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Renewal proposal outlines exciting new initiatives

Certainly the present financial landscape for scientific research remains both a challenge and an opportunity... (wait, let's start this over...)

The 2013-2017 renewal proposal is submitted! In a much-appreciated collaborative effort with our User Community and External Advisory Committee, we have proposed—in 50 pages or fewer—an extremely exciting future in support of high magnetic field research at the MagLab.

Rather than revisit scientific landmark scientific experiments of the past five years, which have been well represented in past issues of *Mag Lab Reports*, let's visit a few of the renewal proposal's primary initiatives:

**Magnet Time**

Our users have requested that a 50% to 100% increase in magnet time be made a high priority (see the User Committee's user survey results on page 26.) The DC Magnet facility will benefit when the Series Connected Hybrid magnet comes online with 36 T in a 40mm diameter bore using only 14 MW. An initiative in the renewal proposal will build a 41 T/32mm insert for this Series Connected Hybrid and enable us to operate it in parallel with the 45 T hybrid magnet, a ~20% increase in total DC magnet time and a doubling of user access to magnetic fields above 40 T. Over the longer term, we propose the launching of regular weekend operations of this unprecedentedly efficient hybrid magnet, which would provide a further increase in DC magnet hours for a total 30% increase by 2017. The High B/T facility proposes converting an existing nuclear demagnetization system for user experiments, which would bring a 50% increase in sub-millikelvin magnet days to reduce user wait times that have exceeded twelve months.

**Fiscal Efficiency**

The March 2009 launch of DC Magnet Flextime allows users to best utilize allocated Megawatt-Hours tailored to their particular experiment. The resultant efficiencies and user requests have encouraged us to add 28 MW magnets to the renewal proposal, comfortable in the knowledge that Flextime users can determine the best magnet utilization within tight electricity budgets.

All three MagLab campuses have updated helium recovery and liquefaction capabilities via multi-million-dollar institutional investments from our three partner institutions: Florida State University, the University of Florida, and Los Alamos National Laboratory.

**Peak Field**

Dramatic increases in peak fields are newly possible and now proposed for the coming two-to-ten years. A few examples: the separately-funded ion cyclotron resonance magnet will arrive in the coming years, increasing peak fields for ICR from 14.5 T to 21 T. The renewal proposal

**DIRECTOR’S DESK**

by Greg Boebinger

ABOVE Congratulations to the gang at the Los Alamos Pulsed Field Facility on their record-breaking 97.4 T pulsed magnet shot, and on the new availability of 92 T pulsed fields for users. Here, the Pulsed Magnet Team, along with Susan Seestrom, Associate Director of Experimental Physical Science at Los Alamos, await the record-breaking shot. (Photo by Robb Kramer/LANL)
requests funding for a 21 T ICR user support scientist. Breakthroughs since 2007 in high-temperature superconducting materials and magnet technologies will increase peak fields for superconducting magnet users to 30 T. The State-of-Florida-funded $7.5M power supply upgrade from 40 MW to 56 MW (the upgrade that enabled the commissioning of the 25 T split magnet earlier this year) will enable the increase of our 31 T/50mm and 35 T/32mm DC magnets to 37 T and 41 T respectively. And finally, advances in strength and ductility of nanocomposite conductors, along with engineering experience over a half-decade of user experiments on the 85 (now 92!) T pulsed magnet, will advance non-destructive pulsed magnets to 100 T.

It is an exciting time to perform high-magnetic-field experiments at your nearest National High Magnetic Field Laboratory. I recommend it highly.

Gregory S. Boebinger
Director of the Magnet Lab

*To see past issues of MLR, search “Mag Lab Reports” at magnet.fsu.edu
The 19th international conference on Electronic Properties of Two-Dimensional Systems (EP2DS 19) and the 15th conference on Modulated Semiconductor Structures (MSS 15) were held together in Tallahassee, Florida on July 25-29, 2011. Just under 400 scientists attended. To view a full gallery of the event, search EP2DS at magnet.fsu.edu, then select “Photo Gallery” on the left menu.
World-record 97.4 T pulsed magnetic field reached

Pulsed Field Facility poised for user research at 92 T

In a three-second span on Friday, August 19, researchers and engineers at the Magnet Lab Pulsed Field Facility at Los Alamos National Lab created a 97.4 Tesla magnetic field—the highest non-destructive magnetic field in the world. The magnet will operate at 92 T for user science.

Two of the magnet’s initial experiments provided data for users, each of which required the higher magnetic fields to observe new phase transitions.

Two parallel experiments were conducted during the testing of the magnet, along with an in situ calibration based on the de Haas- van Alphen effect in polycrystalline copper. The experimental data awaits detailed analysis before being made public, but one compound studied exhibits a well-formed magnetization plateau that evidences a new type of antiferromagnetic state. The second compound revealed new phase transitions via dilatometry measurements, a thermodynamic probe that detects the expansion and contraction of a material when it passes through a phase transition.

The previous record, 91.4 Tesla, was set in June of this year by the High Magnetic Field Laboratory Dresden.

This record marks important progress toward the lab’s goal of reaching 100 Tesla. The project is funded by the 100 Tesla Multi-Shot Program, a collaborative effort between the National Science Foundation and the Department of Energy’s Office of Basic Energy Sciences.

“Years of effort by the best technical staff, engineers and scientists have gone into this achievement. This capability marks a significant milestone in the ability of US science to unravel the complex nature of materials that our future may depend on,” said Pulsed Field Facility Director Chuck Mielke.

The record-breaking field was created by upgrading the lab’s existing 85 T pulsed magnet system, previously tested to just over 89 Tesla.

The increase in magnetic field was achieved primarily through the development of a new insert magnet (Figure 1, inset).

The insert magnet upgrade is the second generation of technical development for the 100 T pulsed field program. Numerous changes have been made in the selection of materials and in the engineering design. The new insert employs a series of Cu-Nb nano-composite conductors which have been graded in strength and electrical conductivity for each layer to compensate for growing magneto-resistance at 100 T fields. This conductor effort entailed the specification and development of new Cu-Nb nano-composite wire designs specifically for this project. The reinforcement design on the insert’s outer layers has also been improved; a new grade of MP35N reinforcement was developed for this effort. The engineering design and production procedures for the reinforcement fabrication was also improved. The internal coil design was modified based upon our operational experience with the 85 T magnet systems. Such development opportunities are uniquely possible at the Magnet Lab’s Pulsed Field Facility in Los Alamos due to the unmatched intensity of successful operations at 85 T, and the experience and dedication of our generator operations and magnet development groups.

FIGURE 1. Magnetic field pulse from the “100 tesla” multi-shot magnet with the waveform of the “insert” magnet down in the left inset and a cross-section of the insert itself shown on the right.

Photo courtesy of Robb Kramer/Los Alamos National Lab
Sodium MRI in a rat migraine model and a NEURON simulation study support a role for sodium in migraine

by Michael G. Harrington1, Eduard Y. Chekmenev2, Victor Schepkin3, Alfred N. Fonteh1, Xianghong Arakaki1.

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3. Center for Interdisciplinary Magnetic Resonance, National High Magnetic Field Laboratory, Florida State University, Tallahassee, FL 32310.

Introduction

Increased lumbar cerebrospinal fluid (CSF) sodium has been reported during migraine. We used world-unique, ultra-high-field MRI to investigate cranial sodium in a rat migraine model, and simulated the effects of extracellular sodium on neuronal excitability. We found that in the rat migraine model, sodium rises to levels that increase neuronal excitability. We propose that rising sodium in CSF surrounding trigeminal nociceptors increases their excitability and causes pain, and that rising sodium in vitreous humor increases retinal neuronal excitability and causes photosensitivity.

We are investigating the pathophysiology of migraine, which episodically disables 30 million Americans, cause unknown. I wanted to observe changes in intracranial sodium in vivo in a rat model of migraine. Since this had never been done before and my preliminary data indicated that the changes might be as low as 4 mMol1, 2, I wanted to use the most sensitive detector; thus I came to the FSU Magnet Lab, specifically to use the 21 Tesla 900 MHz magnet.

Elena Oborina, a wonderful Russian analytical chemist, had completed some preliminary sodium studies on human cerebrospinal fluids in my lab in Pasadena, and her husband, Ed Chekmenev, helped me in the design of these studies. Ed is now on the faculty at Vanderbilt University, but he had worked with Tim Cross and colleagues at the Magnet Lab a few years ago.

After getting permission to be users on the 900 MHz instrument, Ed and I traveled to Tallahassee in June 2010 for our first experiments. A Scotsman, I was primarily working with Ed, Victor Schepkin the resident sodium MRI guru, and with probe advice from Peter Gor’kov. At times, I thought I was in a Floridian satellite of Russia. But nyet! While speaking our own respective versions of American English, we embarked on experiments that are the first to show that sodium increases in the eyes, the cerebrospinal fluid, and the brain in the rat migraine model in vivo. We returned in October 2010 for a second week, and our results are summarized in the boxed abstract from our first paper that is now in press in the journal Cephalalgia. The data achieved in these first-of-a-kind experiments has also helped us to garner the always-coveted NIH funding with a 5-year RO1 grant to continue this work.

This work is the first that we’re aware of that demonstrates a functional change in the central nervous system (treated rats have a decreased pain threshold and increased light sensitivity compared to controls) associated with increased sodium to levels that increase neuronal excitability. We have proposed that this may be the origin of the pain and light sensitivity in migraine, and are testing this further with anti-migraine pharmacological experiments. Using more invasive methods of cerebrospinal fluid sampling, Elena and I
had previously determined that humans have a distinct 12-hour sodium rhythm\(^3\) that is likely to have many physiological implications, many of which we may be able to monitor \textit{in vivo} using the 21 T magnet. To extend our studies of migraine and physiological variations with direct brain measurements in this rat model system, we plan to return to the Magnet Lab to make further use of this unique facility.

This was not a purely Scottish/Russian effort. We could not have done this work without the exclusive Magnet Lab facility and the help of Kathy Harper, veterinarian, Ashley Blue, problem-solver and provider of machine maintenance and crucial keys, and Fabian Calixto-Bejarano, who helped with much of our experimental setup. The logistics and technicalities of the behavioral rat experiments with \textit{in vivo} imaging between Pasadena and Florida is not trivial, yet the staff, administration, and overall resources at the Magnet Lab made this one of my most successful experimental efforts. I cannot speak highly enough of the aforementioned Magnet Lab staff that allowed these experiments to succeed. I think the ability to perform \textit{in vivo} sodium MRI with the sensitivity to reproducibly detect changes of 7-17\% in magnitude will be of enormous relevance for many neuroscience questions. I just hope that I will still get access to the Magnet Lab as hordes from the neuroscience world may now race to Tallahassee.

In summary, we have established a non-invasive method (sodium MRI) to detect changes during migraine that may be involved in its pathophysiology. We intend to apply this method to test migraine triggers and treatments, and develop methods to monitor humans with the ultimate goal of lessening the serious burden of migraine.

### Methods

Behavioral changes in the nitroglycerin (NTG) rat migraine model were determined from von Frey hair withdrawal response and photography. Central sensitization was measured by counting cFos-immunoreactive cells in the trigeminal nucleus caudalis (TNC). Sodium was quantified \textit{in vivo} by ultra-high field sodium MRI at 21 Tesla. Effects of extracellular sodium on neuronal excitability were modeled using NEURON software.

### Results

NTG decreased von Frey withdrawal threshold (\(p = 0.0003\)), decreased eyelid vertical height:width ratio (\(p < 0.0001\)), increased TNC cFos stain (\(p < 0.0001\)), and increased sodium between 7.5 and 17\% in brain, intracranial CSF, and vitreous humor (\(p < 0.05\)). Simulated neurons exposed to higher sodium have more frequent and earlier spontaneous action potentials, and corresponding earlier sodium and potassium currents.

### Conclusions

In the rat migraine model, sodium rises to levels that increase neuronal excitability. We propose that rising sodium in CSF surrounding trigeminal nociceptors increases their excitability and causes pain and that rising sodium in vitreous humor increases retinal neuronal excitability and causes photosensitivity.

### REFERENCES

25 T Split Magnet adds new, world-unique user capabilities to Magnet Lab

While the Magnet Lab has developed 14 previous world-record resistive magnets over the past 17 years, the new 25 T Split Magnet is not simply the next in the line. This magnet required a complete rethinking of resistive magnet technology's limits.

by Mark Bird, Scott Bole, Jim O'Reilly and Jack Toth
In late June, as engineers, scientists, and passers-by looked on, the lab’s new Split Magnet was charged successfully to 38.25 kA, consuming a total 28 MW of power and providing a flux-density of 25 T available in the system’s experimental space.

The Split Magnet consists of 5 resistive coils, with the first two inner-most coils electrically connected in parallel and 3 more outer coils electrically connected in series. All coils employ axial current grading for field optimization and stress management. To meet the unique design challenges, Split Florida-Helix technology is used around the mid-plane of the two inner most coils and state-of-the-art Florida-Bitter technology is used for the regular winding in all five coils.

All the resistive magnets developed previously at the Mag Lab were solenoids; most used the Florida-Bitter technology developed at the Magnet Lab (now used by five of the six largest resistive magnet labs worldwide). However, relying solely on Florida-Bitter technology wasn’t an option; the most efficient part of any high-field solenoid is the mid-plane region, and in the 25 T split, more than half the midplane region is missing to provide vacuum space for scattering. The vacuum space is surrounded by a vacuum jacket consuming over 10% of the available mid-plane, as well as an annular gap allowing the passage of cooling water (220 l/s consuming another 8% of the mid-plane space). That leaves less than one third of the total available space in the mid-plane for the actual conductor, which must carry 4 x 40 kA of current and over 580 tons of magnetic clamping force (distributed over the 5 coils).

Resistive split systems are more challenging than superconducting systems, due to the large quantities of cooling water and the large currents required. There are two approaches from which to choose: allow the cooling water to pass through the mid-plane, or restrict water flow to the separate halves of the magnet, above and below the mid-plane. The latter approach simplifies construction and leads next to a choice between axial and radial cooling. Axial cooling would require a large gap between the coils and the scattering space to allow cooling water flow, which would reduce the field significantly. Radial cooling results in coils that are not as efficient as the axially-cooled Florida-Bitter coils. Allowing cooling water to pass through the mid-plane would allow the 2 halves of the magnet to have the minimum separation, resulting the highest possible field; however, to enable this, the Split Florida-Helix needed to be invented.

The Split Florida-Helix for a coil starts as two cylinders of the high-strength, high-conductivity copper-beryllium alloy Hycon-3, one each for the top and bottom halves of the coil. The inner and outer diameters of the cylinder are cut to the correct sizes on a lathe. The 4 scattering ports have the shape of elliptical cones and are cut via wire Electro-Discharge Machining (EDM). Narrow cooling holes are needed parallel to the axis of the coil and require starter holes fabricated via EDM hole-drilling followed by wire EDM to convert the round starter hole into an elongated cooling hole. The most complicated step is cutting the helix into the part which requires 5-axis wire EDM.

Several of these processes required the development of special tooling not available commercially. Only state-of-the-art wire-EDM hardware and control software is capable of producing these features. Consequently, we were unable to locate commercial vendors with these manufacturing capabilities willing to take on a project involving so much R&D and we developed the complete EDM manufacturing capability in-house to produce these critical parts.

Like all high-field resistive magnets, the 25 T split operates at high power density (15 W/mm3), high stress (>90% yield stress) and high heat-flux. To be confident the magnet would reach field without tearing itself apart, very sophisticated 3-dimensional coupled physics calculations (thermo-electro-magnetic-mechanical) had to be performed using the finite-element method prior to finalizing the design.

With that, this magnet became by far the most complicated resistive magnet ever developed.

### Mid-plane data of the 25 T Split Magnet

<table>
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<th>A1</th>
<th>A2</th>
<th>B</th>
<th>C</th>
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<td>51%</td>
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</tr>
<tr>
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<tr>
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<tr>
<td>Conductor Space</td>
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<td>16%</td>
<td>17%</td>
<td>27%</td>
<td>35%</td>
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Split magnet to offer rich research opportunities

The following are excerpts from longer articles about split magnet research printed in Volume 17, Issue 1 of Mag Lab Reports, on page 9. Visit magnet.fsu.edu to read the full text.

Possibilities in Optics by Stephen McGill & Dmitry Smirnov

Optical magneto-spectroscopy, from UV to THz wavelengths, is a powerful tool used to reveal details of electronic transitions between size-quantized states in low-dimensional structures. These optical experiments at very high magnetic fields are mostly performed in the optical-Faraday configuration (i.e. the incident light propagates parallel to the magnetic field). However, the 25 T Split Magnet permits experiments in the optical-Voigt geometry (i.e. the incident light propagates perpendicular to the magnetic field) opening up opportunities to explore transitions governed by different selection rules. This permits new experimental possibilities that merit attention. For example, in doped quantum well structures, the incident light couples directly to intersubband transitions if it is polarized in the direction of confinement and in the direction of applied magnetic field when exploring the effects of orbital (Landau) quantization. Another important case is found in the spectroscopy of carbon nanotubes. An axially applied magnetic field can be used to control the nanotube’s electronic structure through the Aharonov-Bohm effect. Since in the Faraday geometry the light polarized perpendicular to the nanotube axis is not absorbed, the Voigt configuration is therefore required to explore magneto-optical effects associated with intersubband transitions.

Non-optics research opportunities by Eric Palm

The first configuration of this magnet (the scattering configuration) will be ideal for optics users as mentioned above. In addition to direct optics experiments that will make use of this system, we expect that it will be very useful for Fourier transform infrared resonance (FTIR) experiments as the light path into and out of the magnet can be shortened greatly decreasing lost light and improving signal to noise. We also expect other novel experiments such as EMR or x-rays will make use of this magnet in this configuration. The second configuration has the magnet rotated as 90 degrees to the original orientation so that the field direction is parallel to the floor. Two of the scattering ports will be opened up to a minimum diameter of 32 mm. This will allow us to place a conventional dewar in this magnet so that the tails of the dewar are at right angles to the field. Thus when the dewar (or probe) is rotated about its axis, a sample in field center is rotated with respect to the magnetic field. This rotation configuration will enable angular dependent experiments with instrumentation that are not compatible with our current rotators, such as wave guides, high frequency rigid coax, or large heat capacity cells.

Above in dynamic light scattering, one measures the time correlation of the intensity of quasi-elastically scattered light. In liquid crystals, the main scattering comes from dielectric fluctuations associated with long-wavelength, collective rotational motions of the molecules. The time correlation function is directly related to the orientational elastic constants and associated viscosities of the liquid crystal. The number and type of collective modes contributing also reflects the symmetry – and therefore the structure – of the liquid crystal phase.
Dynamic light scattering in the Magnet Lab Split Magnet

Research Summary
Magnetic fields can be used to suppress fluctuations of orientational order in liquid crystals, and sufficiently high fields may also induce a transition from a state of no (or partial) orientational order to a more fully ordered state. In the first experiment in the Split-Helix magnet, we have collected data using photon correlation spectroscopy that demonstrates a field-induced transition from isotropic to orientationally ordered (nematic) phase and, additionally, suggests an unusual transition from a surface to bulk ordered nematic state. The measurements were carried out on 4-cyanoresorcinol bisbenzoate, a liquid crystal made-up of bent-shaped (bent-core) molecules.

Bent-core liquid crystals are particularly interesting because the reduced symmetry shape of the molecules may promote exotic forms of orientational order in a fluid system, including optically biaxial and polar nematic phases of potential impact in applications ranging from advanced electro-optic displays to “personal scale” green power generation. Light scattering data obtained via the unique capabilities of the Split-Helix may ultimately settle the question of the existence of these phases as a consequence of reduced molecular symmetry.

First split magnet user
Sam Sprunt, Kent State University

Title
Dynamic light scattering studies of bent-core liquid crystals in high magnetic fields.

Other participants
Jim Gleeson, physics professor, Kent State University

Synopsis of latest experiment, the first done in the 25 tesla Split Magnet system
The first experiment demonstrated the expected effect of high magnetic field on orientational fluctuations in rod-like, bent-core crystals, and demonstrated the exceptional performance of the new split-helix magnet for light scattering experiments for angular ranges up to 40 degrees.

Facility
Resistive Magnet Cell 5

Equipment
Split-helix magnet system; argon-ion laser, conveyed by a single-mode, optical fiber from Cell 1 to Cell 5; detection system consisting of multiple optical fibers, photomultiplier tubes, amplifier discriminators, digital electronic correlator; temperature-controlled sample insert for high field.

Techniques
Dynamic light scattering

Figures courtesy of Steve McGill (Magnet Lab) and Sam Sprunt (Kent State University)
Magnetic Circular Dichroism (MCD) in the Split Magnet: Bridging Quantum Chemistry to Solid State Physics

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2. National High Magnetic Field Laboratory, Tallahassee, FL, 32310

Access to optical probes is critical to accelerate progress in understanding high-field phases of complex materials. Optical techniques allow one to characterize the states of the charge, spin, and lattice subsystems on macro- to nano-length scales. Optical absorption, transmission, and reflectivity probe electronic configurations, while magneto-optical Kerr effects, second harmonic generation, and other nonlinear techniques reveal the symmetry of ferromagnetic, antiferromagnetic, and even ferrotoroidic magnetic orders. Raman, THz, FTIR, and X-ray techniques expose structural order, just to mention a small subset of their capabilities which extend to investigations of phase transitions, collective excitations of charge and spin, and AC conductivity. Sufficiently well-designed experiments are capable of recording the dynamics of these individual subsystems simultaneously using independent probes. This optically-oriented paradigm presents a transformative alternative to existing methods of materials investigation. In many cases progress is made by synthesizing materials and constructing phase diagrams as functions of doping, temperature, magnetic field, and pressure. Theorists are often compelled to make insightful suppositions at the important underlying quantum interactions and then compute theoretical phase diagrams in an attempt to reproduce the observations. This existing paradigm requires great quantum insight on the part of investigators because many existing high-field probes such as magnetometry, calorimetry, DC transport, etc. provide information on ensembles of coupled quantum states.

Now we are poised to introduce advanced spectroscopic techniques to systems in high magnetic fields where photons can probe individual quantum states and interactions. It is a fundamental quantum mechanical fact that the presence of a magnetic field lifts the orbital and/or spin degeneracy of electronic states. At the same time, conservation of angular momentum dictates that right or left circularly polarized photons absorbed by electronic systems couple selectively to electrons/excitons states with angular momentum +1 or -1. In a magnetic field this results in different absorption coefficients for right and left circularly polarized light. This difference, called Magnetic Circular Dichroism (MCD), is a direct measure of angular momentum of electrons that is very sensitive to changes in the environment around the electrons. As an experimental tool, MCD spectroscopy is employed in a wide range of studies from dynamic processes in biological molecules such as myoglobin, to spin exchange between magnetic dopants and conduction electrons in diluted magnetic semiconductors or semiconductor nanocrystals. In carbon-based systems where the spin-orbit coupling is very weak, MCD is a direct measure of the orbital angular momentum of the electronic state, whereas in systems with significant spin-orbit coupling, such as inorganic semiconductors, MCD measures the total angular momentum $J = L + S$ of electronic states (often referred to as "carrier spin").

The unique combination of high magnetic fields and free-space optical access, now possible in the new split magnet, opens the possibility of investigating magnetism and spin-dependent phenomena in systems with weaker spin-orbit coupling such as molecular crystals and wide-bandgap semiconductors. It is noteworthy that, for most of these systems, MCD levels are of the order of $10^{-4}$, a polarization difference that is impossible to measure with optical fibers, making the split the only magnet where this technique can be afforded for $B > 10$ T.

The system chosen for the first MCD experiments at $B < 27.5$ T was a crystalline Cu-Phthalocyanine (CuPc) thin film. Metal-Pc’s are popular molecules best known as p-type materials for organic photovoltaic applications. More recently low temperature ($T < 5$K) paramagnetism has been reported for this material. Since the CuPc crystallizes in a triclinic phase with the disk-shaped molecules stacked along the c-axis, the film resembles a collection of quasi-1D spin-$\frac{1}{2}$ chains since $Cu^{2+}$ has a single unpaired electron localized on its d-like orbital. It was hypothesized the observed paramagnetism originates from an indirect exchange between adjacent Cu spins, mediated by a double-degenerate delocalized state belonging to the $\pi$-electrons system of the Pc ligand. In this respect CuPc constitutes the organic analogue of a diluted magnetic semicon-
Our plan is to demonstrate that high field MCD is a powerful tool that can identify the electronic state responsible for magnetism in such systems.

Figure 1 is a photo of the optical setup we employed to measure high field MCD spectra in the split magnet. The narrow-band (~2nm) continuously tunable quasi monochromatic light provided by an Oriel Xenon lamp coupled to a 0.3m grating monochromator is collimated and directed to the sample using a custom 2f lens system. A combination of a linear polarizer and piezoelectric modulator (PEM) placed in the beam path modulate the beam’s circular polarization at a frequency of 50 kHz. The sample is mounted on a cold finger inserted in the magnet bore. A 45-degree mirror is mounted below the sample to achieve the Faraday geometry (\( K_{inc} \parallel B \)) required for this experiment. Light transmitted through the sample is collected using an optical fiber and focused onto a Si detector. We simultaneously record the average absorbance (\( A_{ave} \)) as well as the \( \frac{MCD \sim (A_{RCP} - A_{LCP})}{A_{ave}} \) by chopping the beam at low frequencies.

Figure 2 displays 300K MCD spectra from the CuPc films recorded at different magnetic fields. Each of the derivative-like features is associated with a distinct transition between states located at the bandgap of CuPc. All features are significantly broadened and redshifted in comparison to the ones observed in monomers. Since the MCD magnitude is proportional to the time-average of the total change in orbital momentum associated with a particular electronic transition and the electronic g-factor, it is expected that MCD increases linearly with applied magnetic field in the absence of any magnetic interactions. This is precisely what we observe at room temperature where MCD(B) (Figure 2-lower inset) can be very well fitted with a straight line. This dependence remains linear at 100K with a slight increase in slope which corresponds to an increase in the g-factor. The results are not surprising since carrier-mediated exchange is only expected to manifest itself at temperatures lower than 100K were not available in the split magnet at the time of the experiment, the data reported here holds great promise for future LHe experiments and proves that, for the first time, polarized beams can be delivered to and from the sample with minimal intensity and polarization losses that approach those of zero-field free-space optics experimental setups. There is no doubt that 5K experiments will be equally successful once the new custom cryostat becomes operational in the Fall of 2011.

Our experiments prove that optical spectroscopy in the split magnet will impact materials studies in a transformative way because it provides an exceptionally unique tool that investigates the quantum mechanical origins of macroscopic behavior. This is an area complementary to the classically-based probes familiar to the user community, that have a long and rich history with the previous generation of DC resistive magnets.

**Acknowledgements**

We are very grateful to prof. Randall Headrick and his Ph. D student, Ishviene Cour (Univ. of Vermont, Material Science) for generously sharing their experience in solution -based deposition methods for crystalline and discotic organic thin films. We thank prof. Rory Waterman and his postdoc, Anthony Wetherby (Univ. of Vermont, Chemistry) for imparting invaluable knowledge of recrystallization and purification techniques. Low field preliminary sample characterization was conducted in Furis’s NSF –sponsored 5T MCD setup (DMR #0821268). The Furis group was supported through NSF CAREER award DMR #1056589.

**REFERENCES**

Abstract
Information about protein structure in biological environment is scarce. To date, most membrane structure determinations have been carried out in detergent preparations and synthetic lipid bilayers, although the importance of membrane environment in supporting the native structure, dynamics, and function of membrane proteins has been well known. Using solid-state magic-angle-spinning NMR, a new approach in directly detecting a recombinant protein in native Escherichia coli membranes is demonstrated.

This work has been published in The Journal of American Chemical Society, DOI: 10.1021/ja204062v, Publication date (Web): July 21, 2011.

Introduction
Our structural knowledge of membrane proteins lags far behind that of soluble proteins, despite the fact that membrane proteins account for approximately 30% of all proteins in the human genome, including biologically crucial molecules such as ion channels and G-protein coupled receptors. As of August 2011, there are only ~300 unique membrane protein structures in the Protein Data Bank. Membrane proteins reside in a unique and complex lipid environment. While this environment is a major determinant of membrane protein conformation and function, it is incompatible with conventional methods of X-ray crystallography and solution NMR spectroscopy. Here, we demonstrate the feasibility of using solid state magic-angle-spinning (MAS) NMR for “in situ” structural characterization of the transmembrane domain (TM) of the human amyloid precursor protein (APP) binding protein LR11/SorLA in native Escherichia coli (E. coli) membranes.

Experimental
We produced human LR11 TM in E. coli using a MBP-fusion expression vector. The recombinant protein is expressed in the membranes at a much higher level relative to the background of E. coli membrane proteins. The fusion protein was cleaved at the native membrane surface, and the membrane fraction was isolated through ultracentrifugation and buffer washes for NMR experiments. LR11 TM comprises 70~80% of the total labeled proteins. NMR spectra were collected on a Bruker 600 MHz spectrometer at the Magnet Lab using a 4 mm or a homebuilt 3.2 mm low-E MAS probe.

Results
To examine sample homogeneity, spectral sensitivity and resolution, and the interference of background signals
from *E. coli* proteins and lipids, $^{13}$C MAS NMR spectra were acquired on a $^{13}$Ca,β-
Alanine enriched LR11 TM in isolated membranes. Figure 1 shows 1D $^{13}$C MAS
spectra of LR11 TM in native *E. coli* membranes using different polarization
methods. Direct polarization (DP) using a single 90° pulse (blue) excites magnetization
of all $^{13}$C spins (mobile and immobile). Despite the fact that lipids are not
labeled in this preparation, their naturally abundant signals overwhelm the 1D
spectrum. Cross polarization (CP) (red) enhances the signals from relatively rigid
parts such as TM while suppresses highly mobile signals. Double quantum filtered
CP (CP-DQF, black) effectively suppresses natural abundant $^{13}$C signals and thus de-
tects the resonances solely from $^{13}$Ca,β-
Ala labeled LR11 TM.

2D $^{13}$C PARIS$^2$ spectra with various mixing times were collected on a uni-
formly $^{13}$C, $^{15}$N enriched sample to pursue resonance assignments of the LR11 TM.
Figure 2 shows $^{13}$C-$^{13}$C PARIS spectra acquired with mixing times of 5 (left)
and 100 (right) ms, respectively. Using these data, we have assigned ~50% of the
LR11 TM residues. All assigned residues show characteristic secondary shifts of an
α-helix as expected for a transmembrane helix.

**REFERENCES**

Multivalued NMR $T_1$ of a Ferroelectric Metal–Organic Framework (MOF): Glassy Phase in a MOF Crystal

by T. Besara$^{1,2}$, P. Jain$^{1,2}$, N. S. Dalal$^{1,2}$, P. L. Kuhns$^1$, A. P. Reyes$^1$, H. W. Kroto$^2$, and A. K. Cheetham$^3$

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This report summarizes our discovery of novel NMR spin-lattice relaxation behavior in a family of metal–organic framework (MOF) compounds. MOFs are extended organic–inorganic crystalline solids with well-defined structures which often exhibit unusual properties promising advanced technological applications. Consequently, MOFs are the focus of much research attention today. The family of MOFs we have studied possess perovskite structures with cubic cavities which contain dimethylammonium (DMA) cations, $(\text{CH}_3)_2\text{NH}_2^+$. We found that the orientations of these cations are not fixed, and in fact a phase transition occurs at a temperature $T_c \sim 156$ K above which the nitrogen atoms can take up one of three distinct but equivalent positions (Figure 1). This ordering confers ferroelectric behavior to this compound — a unique property which has high potential for advanced technical applications. One such advance would involve the synergy that simultaneous electric and magnetic control of data storage/manipulation can give.

The goal of this study$^1$ was to understand the mechanism underlying the ferroelectric transition of the MOF $[(\text{CH}_3)_2\text{NH}_2]\text{Zn(HCOO)}_3$ (DMAZnF). An unexpected result was that single crystals of this lattice exhibit glassy behavior, the first such case for this class of MOFs$^1$.

The NMR relaxation rate ($T_1^{-1}$) measurements across the phase transition (Figure 2) display a well-rounded maximum in $T_1^{-1}$, an indication that the hopping motion of the DMA slows down smoothly at $T_c$ rather than completely freezing in, which would display a sharp divergence. This implies that thermal activation dominates the proton dynamics in the close vicinity of the phase transition. As the hopping slows down, the three N sites become inequivalent and the compound becomes ferroelectric.

Remarkably, the glassy behavior manifests itself in the relaxation measurements in the ferroelectric region of range $\sim 65$ K – $T_c$. Figure 3 displays two different relaxation paths in addition to the main path in this region. This behavior is linked to the thermal cycling of the sample. The relaxation is reversible over the range 65–250 K (main path), if the sample is not cooled below 40 K. However, the additional relaxation paths arise after cooling below this temperature and while warming the sample. Interestingly, these new relaxation paths do not follow a continuous curve; they undergo sudden jumps to the main path at unpredictable temperatures. After such a jump, further measurements of $T_1$ strictly follow the main path and display the same reversibility as before.

Since $T_1$ depends on the local geometry, the different relaxation paths taken up in subsequent thermal cycles show that the DMA cation finds distinctly different local environments depending on the sample cooling history. Further, since jumps from the metastable path to the main stable path are unpredictable it is implied that the various infrastructures attained on cooling below 40 K are very close in energy. The

FIGURE 1. Crystal structure of DMAZnF. The green spheres represent the three dynamically disordered nitrogen sites in the DMA cation.
existence of several different local domain structures with close-lying ground states is a typical manifestation of glassy behavior. The existence of a glassy state is confirmed by the $C_p$ measurements. For glasses, the phonon heat capacity at temperatures in the range of 2–30 K is generally larger than that predicted by the Debye $T^3$ law. The excess specific heat, indicated by a peak in the $C_p/T^3$ vs. $T$ curve (Figure 4), is a signature of glass dynamics. This observation suggests that the ferroelectric ordering was not the only cooperative phenomenon involved below $T_c$; there exists another order–disorder process, such as glass formation, at shorter length scales, below 40 K. We reiterate that this is first such observation for a MOF lattice, and should elicit further theoretical and experimental investigations.

We acknowledge financial support from European Research Council for an Advanced Investigator Award (to A.K.C.), from FSU (to H.W.K. and P.J.), and from HRH Sheikh Saud bin Saqr al Qasimi (to P.J.). The work at FSU was partially supported by the NSF Grant DMR-0506946. The work at NHMFL was performed under the auspices of the NSF through the Cooperative Agreement DMR-0654118 and the State of Florida.

REFERENCES

Theory and high-field pulsed EPR techniques are employed to study decoherence in the molecular magnet Fe₈. It is found that intrinsic decoherence sources can be significantly controlled through optimization of experimental conditions. Published in *Nature* 476 (7358), 76-79.

**Introduction**

Paramount to applications in quantum information storage and processing is the ability to fundamentally understand and control decoherence processes. Small couplings with the (fluctuating) local environment limit the stability of quantum states and lead to decoherence. Previous studies on molecular magnets have demonstrated quantum-coherent behavior; however, the sources that contribute to decoherence were not constrained. Decoherence in molecular qubit systems falls into two general categories: intrinsic (phonons, nuclear spins and dipolar interactions) and extrinsic (defects and impurities). Recently developed theory predicts that under optimized experimental conditions intrinsic decoherence sources can be substantially suppressed, while it was shown experimentally that high magnetic fields can substantially reduce decoherence processes in spin systems.

In the current study, crystalline Fe₈ single-molecule magnets (Figure 1) were employed because they have few of the impurities and defects that cause extrinsic decoherence, and because they exhibit a well-ordered spin array that allows us to calculate the intrinsic decoherence precisely. In crystalline molecular magnets, where each electronic spin behaves as a two-level quantum qubit, quantum coherence is observed in the collective (magnon) motion of the spins, rather than in single qubit dynamics such as that observed in NV centers in diamond.

**Experimental**

Single crystals of Fe₈ (natural isotope distribution and deuterated) were synthesized according to previously reported methods. Crystals of Fe₈ were face-indexed to facilitate an accurate field alignment. Hahn-echo Pulsed EPR experiments were carried out employing a 240 GHz spectrometer equipped with a 12.5 T superconducting magnet, quasiop-
Results and Discussion

Figure 2 shows the theoretically calculated intrinsic contributions and the experimental data plotted collectively, and the results agree extremely well without any adjustable parameters. It should be noted that the individual contributions from intrinsic decoherence sources are extremely sensitive to magnetic field strength, field orientation and temperature. This analysis indicates that for Fe8 single crystals with natural isotope distributions an optimum decoherence time ($T_2$) of 50 μs could be achieved, and that the decoherence time can be significantly extended (as high as ~500 μs) if nuclear isotopes are used to eliminate sources of nuclear-spin decoherence.

Conclusions

For the first time, decoherence mechanisms in complex molecular magnets have been isolated and controlled and that the theoretical predictions and experiment agree amazingly well. This study shows that it is essential to continue to expand and test current theory in order to gain a quantitative understanding of decoherence processes for future applications.

Acknowledgements

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REFERENCES

Spring & Summer Outreach

01 Inverness Middle School teacher and Research Experiences for Teachers participant Steve Crandell spent the summer working in Malli Warusawithana’s molecular beam epitaxy lab. “It’s amazing and wonderful and edifying,” said Crandell, who has been teaching at Inverness since 1987. “I’m really getting recharged this summer, ready to come back and try to share with other teachers and students.”

02 Mel Kochanowsky, a participant in this Spring’s Middle School mentorship program, demonstrates the thermal expansion properties of her rubber band engine. The Middle School Mentorship program pairs motivated students from Tallahassee’s School of Arts and Sciences with researchers at the lab, completing a ten-week project and familiarizing young people with lab environments and basic research.

03 Jorge Gonzalez of the University of Puerto Rico, Mayaguez, a Research Experiences for Undergraduates participant, spent his summer working with Eric Hellstrom of the Applied Superconductivity Center.

04 Alie MacVicar learns about facial reconstruction at the FSU Underwater Forensics School in Panama City Florida, as part of a SciGirls activity.

05 (L to R): Heather Kennedy, Trisha McCaul, Emmy Wycoff, Alie MacVicar, and Carina Richardson make a new friend at Seacrest Wolf Preserve. All were participants in SciGirls.

06 Terrie Kweito, an undergraduate at Virginia Tech, worked with CIMAR/ICR postdoctoral associate Vlad Lobodin during his Research Experiences for Undergraduates experience.

GET INVOLVED: For more information on summer opportunities with the Magnet Lab’s Center for Integrating Research and Learning, visit www.magnet.fsu.edu and select the “Education” tab.
What happens when good food, libations and science mix? Lively conversation. That’s been the premise — and the outcome so far — of Science Café in Tallahassee. The monthly cafés premiered last fall and, because of their popularity, will resume in September.

“The venue was great, and the audience was very warm and appreciative,” said Harry (“Sir Harold”) Kroto, a café presenter and a 1996 Nobel Laureate in Chemistry. “I enjoyed the opportunity to do something a bit different from my usual stuff.”

Science Café is held in the early evening (6:15 p.m. to 7:30 p.m.) at a popular tavern, Ray’s Steel City Saloon, in a special-events room that holds about 100 people. But this is no lecture-hall experience. The featured scientist’s initial presentation is brief — about 15 minutes — and followed by audience questions that steer the direction of the remaining hour-or-so-long conversation. Topics have included contaminants in drinking water, brain research, alternative energy, the physics of time, a deconstruction of the Deep Water Horizon oil spill, and more.

Each café has attracted an interesting mix of people: working adults (both scientists and nonscientists), retirees, college students and even several moms with babies. You may see people sporting tattoos and piercings near others wearing suits.

“Science education is critical, especially these days, so I think having an audience that’s diverse is a good thing,” said Penny Gilmer, a retired biochemistry professor who’s been to several of the cafés.

Joe Cain, who also attended several of the events, said the most memorable café for him was hosted by Scott Hannahs, a Magnet Lab physicist who talked about green energy without resorting to a powerpoint or other props — except, perhaps, the cold brew he sipped as he listened to audience members talk.

“I guess I remember that one,” Cain said, “because I participated and made my own comments.”

Which is exactly the point. Science cafés, a phenomenon launched in Europe in the 1990s, are purposely held in restaurants, bars and coffee shops in order to offer a relaxed atmosphere where nonscientists feel welcome and at-ease enough to ask questions.

“When you have a person up there with graphs and charts, that’s intimidating,” said geochemist Vincent Salters, who presented a café on chromium-6 in tap water. “When people ask a question, they don’t want to look dumb. I like that it’s causal because it breaks down the barriers.”

For the same reason, researchers who use plain language rather than jargon and who project a relaxed manner make the best hosts. During the conversation, people continue to eat their dinners and drinks are served. Presenters who can go with the flow and work around a cash-register ring will enjoy the venue. Others may not.

Most of the capital city’s cafés have been standing room only — an excellent sign of success! The tight quarters did, however, prompt consideration of a change in venue for the coming fall, which will include discussions on the collapse of bee colonies, climate change and energy. The logic behind the final decision on venue is perhaps best summed up by audience participant Cain.

“If you had someplace bigger, maybe a lecture hall, people could spread out and maybe they would be more comfortable,” the semiretired scientist said. “But you would lose something. … I think (the tight quarters) gets people closer together and makes it more personal.”

So for the coming year, at least, we’ll see you at Ray’s.
**HONORS & AWARDS**

Former user **Kirill Bolotin** has received an NSF career award. Bolotin, an assistant professor and formerly a postdoc with Philip Kim at Columbia, is an assistant professor at Vanderbilt. As explained by the National Science Foundation, “the Faculty Early Career Development (CA-REER) Program offers the National Science Foundation’s most prestigious awards in support of junior faculty who exemplify the role of teacher-scholars through outstanding research, excellent education and the integration of education and research within the context of the mission of their organizations.”

**Alan Marshall**, Robert O. Lawton Professor of Chemistry and Biochemistry and Director of the Ion Cyclotron Resonance Program at the National High Magnetic Field Laboratory, has received two major awards. One, the 2012 American Chemical Society New York Section William H. Nichols Medal, is presented annually in recognition of an outstanding contribution in the field of chemistry. The Nichols Medal is one of the oldest and most prestigious awards of the American Chemical Society: sixteen prior recipients have gone on to win the Nobel Prize in Chemistry. The Nichols gold medal will be presented at an award symposium in White Plains, NY in March 2012. The Pittsburgh Analytical Chemistry Award will be given to recognize Marshall’s significant contributions to the field of analytical chemistry. He will receive that award during Pittcon, the world’s largest conference and exposition on laboratory science, in Orlando, Fla., in March 2012. He will become only the fourth scientist to receive the three main awards of the SACP. The other two are the SACP Maurice F. Hasler Award, which Marshall won in 1997, and the Spectroscopy Society of Pittsburgh Spectroscopy Award, which he won in 2002.

**COMING & GOING**

Former ICR staff member and current External Advisory Committee member **Carol Nilsson** is leaving her position as a senior principal scientist at Pfizer’s Global & Research Development group to join the faculty at the University of Texas Medical Branch as a Professor of Pharmacology & Toxicology.

**WORTH NOTING**

**Vladimir Dobrosovic** is the recipient of the 2011 PAI award, administered by the Florida State University Department of Physics. The award recognizes faculty who have demonstrated excellence in both research and teaching.

**Danica Krstovka** has joined the Magnet Lab’s Condensed Matter/ Experimental group as a visiting scientist/researcher. She is a recipient of a 2010-2011 Fulbright Visiting Scholar award.

**Hanna Terletska**, a graduate student in the Condensed Matter/ Theory group, is the recipient of the 2011 Dirac-Hellman Award in Theoretical Physics, presented by the FSU Physics Department.

Got your own good news, job transition, etc. to share? Send it to winters@magnet.fsu.edu.
How do you think your experience at the lab has shaped your scientific career?

Postdoctoral research experience was a prerequisite for a tenure track faculty appointment. For an experimental physicist specializing in magnetism, there was no better place for a postdoc. I gained valuable experience in writing proposals, supervision of graduate students, and overseeing instrumentation development. Working with Alex Gurevich and David Larbalestier, I also had the opportunity to conduct research on the materials science of high temperature superconductors.

What’s the most important lesson you’ve taken away from this period of study?

I’ve played soccer for many years and trained in taekwondo so I am used to the idea of competition but science is the most competitive of all endeavors. Collaborative relationships with other scientists are important. Colleagues can play a key role in helping you get things done.

Describe someone at the lab whom you consider a mentor.

I have been extremely fortunate that at every stage of my development as a scientist, in spite of the challenges, there have been other senior scientists of very high standing who have been willing to give help and encouragement to get to the next level. Greg Boebinger stands out as one such scientist. Also, while at the MagLab, Justin Schwartz recognized how to best utilize my expertise and gave me the opportunity to gain new experience and take on additional responsibilities that are required of an academic scientist. He also expressed confidence in my potential to succeed at this level. Jan Jaroszynski’s broad range of knowledge and experience in the areas of experimental nanoscience, cryogenics, and the entire range of magnet systems available at the lab made him an invaluable resource.

What makes the magnet lab special?

The Magnet Lab is the premier magnet research and user facility in the world, offering many unique magnet capabilities but it’s the people who make the atmosphere within the lab an excellent research environment. Among many others too numerous to mention here in addition to the people at ASC, Eric Palm, Scott Hannahs, and Alice Hobbs of the DC User Program, Patrick Noyes, Jun Lu and Mark Bird of MS&T, Bryon Dalton, Larry Gordon, and Angel San Martin of the Control Room, Vaughan Williams and the staff of the Machine Shop, the Liquid Helium Supply staff, and at Los Alamos, Scott Baily, Fedor Balkirev, Ross McDonald, Chuck Meilke, Marcelo Jaime and Jon Betts of the Pulsed Field Facility are outstanding in carrying out the lab’s mission under Greg Boebinger’s leadership.
USER COMMITTEES

Magnet Lab Career Impact Survey

What first attracted you to the Magnet Lab?

- The unique facilities: 38%
- In-house experimentalists: 13%
- The opportunity to fast-start my career: 10%
- The chance to collaborate with other high-field scientists: 15%
- Right place, right time: 10%
- The experimental support team: 10%
- The Theory team: 2%
- Other*: 2%

Other answers:

- "To work with a specific scientist or scientists."
- "Access to high field NMR for study of proteins."
- "The external user program."

Is the Lab offering new opportunities?

Please rate innovation at the Lab on a scale of 1–10, with 1 being the lowest and 10 being the highest.

From what point in your career did you begin collaborating at the Magnet Lab and performing experiments at the Lab?

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<th>under-graduate</th>
<th>mid-career faculty member</th>
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<td>3% 27% Since grad school</td>
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<td>20% New faculty member</td>
<td>9% 14% As a full professor</td>
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Over the past three years, has the amount of magnet time allotted by the Magnet Lab been sufficient to meet your needs?

- Yes, I am fully satisfied
- I am satisfied, but could use more time
- No, I am definitely not getting enough time to meet my programmatic needs

Overall, how would you rate your experience while at the Mag Lab?

Use the same scale as before with 1 as the lowest and 10 as the highest.

Please estimate how much additional magnet time you could use (keeping quality the same)

- 100% more time
- 50% more time
- 25% more time
- None

Thanks to UT Knoxville’s Jan Musfeldt, chair of the User Committee, for creating and administering this survey.