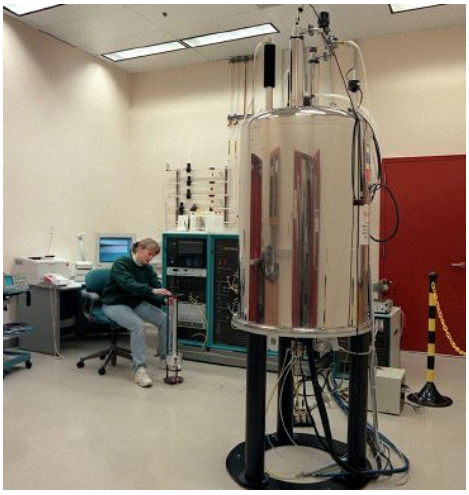

Figure 8. Varian/Chemagnetics Infinity 500-MHz NMR spectrometer.

Varian Unity+ 500 Narrow Bore. The Varian 500 (Figure 9) is a Unity+-based spectrometer utilizing an Oxford 11.7-T magnet with a 51-mm, room-temperature bore. This system is capable of high-resolution liquid-state NMR. There are three RF channels with waveform generators and pulsed-field gradients. We currently have a 5-mm HCN probe with Z gradients and a 10-mm HX probe.

Figure 9. Varian Unity+ 500-MHz narrow-bore NMR spectrometer.

Varian/Chemagnetics Infinity 300. The Chemagnetics 300 (Figure 11) is an Infinity-based spectrometer utilizing an Oxford 7.02-T magnet with an 89-mm room-temperature bore. This system is capable of high-resolution liquid- and solid-state NMR. It has three RF channels and is equipped with both 16- and 14-bit ADCs. The solution-state probes for this instrument include a 5-mm HX probe and a 10-mm HX probe. The solids probes are a 7.5-mm HX MAS probe (X tuning range is 136.7 MHz to 29.5 MHz; H tuning range is 274.7 to 349.1 MHz; spin rate is rated to 7 kHz) and a 5-mm HXY MAS probe (X tuning range is 129 to 57.4 MHz; Y tuning range is 85.1 to 21.2 MHz; spin speed is rated to 12 kHz).

Figure 11. Varian/Chemagnetics Infinity 300-MHz NMR spectrometer.

Varian Unity+ 300. The Varian 300 (Figure 12) is a Unity-based spectrometer utilizing an Oxford 7.04-T magnet with an 89-mm, room-temperature bore. This system is capable of solid-state NMR, micro-imaging, and small-animal MRI. There are two RF channels with wide-line ADCs running at 5 MHz. We currently have a 7-mm HX MAS probe (10-kHz spinning), an HX single-crystal probe, a ^{1}H CRAMPS probe, a single-tuned HX 5-mm, low-temperature MAS probe (35 to 300K, 12-kHz spinning), a 7-mm HX high-temperature probe (-100 to 500°C, 7-kHz spinning), a microscopy probe, and a 40-mm imaging probe.

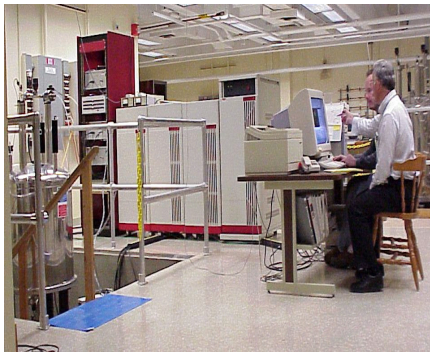

Figure 12. Varian Unity+ 300-MHz NMR spectrometer.

Horizontal-Bore 2-T Magnet. The 2-T magnet (Figure 13) provides unique capabilities for the HFMRF. It is connected to a Varian Unity+ console with two RF channels and wideline 5-MHz ADCs. It has a 30-cm, room-temperature bore and is equipped with an imaging gradient set capable of 50 gauss/cm. It is suitable for small animal or large sample imaging and *in vivo* spectroscopy. Three homemade birdcage coil probes are available: 8-cm and 5-cm imaging/spectroscopy probes and a 5-cm ^{3}He probe.

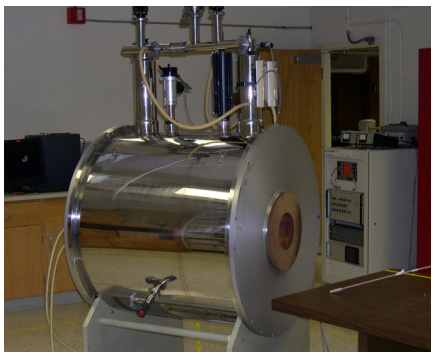

Figure 13. Horizontal-bore 2-T magnet.

Bruker Pulsed EPR/ENDOR/ELDOR Spectrometer. This multi-functional pulsed EPR spectrometer (Figure 14), operating in the X-band near 9.5 GHz, permits application of modern pulsed magnetic resonance techniques to systems containing unpaired electron spins. The system is based on the Bruker EleXsys console and SuperX-FT microwave bridge, which allow both ELDOR (electron-electron double resonance) and ENDOR (electron nuclear double resonance) measurements. A number of probes for both continuous-wave and pulsed spectroscopy are included, with an operating temperature ranging from room temperature to below liquid helium. System capabilities include measurement of g-tensors; hyperfine and nuclear quadrupole-coupling tensors for the study of electronic wavefunction of free radicals and metalloproteins; and determination of small dipolar interactions by pulsed ELDOR and double electron-electron resonance methods for the measurement of distances between radicals in solids or between spin labels in proteins.

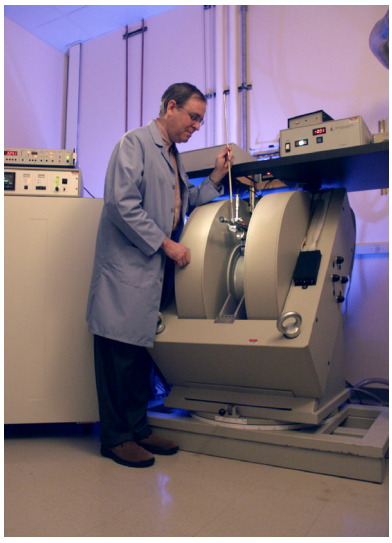

Figure 14. Bruker pulsed EPR/ ENDOR/ ELDOR spectrometer.

Upgrades

In fiscal year 2005, HFMRF has improved research capabilities both by upgrading current instrumentation and acquiring/developing new instrumentation.

- A new double-resonance MAS probe has been developed and employed for a wide range of nuclei for users of the 900-MHz (63-mm bore) NMR system in mid-fiscal year 2005. A high-speed 3.2-mm cross-polarization magic-angle-spinning (CPMAS) HX probe with a tuning range of ^{87}Sr to ^{23}Na was the first CPMAS probe developed for the ultrahigh-field 21.1-T 900-MHz system. Because of its high spinning speed (>23 kHz) and high-performance RF section, this probe has enabled several new solid-state experiments to be performed at this high-field level. One of the first user projects to benefit from the new probe has been a study of environmentally challenging waste site cleanup samples containing strontium.

- The 600-MHz NMR console (delivered at the end of fiscal year 2004) paired with the arrival the new "penta-probe" (mid-fiscal year 2005) has allowed the facility to support many EMSL user projects related to a wider variety of solution-state biomolecular NMR experiments. The penta-probe allows data acquisition on five important nuclei for biomolecular studies: ^{87}H, ^{31}P, ^{13}C, ^{15}N, and ^{2}H. The ability to observe ^{31}P both directly and indirectly supports a new class of experiments for RNA, DNA, and protein structure determinations. In addition, spectrometer performance has been improved by use of fine attenuators on "shaped-pulses," which was not possible using the earlier console.

- The 800-MHz Cryoprobe system arrived at the end fiscal year 2005. After development and testing, the Cryoprobe became available for user projects early in fiscal year 2006. Users of this system have gained more than threefold signal-to-noise increases, thus allowing more dilute systems to be observed. The 800-MHz Cryoprobe system is a flagship capability, offering greatly increased sensitivity to aid in determining structure and dynamics of biomolecular complexes.

- The console of the Bruker pulsed EPR spectrometer capability was been upgraded to provide increased resolution and sensitivity for metallo-protein structural studies and redox chemistry mechanism determination. The upgrade facilitated development of a new class of experiments to aid in structural information of membrane proteins; this structural information was not obtainable from methods such as x-ray diffraction and NMR spectroscopy.

- The 2-T ultra-wide-bore imaging system has facilitated significant advances in the ability to image lung tissue. This tissue is traditionally difficult to image utilizing conventional methods, but a combined effort of PNNL researchers and collaborators at the University of Utah to develop and employ a new hyperpolarized gas production system accessory has afforded dramatic improvements in imaging of lung tissue. In addition, the new methodology has reduced sample preparation to half the usual time. A study of the interaction of gases produced on fuel cell membranes is slated for fiscal year 2006.

Future Directions

Building upon the several successes of the facility in the areas of biology, materials science, environmental remediation, hydrogen storage, and catalytic materials, we expect user projects to continue to advance research in these areas in fiscal year 2006. The following areas are expected to be highlights for the facility in the coming year.

- Based on previous work to improve the NMR spectra from biological materials by spinning them slowly (less than 10 Hz) at the magic angle (magic-angle-turning), a new probe is being constructed to study chemical reactions. This probe is referred to as a discrete magic-angle-turning probe, and is part of an effort to build *in situ* catalysis probes to study catalysis and complex reaction mixtures. For this probe, the sample chamber is rotated slowly through only part of a revolution. This produces the desired improvement in the spectrum, but allows the connection of feed lines to flow reaction materials into the probe. If the probe were to spin freely and complete thousands of full revolutions in an experiment, these connections would not be possible. A prototype has been produced and tested and is now available to users; the final probe will be completed and tested in fiscal year 2006.

- Fuel cell efficiency depends greatly on the chemistry at the fuel cell-membrane interface. Last year, our 500-MHz wide-bore imaging system was used to image a prototype fuel cell for the first time, and the experimental methodology proved to be viable in following the movement of water in a test system. Imaging of a working fuel cell to study the movement of water across fuel cells and correlating water movement to fuel cell efficiency will be the next step in this research. This new technique shows promise in directly understanding the science behind the "drying effects" of water displacement on one side of a working membrane to the "flooded side," where water is moved across the membrane as protons are produced.

- A hyperpolarized gas chamber has been developed at HFMRF to improve the quality of lung images in studies of aerosol particulate damage with lung tissue. This work integrates the experimental data produced by magnetic resonance microscopy with theoretical lung models calculated using the Molecular Science Computing Facility supercomputer. This work, which is funded by the National Institutes of Health, will impact human health models of air quality.

- A project slated for fiscal year 2006 to use the hyperpolarized gas capability is a study of the interaction of gases produced on fuel cell membranes. The study also requires tracking water movement with imaging technology. Real-time, three-dimensional analysis methods are currently needed to probe polymer electrolyte membrane (PEM) fuel cell performance and degradation. MRI has tremendous potential to serve as a visualization tool for this purpose, but its utility for fuel cell research has yet to be fully realized. The goal of proposed work is to develop MRI methods for *in situ* diagnosis of fuel cell performance. Specific aims are to 1) visualize water distribution in the polymer electrolyte membranes, as well as in catalyst and gas diffusion layers; 2) directly correlate electrochemical performance to water management and distribution; 3) exploit magnetic resonance thermometry for mapping heat sources and sinks so results can be correlated with electrochemical activity, and 4) map the flow of gas-phase fuel and oxidant in cell and stack manifolds. With world-class facilities and personnel in both MRI and fuel cell technology, PNNL expects the proposed research to yield a clearer

understanding of how PEM fuel cells function, thereby gaining new insight into how to extend cell lifetimes, increase power density, and ultimately, reduce life-cycle costs.

- New projects that will use HFMRF instrumentation include 1) zero-emissions research, 2) technology materials development research, and 3) new hydrogen storage projects that aim to develop new classes of organic clathrates for high-density storage of hydrogen.

- A new user for the facility this year is using solid-state NMR to study the structure of components used in fuel cells. The detailed atomic structures are difficult to determine by other methods, making NMR uniquely powerful for this role. This DOE-funded project has produced data at medium magnetic fields, but it is clear that the project will benefit from access to higher magnetic fields. The 900-MHz NMR is the only spectrometer system in the world with substantial solid-state NMR capabilities, making it ideal for this project. It is expected that the improved spectra using this system will provide a substantial contribution to advancing this project.

- The cryoprobe for the 800-MHz NMR was installed in late fall 2005 and was made available to users' projects in January 2006. This addition will significantly expand the power of our 800-MHz system for studying structure and dynamics of biomolecular complexes in solution. Our experience with the cryoprobe on our 600-MHz system indicates that we can expect about a factor-of-three increase in signal-to-noise performance. This sensitivity improvement can be leveraged in several ways. It will enable the study of more dilute protein solutions in the same amount of time as currently used. This is important because many of the larger proteins and protein complexes studied on the 800-MHz NMR are less soluble than smaller proteins. At conventional protein concentrations, it will allow either higher throughput (data collection in less time) or collection of higher-resolution data, which is currently limited by experiment time. We expect to produce several publications in the area of bio-complexes that are presently limited by concentration limits. As we plan for the future, we envision that acquisition of a second 800-MHz NMR dedicated to high-field, solid-state research, allowing the cold probe system to remain dedicated to high-field liquids work, would be very advantageous for keeping the HFMRF on the cutting edge of research.

- User interest in the oldest 600-MHz NMR had abated in the past several years because of several factors, the most important being the age (nominally 15 years old) of the console. This aging console was capable of only a limited set of modern experiments relative to current models. However, we have now upgraded the console to INOVA and have also purchased a room-temperature penta-probe to further expand the experimental repertoire of this instrument. The new console and penta-probe will allow the facility to support many EMSL user projects with a wide variety of biomolecular NMR experiments. For example, the system is now capable of using fine attenuators for shaped pulses to implement the latest experiments to determine RNA, DNA, and protein structure. The penta-probe will allow direct and indirect detection of ^{31}P experiments at 600 MHz while retaining the capability of traditional hydrogen, carbon, and nitrogen triple-resonance experiments with the use of pulse-field-gradient technology.

- To accommodate the anticipated need for the Biogeochemistry and Biology Scientific Grand Challenges, the facility must continue to increase its capabilities in solid-state

probe development. The capabilities development within the group will focus on two magnets: a 21.15-T (900-MHz) NMR and an 18.8T (800-MHz) NMR. This year, HRMRF has developed a double-resonance MAS probe for the 900-MHz system that can be utilized to observe a wide range of nuclei; this probe has been employed by users for observation of low-gamma nuclei (30 to 50 MHz), including several projects characterizing ^{87}Sr, ^{99}Ru, and ^{25}Mg. This year, a new distinguished user (Claire Grey, State University of New York, Stonybrook) will use this probe (with slight modifications) to study Zr and Ga for a novel fuel cell project. A 3.2-mm HXY probe for biological solid-state NMR on the 900-MHz NMR system has been ordered and will be vital for undertaking structural studies of membrane proteins in support of the EMSL Membrane Biology Grand Challenge. Arrival of the system is expected in mid-fiscal year 2006. Application of a third-generation design of a cryogenic (10 K) static solid-state probe on the 18.8-T magnet has decreased the experiment time required from 11 hours (previously required on a 9.4-T magnet system) to one hour. During the next year, the facility expects to add two probes to the 900-MHz (21.1-T) system that will be available to users: 1) a triple-resonance HXY MAS probe dedicated to the biosolids studies mentioned above, and 2) if this capability development is approved, a "flat-coil" solid-state, triple-resonance NMR probe for oriented membrane proteins.

- The facility will continue to issue biannual calls for proposals to attract new users, with the expectation that user results are to be published and disseminated in the scientific literature. The facility seeks to leverage resources for maximum scientific and user advantage.

Hydrogen Storage Materials

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Hydrogen offers a potential clean energy source; however, new materials must be developed that provide high-volume storage capacity for hydrogen. This research aims to advance the fundamental understanding necessary for development of these materials.

Growing demands for clean energy sources that do not add more carbon dioxide and other pollutants to the environment have resulted in increased attention worldwide to the possibilities of a "hydrogen economy" as a long-term solution for a secure energy future, based on potentially renewable resources. Some of the greatest challenges are the discovery and development of new on-board hydrogen storage materials and catalysts for fuel-cell-powered vehicles. New materials that can store both high-gravimetric, high-volumetric densities of hydrogen, which release hydrogen at temperatures less than 100°C and uptake hydrogen at pressures less than 100 bar, are highly desired. The volumetric constraints eliminate pressurized hydrogen systems from consideration and point to development of solid storage materials. There are no currently known materials that meet these requirements. As such, there is a need for fundamental understanding of the chemical and physical properties of hydrogen-rich materials. The following highlight is a portion of our experimental analysis for studying molecular attributes that facilitate the release and uptake of molecular hydrogen.

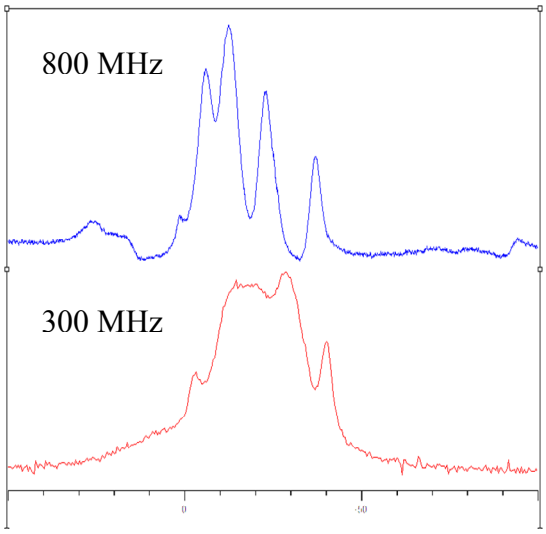

Figure 1. 11B{^{1}H} SSNMR (spinning speed 10 kHz) of NH_3BH_3 reaction products as a function of field. The increased resolution at 800 MHz allowed identification of two new products.

We recently suggested that efficient storage of hydrogen might be accomplished in compounds that have alternating electron-rich and electron-deficient sites capable of covalently binding H+ and H-, respectively. There are two fundamental premises that will guide us towards the discovery of novel hydrogen-rich materials that are operational at temperatures between ambient and 100°C: 1) binding of hydrogen requires formation of chemical bonds, and 2) inherent polarity of low-molecular-weight, species-bearing, electron-rich, and electron-deficient sites will likely result in the formation of molecular solids.

These guidelines led us to initially consider the ammonia borane (AB = NH_3BH_3). This inorganic analog of ethane yields far more favorable volumetric densities, as it is a solid (melting point 115°C) rather than a gas. The molecular crystalline solid is composed of a network of dihydrogen bonds formed between the protic H+ attached to nitrogen and hydridic H- attached to boron.

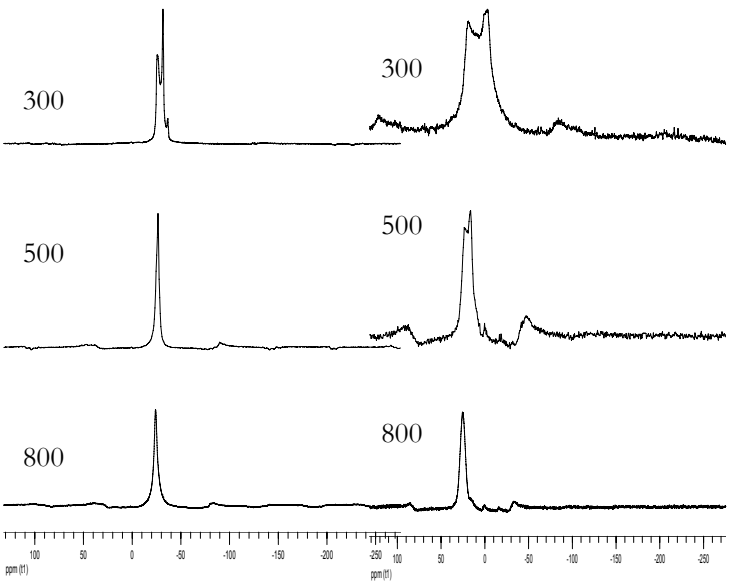

Figure 2. SSNMR 11B{1H} spectra as a function of field, spinning at 10 kHz. The starting material (left) and the final reaction products (after heating at 170°C) (right) are shown at all three fields. For some compounds, such as NH_3BH_3 (left), 500 MHz provides maximum narrowing. For some of the products (right), 800 MHz is needed to sufficiently narrow the peaks. Investigating at all three fields provides important information about the coupling constant, the symmetry, and the products.

Preliminary results showed the rates of hydrogen release from the bulk-phase solid ammonia borane follows an apparent nucleation and growth kinetic model. However, little is known about the nucleation events and the role of the inter-molecular dihydrogen bonding in the formation of molecular hydrogen. Solid-state nuclear magnetic resonance (SSNMR) $^{11}B\{^1H\}$ spectra of these reactions taken at 300-MHz ^1H frequency aided in determining reaction mechanisms; however, some products remain unidentified because of spectral overlap. NMR experiments run using higher fields (500 MHz and 800 MHz) enhanced resolution and reduced the quadrupolar coupling (Figures 1 and 2), which simplified the distinction between quadrupolar coupling and multiple-species reaction products formed as a result of hydrogen release (Figures 1 and 2). In some cases, 500 MHz was adequate to identify products, but 800 MHz was needed to clear ambiguities, as shown in Figure 2.

In addition to the ^{11}B SSNMR experiments, ^1H SSNMR spectra were also attained at 800 MHz. Each of the starting materials and reaction products were studied. As expected, chemical shift differences were observed. These observations will be further quantified using heteronuclear correlation to correlate the ^1H and ^{11}B resonances to provide further

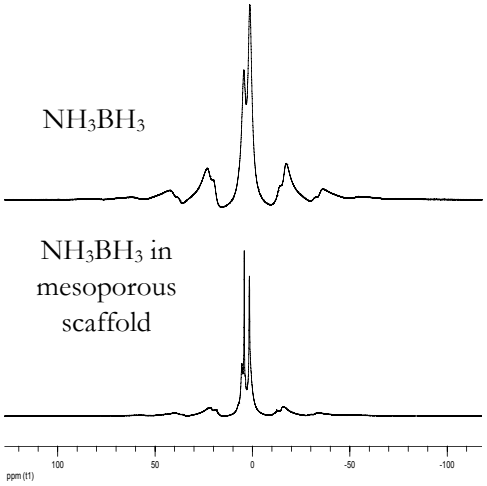

Figure 3. ^1H SSNMR (spinning speed 15 kHz) reveals a distinct narrowing of the two resonances as a function of scaffolding NH_3BH_3 into mesoporous silica.

product information. ^{1}H experiments are also being used to investigate scaffolding properties. We found that scaffolding the NH_3BH_3 into mesoporus silica reduces the temperature for hydrogen release. The chemistry and thermodynamics behind this observation are not understood; however, the extreme narrowing in the proton NMR would suggest either an increased ordering or a more liquid-like behavior in the scaffold (Figure 3). Further experiments, such as rotational echo double resonance for the direct investigation of atomic distances, are necessary to propose a mechanism, but the initial insight provides evidence of a fundamentally different organization within the scaffold.

Magnetic Resonance Imaging (MRI) of Respiratory Structure and Function in Laboratory Animals

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State-of-the-art MRI methods are currently being developed for quantifying respiratory structure and function in laboratory animals, with the goal of developing better models for extrapolating potential human health hazards from animal testing data.

During toxicology testing, laboratory animals are widely employed as human surrogates to assess the potential health risks associated with inhaled pollutants. However, significant differences in respiratory structure and function complicate the determination of human health risks from animal tests. One important factor that ultimately affects the fate of inhaled pollutants is the architecture of respiratory airways. Consequently, detailed descriptions of the three-dimensional airways in laboratory animals must be compiled before better computational methods can be developed to extrapolate human health risks from animal testing data. In recognition of this fact, scientists at the High-Field Magnetic Resonance Facility (HFMRF) have been working closely with users from across the nation to develop high-resolution MRI methods for visualizing excised respiratory tissue and airway casts. Airway structures are then digitally segmented from MRI data to reveal detailed three-dimensional models of airway architecture (Figure 1) (Timchalk et al. 2001).

Major differences in airflow dynamics between animals and humans may further complicate development of predictive models; however, noninvasive methods for visualizing airflow in living animals do not currently exist. To overcome this problem, scientists at the W.R. Wiley Environmental Molecular Sciences Laboratory (EMSL) worked with collaborators at the University of Utah to develop a unique optical pumping system for hyperpolarizing inert ^{3}He gas. In practice, this system establishes a highly non-equilibrium state characterized by dramatic polarization of the spin-1/2 nucleus of ^{3}He. As a direct result of this so-called hyperpolarized state, ^{3}He gas can be visualized directly with magnetic imaging. At HFMRF, techniques are being developed to image the flow velocity of ^{3}He gas in airway models, so that the dynamics of respiratory airflow can be clearly elucidated with experimental methods (Figure 2). Ultimately, we envisioned that such imaging technology will provide a quantitative

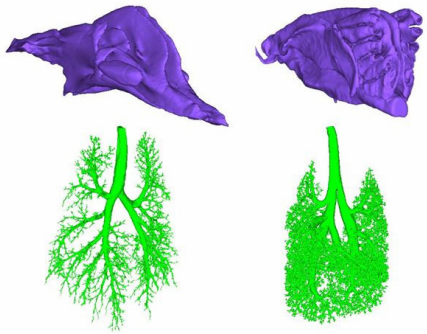

Figure 1. Montage of three-dimensional airway structures. Purple shows nasal sinuses of a monkey and rat on the left and right, respectively. Green shows pulmonary airways for each species.

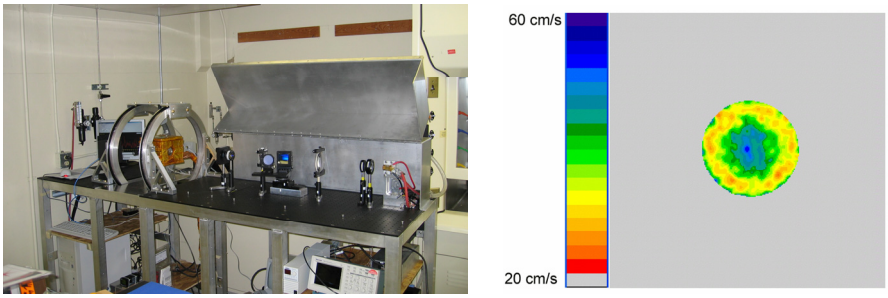

Figure 2. Left – Optical pumping system for hyperpolarizing ^{3}He gas. Right – Magnetic resonance imaging map of ^{3}He gas flow in a straight glass tube with a 3.4-mm diameter. Slice thickness is 6 mm and planar resolution is 156 microns. The calibrated color scale shows how flow velocity varies across the tube.

basis for validating computational fluid dynamics predictions that can then be used for understanding how risk assessment is influenced by cross-species differences in airflow.

As computer models become more sophisticated, new experimental methods will ultimately be required for their validation. For example, simulations based on airways shown in Figure 1 are capable of predicting the fate of individual particles. Current experimental techniques, however, lack comparable precision. EMSL scientists have developed a new noninvasive MRI method that exploits the interaction of ^{3}He gas with magnetic particles, allowing researchers to detect both their location and amount. Remarkably, initial tests in model systems indicate that this novel approach may be sensitive enough to detect single magnetic particles in the respiratory tract of a live rat (Minard et al. 2005). Current efforts to validate this technique has benefited from the expertise of researchers at the University of Washington, who specialize in the use of cryomicrotoming for visualizing the deposition of fluorescent microspheres in excised lungs. By exploiting microspheres that are both magnetic and fluorescent, future experiments will examine the correlation between *in vivo* magnetic particle detection measured with ^{3}He gas MRI and fluorescent analysis performed after sacrifice (Figure 3).

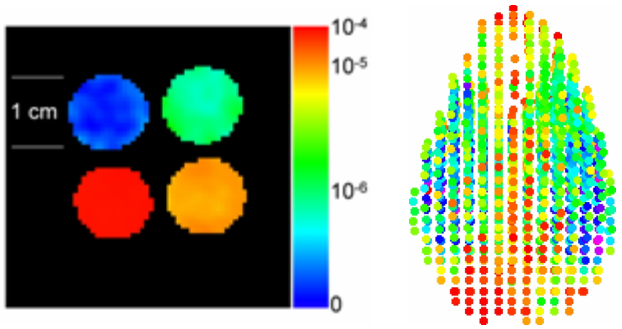

Figure 3. Left – ^{3}He MRI map of regional particle deposition in a foam model of alveolar airways (Timchalk et al. 2001). Four pieces of foam are shown; each was loaded with a different volume fraction of 2-micron-diameter magnetic particles in gas-filled voids. The colorized scale shows the measured volume fraction of magnetic particles and results agree within 15 percent of known values. Right – Three-dimensionsal color map of particle deposition in the rat lung measured using fluorescent microspheres and a cryomicro-toming approach. The color scale is not the same as for magnetic resonance data and the lung is viewed front-on from the abdominal side.

Citations

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Timchalk C, HE Trease, LL Trease, KR Minard, and RA Corley. 2001. "Potential Technology for Studying Dosimetry and Response to Airborne Chemical and Biological Pollutants." *Toxicology and Industrial Health* 17(5-10):270-276.

Detection and Characterization of ZSM-5 in a Mesoporous Host Matrix

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In this work, ultrahigh-field ^{27}Al magic-angle-spinning (MAS) and multiple quantum magic-angle-spinning (MQMAS) nuclear magnetic resonance (NMR) spectrometries have proven to be essential tools for the detection of zeolite nanoclusters and the different aluminum environments in nanozeolite/mesoporous aluminosilicate composites. Demonstration of the presence of zeolite nanoclusters in these composites is the first step in evaluating their potential as a catalyst of the future—critical for converting crude oil into gasoline and other petroleum products.

Zeolites are catalysts that are critical for converting crude oil into gasoline and other petroleum products; without them, many fuels and petrochemicals would not be readily available. Future improvements in catalyst performance require a good understanding of their internal structure. While methodologies such as x-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy have provided limited structural information, NMR spectroscopy has given the most detailed insights. To obtain the high-quality data needed for structure determinations, higher magnetic fields are needed, such as the 750- and 800-MHz NMR spectrometers available at the W.R. Wiley Environmental Molecular Sciences Laboratory. Data collected at 500 MHz has proven useful, but not all of the aluminum atoms present can be detected at this field.

Zeolites are very open frameworks of aluminosilicates or silicates composed of corner- and edge-sharing SiO_4^{4-} and AlO_4^{5-} tetrahedra. They contain regular systems of cavities and channels of molecular dimensions, which control the uptake of organic molecules in terms of shape and size selectivity. Zeolites are powerful acid catalysts, but their small pores limit diffusion, which in turn limits the speed of catalysis. Improvement in catalytic rates would bolster the efficiency of the oil refining industry. It has been shown recently that the coating of protozeolitic nanoclusters onto the surface of preformed mesostructured aluminosilicates can greatly improve their hydrothermal stability and acidity, two properties that are essential for catalysis (Figure 1). Although FTIR observations confirm the presence of zeolite nanoclusters in the mesopore channels, XRD diagrams fail to indicate the presence of zeolite crystals in the coated material. Ultrahigh-field (17.6-T) ^{27}Al MAS and MQMAS NMR were used to detect the zeolite nanocrystals and quantify the multiple aluminum environments in these materials. A complete account was published by Do and co-workers (2004).

Figure 2a shows the various aluminum environments detected by ^{27}Al MAS NMR at 17.6 T. The parent sample shows two broad peaks (one tetrahedral and the other octahedral) characteristic of amorphous materials, and the calcined zeolite-coated samples show two additional sharper peaks consistent with the chemical shift values of the corresponding zeolite. The broadening of these peaks, compared to the ones obtained from perfectly

crystalline zeolites, results from their being in a less ordered environment. As Figure 2b shows, it is possible to discriminate the higher degree of ordering of the zeolite (longer T2) from the amorphous mesoporous framework (shorter T2) through a series of spin echo experiments. ^{27}Al MQMAS NMR confirms these results, as only two partially resolved signals are observed, which have been assigned to the tetrahedral aluminum sites in the zeolite (Figure 2c).

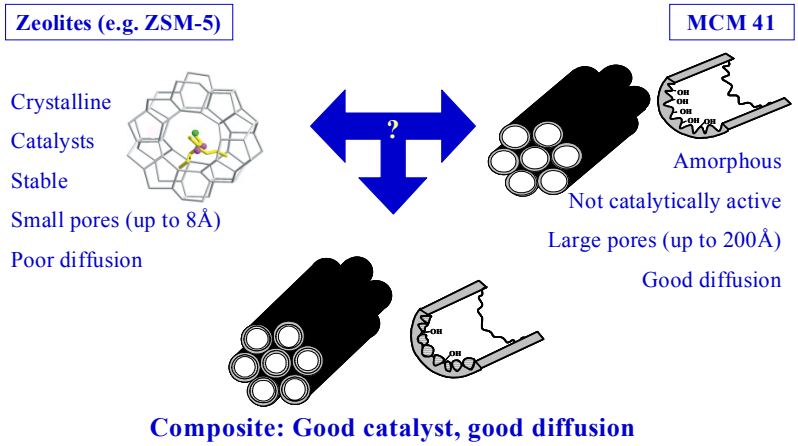

Figure 1. The incorporation of zeolite nonacrystals in the mesoporous structure.

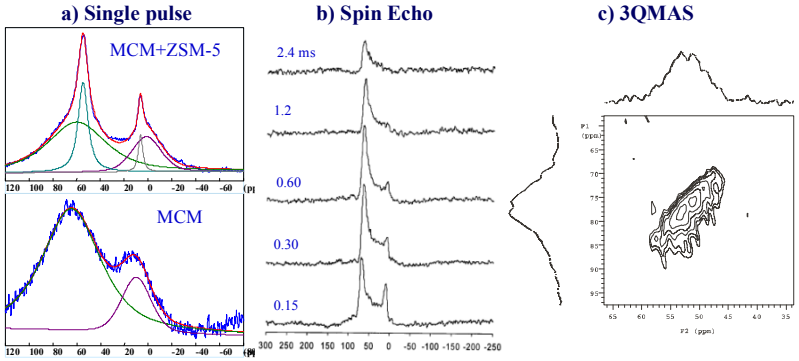

Figure 2. The use of ultrahigh-field ^{27}Al NMR to discriminate the nanozeolite from the mesostructure.

Ultrahigh-field ^{27}Al MAS and MQMAS NMR have proven to be essential tools for the detection of zeolite nanoclusters and the different aluminum environments in nanozeolite/mesoporous aluminosilicate composites, as these features cannot be detected by conventional XRD techniques. The demonstration of the presence of zeolite nanoclusters in these composites is the first step in evaluating their potential as a catalyst of the future.

Citations

Do Trong-on, A Nossov, MA Springuel-Huet, C 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.

Development of Multipurpose Tags and Affinity Reagents for Rapid Isolation and Visualization of Protein Complexes

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Strategies are being investigated to develop reagents and technology for high-throughput processes that will foster rapid characterization of protein complexes in microbial cells. The end result will be data collection of time-dependent changes in protein complex formation in response to environmental conditions, providing a systems-level understanding of how microbes adapt to environmental changes. Ultimately, these strategies will help to optimize methods pertaining to efficient energy use, carbon sequestration, and environmental remediation

This research is being conducted to develop the necessary reagents and technology for high-throughput methods that will ensure rapid and quantitative characterization of protein complexes in microbial cells. We are investigating a strategy focused on the development of multiuse protein tags engineered around a tetracysteine motif (i.e., CCXXCC), which has previously been shown to provide a highly selective binding site for cell-permeable, arsenic-containing affinity reagents that can be used to identify and validate protein complexes in living cells. Use of novel affinity reagents that become fluorescent upon binding to engineered tags will permit quantification of expressed proteins and purification and stabilization of protein complexes. Ultimately, high-throughput data collection of time-dependant changes in protein complex formation in response to environmental conditions will be available, thus permitting a systems-level understanding of how microbes adapt to environmental change.

Important to our progress has been access to the High-Field Magnetic Resonance Facility (HFMRF). Nuclear magnetic resonance (NMR) spectrometry analysis has contributed to the production of cell-permeable orthogonal fluorescent probes by facilitating regular identification and characterization of synthesis pathways and resultant products. NMR analysis also has been implemented to investigate the structure, dynamics, and binding kinetics of peptide-reagent interaction.

This research is currently in a synthesis-intensive phase to optimize the fluorescent dye and tag pair by sensible design and synthesis of new dyes and screening of the tetracysteine peptide library. Research is also underway to expand the applications of the FlAsH dye (a Fluorescein Arsenical Helix binder; see Figure 1 for structure) to study protein-protein interactions. By taking advantage of the large increase in the fluorescence signal associated with binding the proposed fluorescent affinity reagents to the protein tag, it will be possible to use online detection to monitor affinity isolation of protein complexes and rapidly identify the proteins in the complex by using mass spectrometry.

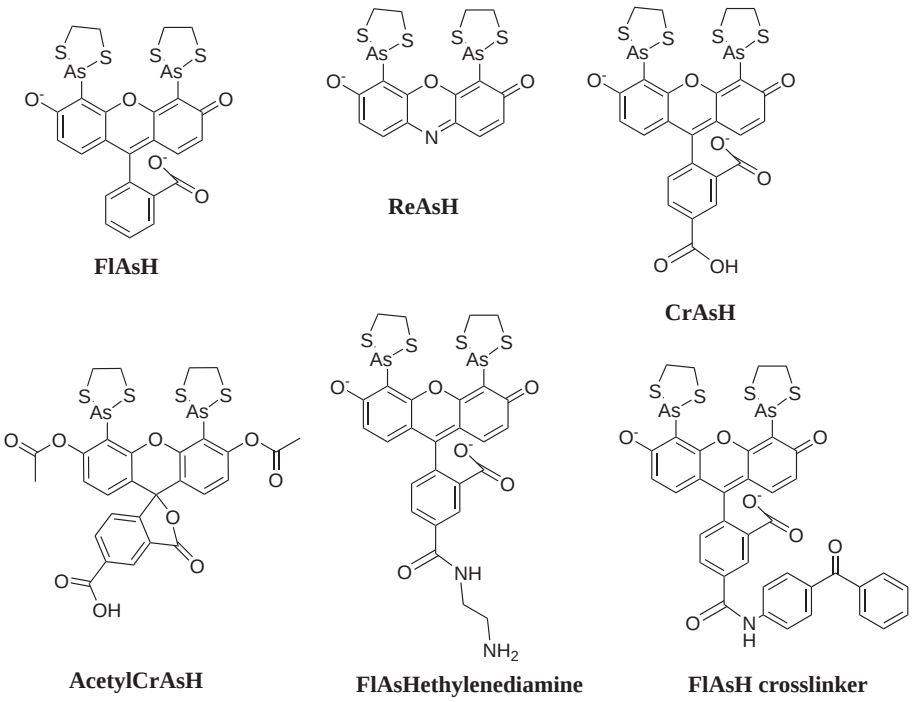

Figure 1. Sample fluorescent probe compounds characterized using FlAsH dye.

Ultimately, these methods will permit optimization of useful metabolic pathways to fulfill U.S. Department of Energy goals involving efficient energy use, carbon sequestration, and environmental remediation.

Papers that describe our successful work in this area in detail have been published by Chen and co-workers (2005a, b) and Mayer and co-workers (2005).

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Mistranslation Fragment of an *In Silico* Designed Novel-Fold Protein Forms an Exceptionally Stable Symmetric Homodimer with a High-Affinity Interface

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The last decade has seen tremendous advances in the field of computational protein design. Designed proteins with high thermodynamic stabilities may allow the generation of longer-lasting species useful in industrial applications and therapeutics.

In silico protein sequence and structure optimization algorithms have been successfully applied to redesign proteins and to create novel protein structures. Designed proteins may achieve thermodynamic stabilities greater than those reported for any naturally occurring proteins. An obvious application of these exceptionally stable proteins is the generation of longer-lasting designer proteins for therapeutics. However, while exceptional protein stability offers advantages in resistance to proteolysis and unfolding, there may also be biological costs once these proteins are expressed or delivered in the cell. Because exceptionally stable computationally designed proteins are created in the absence of specific evolutionary pressure, they provide a rare opportunity to reveal aspects of the cellular protein production and surveillance machinery that are subject to natural selection.

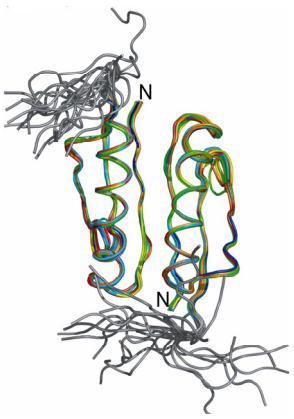

Figure 1. Nuclear magnetic resonance (NMR)-generated structures of CFr. The top 20 NMR models from the final CFr structure calculation are shown as ribbons. Each model is superimposed on the average backbone coordinates for residues 3-51 (structured region, separate color for each model) in both chains from the entire ensemble. The structured regions have an ensemble root-mean-square deviation of 0.33Å over the backbone atoms and 0.75Å over all heavy atoms. Residues from the unstructured tails (52-58) are colored in grey.

Using purely computational techniques, researchers at the University of Washington recently generated an extremely stable, small, globular protein, called Top7, with a sequence and fold not observed previously in nature. In collaboration with scientists at Stanford University, the University of North Carolina, Fred Hutchinson Cancer Research Center, and the High-Field Magnetic Resonance Facility (HFMRF), University of Washington researchers have now demonstrated that a region of the Top7 protein corresponding to the final 49 C-terminal residues is efficiently mistranslated in *Eschericia coli*, and that the solution structure of the resulting CFr protein is a compact, stable obligate homodimer (Figure 1). HFMRF's 600-MHz cryoprobe was instrumental in collecting data that allowed CFr dimer structure determination, including

collection of C^{12}/C^{13} edited/filtered experiments. The solution NMR structure reveals that the CFr dimer has a novel symmetric interface formed by two identical CFr subunits, and analysis of NMR backbone dynamics further confirmed the rigidity of the structure. In addition, the researchers have determined that stabilization of CFr by disulfide-induced covalent circularization yields a super-stable miniature protein that can serve as a robust scaffold for further protein engineering. Current efforts using CFr and SS.CFr as scaffolds include preparation of epitope-peptides for production of antibodies against human immunodeficiency virus and functionalization with peroxide-activating catalysts for bioremediation.

High-Field Magnetic Resonance Characterization of Titanate-Based Ceramics and Glasses for High-Level Waste Immobilization

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(a) The Royal Veterinary and Agricultural University, Frederiksberg, Denmark
(b) University of Cambridge, Cambridge, United Kingdom
(c) Pacific Northwest National Laboratory, Richland, Washington

Researchers have developed a technique that allows normal titanium samples, including materials simulated to study radioactive waste vitrification, to be studied for the first time by nuclear magnetic resonance (NMR) spectrometry.

Characterization of the titanate phases of synroc and titanosilicate glasses is an important step to understanding the materials chosen to immobilize high-level waste streams generated by nuclear fuel reprocessing. However, the spectroscopy of titanium is complicated by several issues, such as titanium's low gyromagnetic ratio and the fact that the material is a quadrupolar nuclide with two active isotopes having NMR frequencies nearly coincident. The NMR active isotopes for titanium are ^{47}Ti and ^{49}Ti, which are present at 7.44 percent and 5.41 percent natural abundance, respectively. On a 500-MHz NMR spectrometer, they differ in resonant frequency by only 6.25 kHz. As quadrupolar nuclei, their linewidths are much larger, so the two hopelessly overlap. The only previous solution was to isotopically enrich samples in one or the other of the two, but this severely limits the samples that can be studied. Using very high magnetic fields, such as the 21.1-T magnet available on the High-Field Magnetic Resonance Facility's 900-MHz NMR spectrometer, enhances the minimal frequency difference and reduces the quadrupolar broadening of the lines so that in some cases, they are barely resolvable.

By using a novel pulse program equipped with frequency-selective excitation, this collaborative group has been able to produce spectra at 900 MHz from anatase containing a natural abundance of ^{47}Ti and ^{49}Ti, where the complete spectrum of the isotopes are collected separately (Figure 1). This requires the highest magnetic field possible, as well as the new pulse program, and creates the opportunity to study titanium by NMR at natural abundance, which is crucial for structural studies on glasses used for immobilizing radioactive waste. This research is discussed further in Larsen et al. (2006).

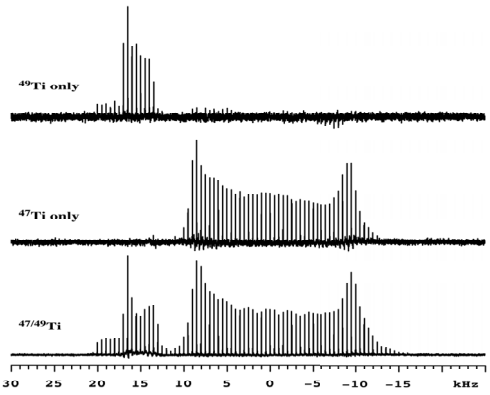

Figure 1. Experimental ^{47}Ti,^{49}Ti spectra of anatase acquired at 21.1T (50.75 MHz) using the Quadrupole Carr-Purcell Meiboom Gill pulse sequence with ^{49}Ti-selective pulses. Top: The ^{49}Ti-selective pulse sequence. Middle: The ^{47}Ti-selective pulse sequence. Bottom: All experi-ments employed a radio frequency field strength of 12.5 kHz, s_1 = 50.0 ls, s_2 = s_4 = 76.0 ls, and s_3 = 24.0, M= 50, s_a = 2.0 ms, a dwell time of 2.0 ls, and 512 scans. All experiments were acquired using a recycle delay of 5 seconds and apodized by Lorentzian linebroadening of 10 Hz.

Citation

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.

Microscopic View of Strontium Interactions in Minerals

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(a) The Pennsylvania State University, University Park, Pennsylvania

(b) Pacific Northwest National Laboratory, Richland, Washington

Characterization of strontium in phyllosilicate minerals with solid-state nuclear magnetic resonance (NMR) will contribute toward an understanding of strontium/mineral interactions in soils exposed to leaking tank waste, thus enabling the design of better models for predicting the fate of strontium in the environment and better cleanup technologies.

The interactions of strontium with clay minerals and zeolites are not well understood and must be studied to construct accurate models for predicting the environmental fate of radioactive ^{90}Sr released from areas such as the Hanford Site. Solid-state NMR spectrometry is a useful tool for probing the molecular structure of materials, including the interactions of cations sorbed by mineral systems. However, there is only one NMR active isotope of strontium (^{87}Sr), and the direct study of strontium with solid-state NMR is experimentally challenging. Strontium-87 has similar chemistry to ^{90}Sr and is a quadrupolar nucleus ($I = -9/2$) with a low natural abundance (~7 percent), a low gyromagnetic ratio ($\gamma = -1.163 \times 10^7$ 1/T·s), and large quadrupolar coupling constants (14 to 25 MHz) (Bowers et al. 2006a, b). These factors contribute to a lack of sensitivity that must be overcome to perform time-efficient studies of strontium in natural samples, such as environ-mentally relevant clay minerals and zeolites. In our on-going studies at Pacific Northwest National Laboratory (PNNL) on the 21.14-T (^1H resonance frequency of 900 MHz) instrument, we are using sensitivity-enhancing techniques to characterize the local electromagnetic environment of strontium nuclei in mineral systems.

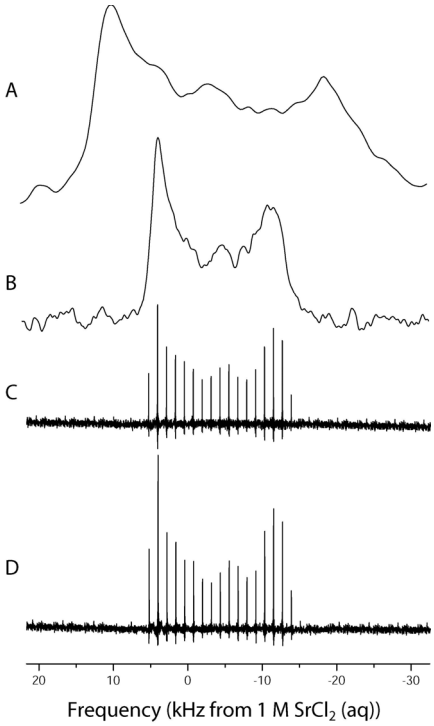

Figure 1. Strontium carbonate ^{87}Sr NMR spectra: (A) static echo at 11.74 T, (B) static echo at 21.14 T scaled by 1/37, (C) QCPMG at 21.14 T scaled by 1/275, (D) DFS-QCPMG at 21.14 T scaled by 1/826.

We have shown in earlier work that the 21.14-T field strength provides an order of magnitude enhancement to the signal-to-noise ratio for ^{87}Sr experiments and that Quadrupolar Carr-Purcell-Meiboom-Gill (QCPMG) magic-angle-spinning adds an additional order of magnitude (Bowers et al. 2006a). Unfortunately, these enhancements were insufficient to observe strontium in complex minerals. In the past year, we were able to demonstrate that adding the double frequency sweep (DFS) preparatory scheme results in up to an additional fivefold enhancement to signal-to-noise performance (Figure 1), permitting the detection of strontium resonances in soil minerals.

With DFS-QCPMG at 21.14 T, we were able to perform solid-state NMR studies of strontium in additional inorganic and organic systems; these studies were impossible with QCPMG alone. One important conclusion from our data is that water-strontium interactions have a profound effect on ^{87}Sr NMR spectra; in fact, we have been unable to observe strontium in any system where there is water in the strontium-hydration sphere (Figure 2). The reasons behind this are the subject of current investigations in our laboratory. DFS-QCPMG at 21.14 T has also been used to successfully examine the strontium-binding environment in a number of heat-treated micas, montmorillonites, titanates, and titanosilicates. To perform such ^{87}Sr NMR analyses at more conventional fields (i.e., 11.74 T) would require on the order of 1800 days rather than the 3 days required to produce the spectrum in Figure 2. The library of quadrupolar parameters and their relationship to crystal structure that has been prepared from our studies of simple systems now allows informed predictions of the strontium-binding environment based on ^{87}Sr NMR parameters and x-ray diffraction studies. This library could not have been developed over the past year without the use of DFS-QCPMG on the 21.14-T, 900-MHz NMR spectrometer at the High-Field Magnetic Resonance Facility. A detailed discussion of the strontium-binding environment in one of the mica samples mentioned earlier is the subject of a manuscript currently under review for publication in the *Journal of Physical Chemistry B* (Bowers et al. 2006b). We also intend to publish the results of our mineral studies in one paper detailing strontium binding in phyllosilicates and another paper outlining strontium binding in designer titanosilicate materials (both to be submitted in 2006).

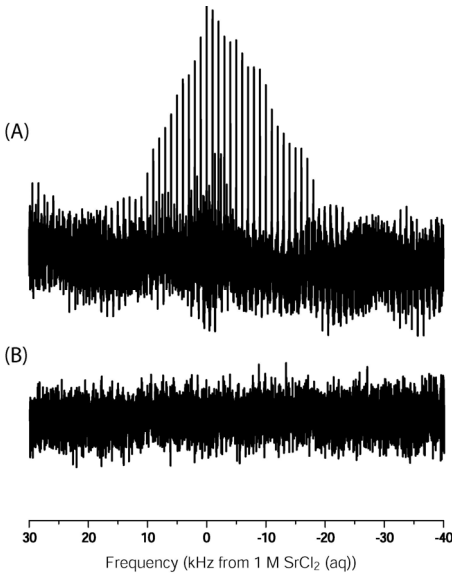

Figure 2. Strontium NMR spectra of Na-4 Mica after (A) and prior to (B) heat treatment at 500°C for 4 hours. The heat-treated sample produces a single strontium resonance fit well by a quadrupolar line shape.

In the coming year, we intend to return to PNNL to study additional titanosilicate materials developed at Savannah River Laboratory specifically to sequester strontium from Hanford-like wastes. Some of these materials have tunable selectivities for cesium and strontium, making this work highly important to the U.S. Department of Energy mission. We will also be continuing our investigations of mineral weathering under near-field exposure to simulated tank waste leachate (Chorover et al. 2003; Crosson et al. 2006) by monitoring the kinetics of mineral dissolution and re-precipitation in samples of natural Hanford sediments.

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Crosson GS, SK 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.

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Structural Genomics Collaborative Access Team

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The goal of structural genomics is to make the three-dimensional, atomic resolution structures of most proteins easily available from their corresponding DNA sequences.

Structural genomics is a global effort to survey the breadth of protein structure space by determining high-resolution x-ray and nuclear magnetic resonance (NMR) structures from every family of proteins. This collection of about 10,000 non-redundant protein structures will help us understand the mutual relationships among sequence, structure, function, and evolution. Structures of many more proteins (e.g., those from an entire process or pathway in a specific organism of interest to human health or the environment) will be accessible through homology modeling.

The Northeast Structural Genomics Consortium (NESGC, www.nesg.org), which is supported by the National Institutes of Health Protein Structure Initiative II, makes extensive use of High-Field Magnetic Resonance Facility (HFMRF) to acquire NMR data for protein structure determination. During the last six years, data collected at HFMRF has been used for the determination of dozens of protein structures. In 2005, approximately 50 weeks of instrument time was devoted to NMR data collection for structural genomics. These activities were conducted through the Structural Genomics Collaborative Access Team, or SG-CAT. While most of SG-CAT's efforts had been in NMR spectroscopy, in 2005 an x-ray structure was completed by SG-CAT members MA Kennedy and S Ni. Shown in Figure 1 are the structures deposited in the Protein Data Bank in 2005 that were determined entirely with data collected at PNNL.

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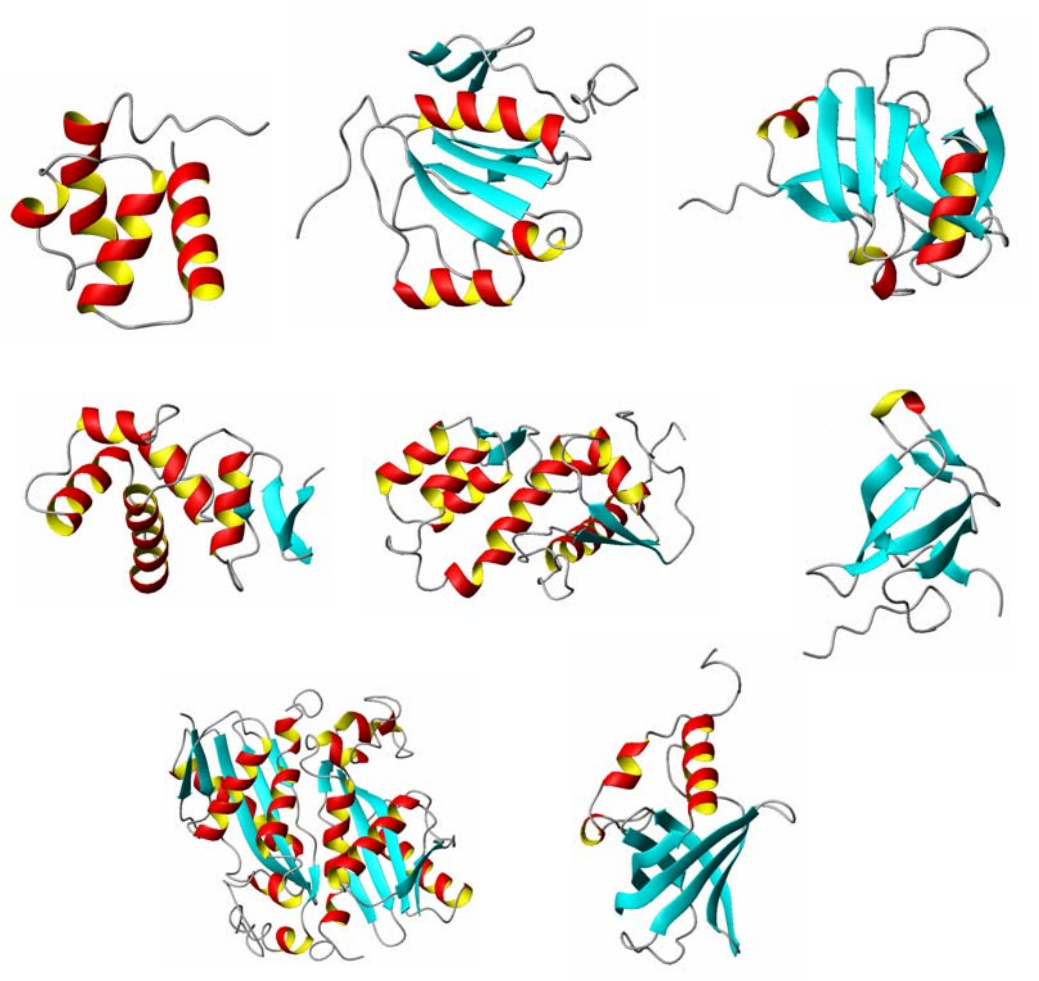

Figure 1. NMR structures (and one x-ray structure) of structural genomics target proteins determined with data collected at HFMRF and deposited in the Protein Data Bank (www.rcsb.org) in 2005. The Protein Data Base ID, source organism, protein name, and functional information are indicated. From top, left to right: 1YWW Pseudomonas aeruginosa PA4738 unknown function/sigma factor response, 2FVT Rhodopseudomonas palustris Rpa2829, 2EXN Bordatella bronchiseptica BB0938 unknown function, 1YX3 Allochromatium vinosum DsrC sufur metabolism/electron transport, 2B3W Escherichia coli YbiA unknown function and new fold, 2AKK R. palustris Rpa4479 PhnA-like domain, 1ZNO (x-ray structure) Vibrio cholerae VC0702, dUTP and dITP pyrophosphatase,1YWU Pseudomonas aeruginosa PA4608 PilZ domain.

User Projects

Structural Studies of Riboswitches
Washington State University Tri-Cities, Richland, Washington
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Pacific Northwest National Laboratory, Richland, Washington
MA Kennedy, GW Buchko, S Ni

Magnetic Resonance Imaging (MRI) of Excised Lung Tissue
University of California, Davis, Davis, California
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High-Field ^{27}Al Nuclear Magnetic Resonance (NMR) of Simulated Tank Waste Precipitates
Pennsylvania State University, University Park, Pennsylvania
GM Bowers, KT Mueller, GS Crosson

High-Field Magic-Angle-Spinning/Nuclear Magnetic Resonance (MAS/NMR) of ^{87}Sr in Environmental Samples
Pennsylvania State University, University Park, Pennsylvania
GM Bowers, KT Mueller, GS Crosson

Structure of Telomerase RNA and Telomeric Proteins
University of Washington, Seattle, Washington
G Varani, TC Leeper

Application for 800-MHz Nuclear Magnetic Resonance (NMR) Spectrometer Time to Facilitate the Structural Study of the Complex Formed by Pox-Virus-Encoded Protein and Human CC Chemokine MIP-1beta
Texas A&M University, College Station, Texas
PJ Liwang, L Zhang

AlphaB-Crystallin – The Core and the Oligomer: A Structural Investigation
University of Washington, Seattle, Washington
P Rajagopal, R Klevit

Characterization of the Structure of the Calcium-Dependent Antibiotic Daptomycin Using Solution and Solid-State Nuclear Magnetic Resonance (NMR)
University of British Columbia, Vancouver, British Columbia, Canada
SK Straus, PC Dave

Determination of the Three-Dimensional Solution Structure of NosL, A Potentially Novel Copper(I) Metal Transporter
Montana State University, Bozeman, Montana
V Copie, LM Taubner, GA Jacobs

Structure and Interactions of a Domain of Dynein Intermediate Chain: Protein Folding Coupled to Binding
Oregon State University, Corvallis, Oregon
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Structure Determination of Membrane Proteins
Case Western Reserve University, Cleveland, Ohio
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Slow-Magic-Angle-Spinning (MAS) of Lipids in Mouse Fast and Slow Skeletal Muscle
University of Washington, Seattle, Washington
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Solid-State Magic-Angle-Spinning/Nuclear Magnetic Resonance (MAS/NMR) of High-Valent, Cation-Exchanged H-MFI
University of California, Berkeley, Berkeley, California
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Study of the Network Structures of Polymer-Derived Amorphous SiAlCN Ceramics
University of Central Florida, Orlando, Florida
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W.R. Wiley Environmental Molecular Sciences Laboratory, Richland, Washington
SD Burton

Hydrogen Storage Materials
Pacific Northwest National Laboratory, Richland, Washington
WJ Shaw, T Autrey, JC Linehan

Structural Genomics of Eukaryotic Model Organisms
Rutgers University, Piscataway, New Jersey
JM Aramini, GT Montelione

Study of the Binding Mechanism of Mutant SN-15 to Hydroxyapatite Using 15N31PREDOR
University of Washington, Seattle, Washington
JM Popham, V Raghunathan, JM Gibson, GP Drobny, PS Stayton

Structure of PR Domain of RIZ 1 Tumor Suppressor
The Burnham Institute, La Jolla, California
KR Ely, K Briknarova

Routine ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) Analysis of Functionalized Semiconductor and Metallic Nanoparticles Synthesized for Biodetection Studies

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Nuclear Magnetic Resonance (NMR) Structural Studies of Clustered DNA Damage

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Structural Investigations of Solid Materials by High-Resolution, Solid-State Nuclear Magnetic Resonance (NMR) at Very High Field

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Investigation of the Role of Mg^{2+} in DNA Repair Proteins APE1, Pol, and FEN1
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Investigation of Catalyst Reaction Mechanisms by *In Situ* High-Field, High-Resolution Nuclear Magnetic Resonance (NMR) Spectroscopy
Pacific Northwest National Laboratory, Richland, Washington
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Development of a Novel Approach for Imaging Inhaled Particulates
Pacific Northwest National Laboratory, Richland, Washington
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***In Situ* Magnetic Resonance Investigations of Metabolism and Mass Transport in Biofilms**
Pacific Northwest National Laboratory, Richland, Washington
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Slow-Magic-Angle-Spinning/Nuclear Magnetic Resonance (MAS/NMR) Methodology Developments
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***In Vivo* High-Resolution, Slow-Magic-Angle-Spinning (MAS) Magnetic Resonance (MR) Spectroscopy in Mice**
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HYSCORE Analysis of Protein Peroxyl Radicals in Myoglobin and Hemoglobin
University of Alabama, Tuscaloosa, Tuscaloosa, Alabama
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Deposition of Cobalt-Doped Oxides for Spintronic Applications
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Structural Genomics Collaborative Access Team
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Grand Challenge in Biogeochemistry
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Sensitivity-Enhancing Nuclear Magnetic Resonance (NMR) of ^{87}Sr Nuclei in Environmental Samples
Pennsylvania State University, University Park, Pennsylvania
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Implementation of Dipolar Recoupling with a Windowless Sequence (DRAWS) to Facilitate Investigations of Biomineralization
Pacific Northwest National Laboratory, Richland, Washington
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Structural Studies of *Gaussia princeps* Luciferase
Lewis and Clark College, Portland, Oregon
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Structural Investigation of a Molecular Chaperone
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Pulsed Electron Paramagnetic Resonance (EPR) of Membrane Proteins
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Properties of Surface-Functional Groups of Black Carbon from Historic Charcoal Blast Furnaces
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***In Situ* High-Field, High-Resolution Nuclear Magnetic Resonance (NMR) Spectroscopy**
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Nuclear Magnetic Resonance (NMR) Study of Effects and Mechanisms of Mechanical Activation on Hydrogen Sorption/Desorption of Nanoscale Lithium Nitrides
Pacific Northwest National Laboratory, Richland, Washington
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An Extended Study of the Molybdenum(IV) Octacyanide Anion: Comparison of Dodecahedral Versus Square Antiprismatic Structure Forms Via Solid-State ^{95}Mo Nuclear Magnetic Resonance (NMR) Spectroscopy
University of Alberta, Edmonton, Alberta, Canada
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Purification and Biophysical Characterization of MR-1 Redox Proteins
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High-Resolution Imaging of the Passive Heart and Cardiac Valves for the Next-Generation Cardiac Models
Pacific Northwest National Laboratory, Richland, Washington
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Investigation of Biodegradable and Nonbiodegradable Thermal-Reversible Gelling Polymers Using Slow-Magic-Angle-Spinning Nuclear Magnetic Resonance (NMR) Spectroscopy
Pacific Northwest National Laboratory, Richland, Washington
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Grand Challenge in Membrane Biology
Washington University in St. Louis, St. Louis, Missouri
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Structure and Function of the Membrane Protein OEP16
Arizona State University, Tempe, Arizona
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Structure of Trityls
University of Chicago, Chicago, Illinois
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Structural Biology of the Human High-Mobility Group A Proteins: Characterizing the Hub of Nuclear Function
Washington State University, Pullman, Washington
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Pacific Northwest National Laboratory, Richland, Washington
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Identifying Value-Added Products from Biomass Conversion Reactions by Nuclear Magnetic Resonance (NMR) Spectroscopy
Pacific Northwest National Laboratory, Richland, Washington
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High-Resolution Nuclear Magnetic Resonance (NMR) Investigation of Nanomaterials

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Nuclear Magnetic Resonance (NMR) Structural Investigations of BRCA1

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Structural Proteomics of *Myobacterium tuberculosis*

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Molecular Probes of Quinol Oxidation by the Cytochrome b6f Complex

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Endor of Trityl Radicals

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Ultrahigh-Field Nuclear Magnetic Resonance (NMR) Studies of Stable Isotope Applications

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Solid-State ^{67}Zn Nuclear Magnetic Resonance (NMR) of Synthetic Metalloprotein Models

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Characterization of the Mineral Phases of Matrix Vesicles during Induction of Crystalline Mineral Formation

University of South Carolina School of Medicine, Columbia, South Carolina
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Nuclear Magnetic Resonance (NMR) Microscopy of Diffusive Transport in Natural Porous Mineral Grains
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Free-Radical-Processesing and Heavy-Ion-Irradiated DNA
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Pulsed Electron Paramagnetic Resonance (EPR) Studies of Transition Metal-Exchanged Zeolites and Molecular Sieves
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Solid-State Nuclear Magnetic Resonance (NMR) Characterization of Metal Phosphines
Australian Nuclear Science and Technology Organization, Menai, New South Wales, Australia
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Probing the Mechanism of the Alkaline Phosphatase Reaction by ^{67}Zn and ^{25}Mg Nuclear Magnetic Resonance (NMR)
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Structure of a Helical-Signaling Domain from Cas
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Spatial Properties of Clustered Free Radicals Produced in DNA and Biodosimeters by Ionizing Radiation
Purdue University, West Lafayette, Indiana
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Electron Paramagnetic Resonance (EPR) and Electron Nuclear Double-Resonance (ENDOR) Characterization of Fe- and Mn-Containing Spin Systems of Relevance to Proteins, Magnetic Materials, and Oxidation Catalysts
University of Vermont, Burlington, Vermont
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Distance Measurements in RNA Using Double Electron-Electron Resonance (DEER) Spectroscopy with Site-Directed Spin Labeling
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Structural Proteomics: Annotating the Genome Using Three-Dimensional Structures
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Crystal Orientation Determination of a Piezoelectric Crystal
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Defect Dynamics on Crystalline Quartz
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Electron Paramagnetic Resonance (EPR) Resonance of Non-Heme Iron Proteins
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Metabonomics Assessment Following ANIT or Acetaminophen Administration to Male Fischer 344 Rats
Colorado State University, Fort Collins, Colorado
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Microscopic Characterization of Porosity, Diffusivity, and Tortuosity in Single Particles of Hanford Sediments Using Nuclear Magnetic Resonance (NMR) Techniques
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Quantifying the Intracellular Spatial State and Dynamics of Water-Macromolecule Interactions: Studies of Living Cells
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Special-Purpose, Re-Configurable ASIC Hardware for Accelerating Protein Structure Analysis Software
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Structural Studies of the Hyaluronan Receptor CD-44 and CD44-HA Complex
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Solid-State Nuclear Magnetic Resonance (NMR) Studies of Chloropropyl Silica Gels
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Composite Gadolinium Oxide and Yttrium Phosphate Nanoparticles for Managing Cancer Therapy with Magnetic Resonance Imaging
University of Washington, Seattle, Washington
M Zhang, NJ Kohler, CG Sun

Interaction of *Escherichia coli* Formamidopyrimidine-DNA Glycosylase (Fpg) with Damaged DNA Containing an 7,8-Dihydro-8-Oxo Lesion
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TRAPDOR Experiments on Siliceous Sinters from Thermal Springs
University of Montana, Missoula, Montana
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Magnetic Resonance Microscopy of Water Dynamics at Hydrophilic Surfaces
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Complexation of Th(IV) by Organic Acids in Aqueous Solution
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Kinetics of Polyphosphate Decomposition in Heterogeneous Environments
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Nuclear Magnetic Resonance (NMR) Detection of Radiation Damage in Ceramics
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Nuclear Magnetic Resonance (NMR) Analysis of Synthesized Organic Compounds for Modification of Nanostructures.
Pacific Northwest National Laboratory, Richland, Washington
LS Fifield, F Zheng, CL Aardahl, RJ Wiacek

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Development of Multipurpose Tags and Affinity Reagents for Rapid Isolation and Visualization of Protein Complexes

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Characterization of Colloid Mobility Using Nuclear Magnetic Resonance (NMR) Techniques

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Stablilization of Soil Organic Matter: Land Use, Erosion, and Burial

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Characterization of Folding and Self-Association of the Survival Motor Neuron Protein

Arizona State University, Tempe, Arizona
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Separation of ^{47}Ti and ^{49}Ti Solid-State Nuclear Magnetic Resonance (NMR) Lineshapes by Static Quadrupolar Carr-Purcell-Meiboom-Gill (QCPMG) Experiments

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Investigation of Lineshapes in Fully ^{13}C/^{15}N-Labelled S1 Domain from RNaseE Using Solid-State Nuclear Magnetic Resonance (NMR)

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Relaxation-Nuclear Magnetic Resonance- (NMR)-Imaging Investigation of Initiated Polymer Degradation.

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Structure Determination of Membrane Proteins from *Mycobacterium tuberculosis*
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Investigation of Soot Morphology and Microstructure with Respect to Oxidation

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Probe Performance Testing at 900 MHz

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Development of Organophosphorus Compounds for Solid-State Lighting Applications

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Nuclear Magnetic Resonance (NMR) for Catalyst Studies

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Membrane-Organized Chemical Photo-Redox Systems
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Solid-State ^{13}C Nuclear Magnetic Resonance (NMR) Study of Cu Complexation with Standard Ligands
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The Structure and Dynamics of the Interaction of Membranes with Amyloid Oligomers
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