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WIDE BORE 900 MHz PROJECT BUCKET TEST

&

Ultrasound Measurements in Pulsed High Magnetic Fields

Summer 2001 Research Experiences for Undergraduates & Teachers

Three-Dimensional Homonuclear NMR Spectra

AMRIS Progress & Research Report #7

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On the Cover: Some of the team members of the 900 MHz project from left to right, top to bottom: George Miller, Ian Dixon, Scott Marshall, Denis Markiewicz, Tom Painter, Ken Pickard, Ed Miller, and Lee Marks. *Photos by Bob Burke, collage by Walter Thorner*

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From the Director's Desk

Jack Crow

NHMFL conference activity

ebbs and flows from year to year as many events are held only every two or three years. In 1998-99, for example, the NHMFL sponsored, hosted, or supported nearly a dozen significant events. The *VIII*th *International Conference on Megagauss Magnetic Field Generation and Related Topics*, *Physical Phenomena at High Magnetic Fields-III*, the *Second North American FT-ICR Mass Spectrometry Conference*, and additional workshops attracted thousands of visitors from all over the world. We were kept extremely busy developing quality conference programs, engaging the broad science communities, and showcasing the laboratory's world-class facilities. A brief respite from major events during 2000 allowed us to hold smaller meetings focused on specific science topics and user needs. These gatherings were attended by members of the NHMFL Users' and External Advisory Committees (and others) and were critically important to development of the laboratory's successful renewal proposal and for charting the laboratory's course for the next five years.

This year, conferences and workshops are again a priority. In mid-March, the *Third Biennial North American Fourier Transform Ion Cyclotron Resonance Mass Spectrometry Conference* was held in Austin, Texas. Attended by approximately 100 scientists, the program featured 24 invited speakers from the United States, Sweden, England, Germany, Switzerland, Canada, and Denmark, including a feature lecture by Professor Fred McLafferty of Cornell University. Several dozen posters, mostly contributed by graduate students and postdoctoral fellows, were also presented. The conference was organized by NHMFL's Drs. Mark Emmett and Chris Hendrickson, and sponsored by NSF, Bruker Daltonics, IonSpec, Finnigan-Thermo, and Oxford Instruments.

On August 13-15, a 60th birthday party of sorts will be held for NHMFL faculty member Zachary Fisk. *The Future of Materials Physics: A Festschrift for Zachary Fisk*, being organized by colleagues at LANL, will bring together a distinguished group of international leaders in materials physics to explore the frontiers in theoretical and experimental research of the complexity of solids. Dr. Fisk served as chair of the In-House Research Program for two years, is a LANL and APS Fellow, and a member of the National Academy of Sciences.

The laboratory is also co-sponsoring several events: the *24th International EPR Symposium* and the *NMR Symposium*, both to be held in conjunction with the 43rd Annual Rocky Mountain Conference in early August in Denver, Colorado; the *Fifth Latin American Workshop on Magnetism and Magnetic Materials and their Applications*, in September in Argentina; and the *2001 Southeastern Magnetic Resonance Conference* in October at the University of Florida.

Activity peaks in October this year when the laboratory co-hosts the *6th International Symposium on Magnetic Suspension Technology* with the Technical University of Turin in Italy, and hosts the *J. Robert Schrieffer Anniversary Symposium* and the *Physical Phenomena at High Magnetic Fields IV* conference in Santa Fe, New Mexico.

Over 100 people have already registered for ISMST6, which is a significant increase from previous years that reflects the growing interest in magnetic suspension-related technologies such as magnetic bearings, electromagnetic launchers, power and control systems, and wind tunnel model suspensions. An exciting new area to be discussed is materials processing and biological studies in low or zero gravity by magnetic suspension. It is interesting to note that 25 percent of the early registrants for ISMST6 are from Japan.

The signature event for the year will surely be the special symposium in honor of NHMFL Chief Scientist and Nobel Laureate Bob Schrieffer, who celebrated his 70th birthday earlier this year. Bob's life-long dedication to science, his leadership within the physics community, and his commitment to the laboratory have been extraordinary. Bob was the first faculty appointment of the NHMFL, and his early affiliation helped shape the laboratory and attract numerous other senior scientists in various disciplines. Under his leadership, the NHMFL has established and nurtured a world-class multi-disciplinary research program, with a collegial spirit that I have experienced nowhere else. On a personal note, those of us at the laboratory in Tallahassee are honored to consider Bob and his wife Anna friends, and thank them for giving so generously of their time, talents, and energies to our community.

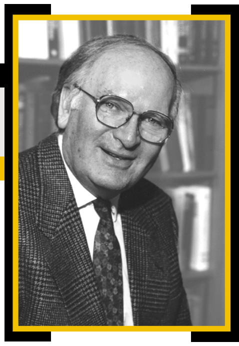
The Schrieffer Symposium on October 18-19 kicks off PPHMF-IV (October 19-25), the major international conference Bob has guided since 1991 shortly after joining the lab. We are pleased to hold this year's conference in New Mexico so that attendees may tour the NHMFL Pulsed Field Facility, including the New Experimental Hall, the 1.4 GVA motor generator, and other outstanding user facilities.

There is still time to register for the fall conferences and I encourage you to do so. For further information, see page 21 or visit our Web site, *www.magnet.fsu.edu*.

I look forward to seeing you.

Director

From the Chief Scientist's Desk



J. Robert Schrieffer

Chuck Mielke and co-workers have explored the important question of whether coherent versus incoherent motion of electrons occurs between planes of organic superconductors. In the temperature range studied between 0.5 K and 5 K, they find strong coherent motion. In addition they find evidence for two-dimensional behavior at low temperature and three-dimensional behavior at high temperature due to the superconductor coherence length being small compared to the interlayer spacing at low temperature and larger than the spacing at high temperature. Furthermore, the transfer integral for electron motion between planes is much larger for their material compared to that studied in a related material corresponding to a smaller interlayer spacing in the former system. These results are important in guiding theory in these interesting materials.

Superconductivity and the Dimensionality of Electronic Bandstructure

Chuck Mielke, NHMFL-LANL
John Singleton, NHMFL-LANL
Neil Harrison, NHMFL-LANL
L.K. Montgomery, Indiana University, Chemistry
Dwight Rickel, NHMFL-LANL

Many of the most interesting materials studied today—cuprate superconductors, ruthenates, organic superconductors—have very anisotropic electronic properties. Indeed, it has been proposed that scattering or thermal fluctuations result in "interlayer incoherence" in most of these systems.[1] In other words, the electronic bands become well defined only in the planes of the layers, and the material becomes a series of weakly-connected two-dimensional systems.

A recent issue of this journal[2] reported the observation of a small peak in the resistivity of the layered organic superconductor κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ when a magnetic field (of 45 T, from the NHMFL Hybrid) was applied exactly within the layers.[2,3] This peak is a robust demonstration that the interlayer transport in this material is coherent, contrary to the expectations mentioned above. Subsequent work has shown[3] that the angular width of this peak is directly proportional to the interlayer transfer integral t, which describes the motion of the conduction electrons in the interlayer direction. Although the value of t derived from the experiments was very small (0.04 meV, equivalent to about 0.5 K), the material nevertheless showed coherent interlayer transport up to at least 5 K.[3]

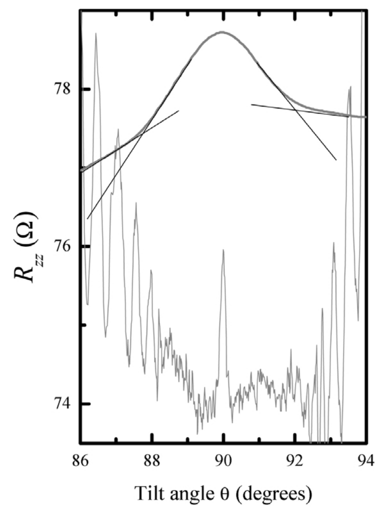


Figure 1. Peaks in the interplane resistance Rzz observed when the magnetic field is close to the in-plane orientation (θ =90°). Data are shown for λ -(BETS)$_2$GaCl$_4$ (thick line; T=1.4 K, B=30 T)[4] and κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ (fine line; T=520 mK, B=42 T).[3] The rapid oscillations at the edges of the figure are angle-dependent magnetoresistance oscillations (AMROs). Note that the peak in κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ is much narrower than that in λ -(BETS)$_2$GaCl$_4$, showing that the interlayer transfer integral is much smaller in the former material.

The same technique has been applied to another layered organic superconductor, λ-(BETS)$_2$GaCl$_4$. Within the planes of the layers, κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ and λ-(BETS)$_2$GaCl$_4$ have very similar electronic bandstructures.[4] However, Fig. 1 shows immediately the contrast between the two superconductors in *the interlayer direction*. The peak feature reported in references 1 and 2 is seen in both materials; however the angular width of the peak for λ-(BETS)$_2$GaCl$_4$ is approximately ten times the width of that in κ-(BEDT-TTF)$_2$Cu(NCS)$_2$, implying that t is ~ ten times larger in the former material (i.e. t = 0.4 meV).[4]

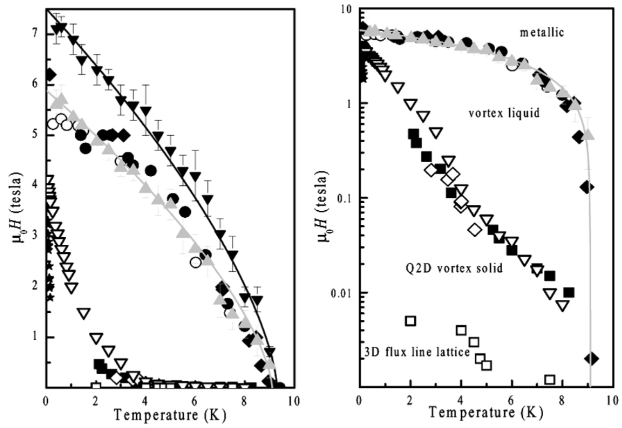


Figure 2. Critical fields in κ-(BEDT-TTF)2Cu(NCS)2, plotted on linear (left-hand side) and logarithmic (centre) field scales; the magnetic field B is perpendicular to the layers. The data for Hc_2 comprise filled triangles (LANL MHz penetration studies, midpoint),[4] inverted filled triangles (LANL MHz penetration studies, onset),[4] filled circles (microwave penetration studies),[5] open circles (thermal conductivity data),[5] and shaded diamonds (magnetization data) (see citations within reference 4). The solid curve is proportional to $(T_c\text{-}T)^{2/3}$. The triangles are the irreversibility field from magnetization; the filled squares and stars represent 2D melting from magnetometry and GHz studies. The hollow squares are from muon-spin rotation and denote the 3D-2D transition (see reference 4 for sources of data).

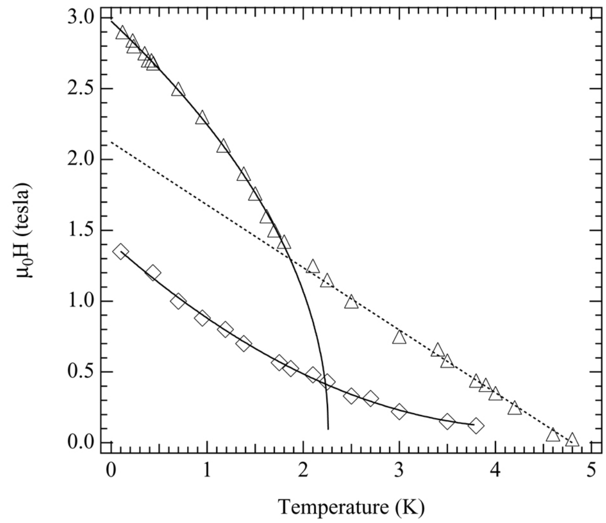


Figure 3. Equivalent phase diagram of λ -$(BETS)_2GaCl_4$ deduced using the LANL MHz penetration-depth technique. Hc2 (triangles) and the melting curve (diamonds) are both shown.[4] Note the dimensional crossover in Hc_2 at about 2 T.[4]

Thus, as far as the electrons are concerned, λ-$(BETS)_2GaCl_4$ is a much more three-dimensional material.[4]

Does this matter? To answer this question, we turn to a MHz penetration-depth technique for measuring the phase diagrams of layered superconductors, developed by Chuck Mielke of LANL. Conventional resistive studies of the upper critical fields in cuprate and organic superconductors are problematic, as dissipative flux motion severely affects the measurement (errors of ~ 50% in H_{c2} are not uncommon).[5] To obtain reliable values of H_{c2}, the accepted wisdom is that one turns to probes such as thermal conductivity and GHz cavity measurements,[5] techniques not always suitable for the pulsed magnetic fields found at NHMFL-LANL. Fig. 2 shows H_{c2} in κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ derived from several different techniques; note that the data obtained using the LANL MHz penetration depth technique are in excellent agreement with the other, more time-consuming techniques, allowing the complete phase diagram to be mapped out. (Recent work has also shown that these MHz techniques work flawlessly on cuprate superconductors in pulsed magnetic fields of up to 60 T).[6]

Fig. 3 shows the superconducting phase diagram of λ-$(BETS)_2GaCl_4$, measured using the LANL MHz technique. Note that H_{c2} shows a very distinct change in behavior at about 2 T; this represents a change from two dimensional (low temperature) to three dimensional (high temperature).[4]

By contrast, the three-dimensional behavior in κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ is restricted to a tiny corner of the phase diagram (see Fig. 2). Thus, the greater three-dimensionality of the electronic properties of λ-$(BETS)_2GaCl_4$ directly affect the superconducting groundstate, resulting in more three-dimensional superconducting properties compared to those of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$.

In summary, these experiments demonstrate that the dimensionality of the electronic bandstructure in organic superconductors may be characterized in a quantitative fashion via the interlayer transfer integral *t*. Moreover, the complete superconducting phase diagram may be probed reliably using MHz techniques. These data show that the effective dimensionality of the superconducting state is determined by that of the underlying bandstructure.

Such experiments underline the satisfying nature of organic superconductors. The availability of a large number of these systems of varying dimensionality (and well-characterized bandstructure) should potentially allow very stringent tests of models of superconductivity in layered materials to be carried out.[7]

1 For a review of such effects, see L.B. Ioffe and A.J. Millis, *Science*, **285**, 1241 (2000); D.G. Clarke and S.P. Strong, Adv. Phys., 46, 545 (1997).
2 NHMFL Reports, **8**, 2, 6 (2001).
3 J. Singleton, P.A. Goddard, *et al.*, cond-mat/0104570.
4 C.H. Mielke, J. Singleton, *et al.*, cond-mat/0103501, *J. Phys.: Condens. Mat.*, in press.
5 S. Belin, T. Shibauchi, K. Behnia and T. Tamegai, *J. of Superconductivity*, **12**, 497 (1999).
6 C.H. Mielke, J. Singleton, *et al.*, in preparation.
7 For a review, see J. Singleton, *Rep. Progr. Physics*, *63*, 1111 (2000).

Wide Bore 900 MHz Project Bucket Test

The wide bore 900 MHz project will soon be tested in the bucket cryostat in Cell 16 of the NHMFL DC Field Facility to verify performance of the magnet assembly and allow for any minor adjustments that may be required prior to installation in its final cryostat. Shortly thereafter, the magnet will be placed into the final cryostat and installed in the NMR wing of the laboratory.

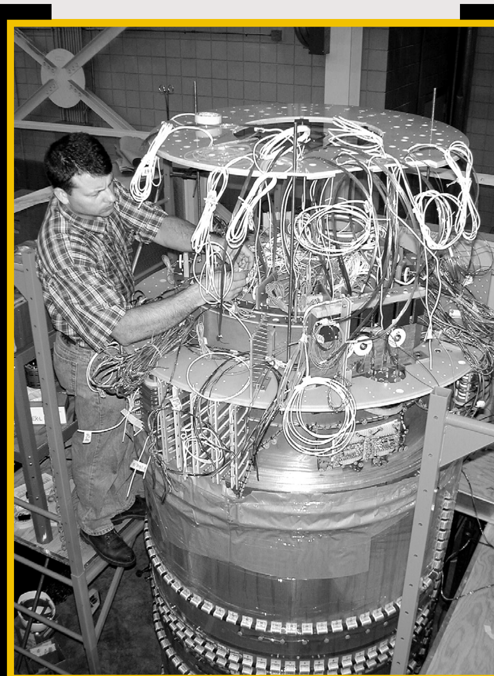


Figure 1. The final wiring on the 900 MHz magnet assembly is presently being completed.

The four critical performance parameters of an NMR system are (1) the maximum operating field, (2) the rate of decay of the field, commonly referred to as the persistence, (3) the field homogeneity over a specified volume at the bore center of the magnet and (4) the cryogenic hold time or the time allowed before the cryogens must be refilled. The object of the bucket test is to verify the basic operating parameters of the magnet in a cryostat that can be readily opened. This provides an opportunity to demonstrate magnet operation and go through the training period of the magnet in an operating environment that will allow relatively easy access to the magnet if trouble is detected. The bucket test is, of necessity, somewhat limited, particularly in the operating temperature to be achieved. The magnet is designed to operate at 1.8 K in the permanent cryostat. The bucket test, due to limitations in the size of the cryostat and the desire to maintain a facility that permits relatively easy access to the magnet, will achieve an operating temperature of about 2.2 K. After a successful bucket test, the magnet will then be installed in the permanent cryostat, which is welded together to minimize the potential for vacuum leaks that would affect the long-term operation of the magnet.

Because of the operating temperature limitations and the magnetic environment of Cell 16, the bucket test will not necessarily demonstrate the final operating specifications of the magnet. The margin in the critical current of the magnet will allow us to reach very close to if not achieve the design maximum operating field of the magnet. The bucket test conditions should show demonstration of magnet persistence, but the magnetic background environment in Cell 16 may not permit demonstration of the final spatial homogeneity. Finally, the cryogenic hold time is a performance parameter of the final cryostat and will not be verified in the bucket test. The bucket test will also demonstrate the operation of all the auxiliary systems, such as the bucking coils, the quench heater system, the superconducting shim system, the quench detection system, and the magnet operating and control systems.

The bucket test comprises three main systems: the magnet assembly, the cryogenics, and the instrumentation/quench detection. The progress and present status of these are described below.

The magnet assembly has seen much progress since last summer including receipt of the five NbTi coils and superconducting shim set from Intermagnetics General Corporation, final fabrication of the five Nb_3Sn coils here at the NHMFL, completion of the production of the superconducting switches, diode packs, shunt resistors, bucking coils, and all the other hardware required for magnet assembly. Presently, the magnet is undergoing its final wiring as shown in Fig. 1. The cryogenic system comprises the baffle assembly (shown being assembled in Fig. 2), the bucket cryostat, helium liquefier, and vacuum system. All of these systems have been designed, hardware procured, and are in the final stages of assembly. The instrumentation and quench detection system comprises (1) the fuse boxes to isolate the operators from possible high voltages coming from the magnet assembly, (2) two quench detection systems, one main and one redundant, which detect unsafe, non-superconducting voltages and trigger the quench heaters to safely discharge the magnet, (3) various power supplies, including capacitive power supplies to power the quench heaters, and (4) the computers required to collect and record all the instrumentation on the magnet assembly. All of these systems have been designed, hardware procured, and are in the final stages of assembly.

Only a few tasks remain before the bucket test can begin. After the wiring is completed on the magnet assembly, it will be connected to the bucket test baffle system, followed by quality control checks of the wiring. Next, the instrumentation and quench detection systems will be connected, followed by more checks. Finally, the magnet will be inserted into the bucket cryostat and cooldown to operating temperatures will commence.

We look forward to a successful test and the commissioning of this very unique ultra-wide bore 900 MHz NMR magnet.

Figure 2. The bucket test baffle system is shown here undergoing its final assembly.

This article was contributed by Tom Painter and Jim Ferner, who may be contacted for further information. (850-644-5752, *painter@magnet.fsu.edu.*)

ATTENTION USERS

Alex H. Lacerda
Director, NHMFL-Los Alamos User Program

It is with great pleasure that I bring to your attention comments on the following article, "Ultrasound Measurements in Pulsed High Magnetic Fields," by B. K. Sarma, A. Suslov, and J. B. Ketterson. For the past decade, ultrasound measurements have proven to be of great importance in the investigation of systems close to magnetic instabilities. Many interesting results can be found in the literature (at reasonably high DC fields) dealing with systems presenting metamagnetic transitions where ultrasound measurements provided important information regarding the electron-lattice coupling. The group Ketterson, Suslov, and Sarma has been the first in the United States to extend this technique to be used in pulsed magnets. Their report that follows describes experimental details of the technique and presents results regarding the lattice behavior around the 35 T metamagnetic transition of the heavy fermion compound URu_2Si_2. I am sure that many of you will find the article very interesting. We are working hard to make this technique available to the user community soon.

Ultrasound Measurements in Pulsed High Magnetic Fields

Bimal K. Sarma, University of Wisconsin-Milwaukee, Physics
Alexei Suslov, University of Wisconsin-Milwaukee, Physics
John B. Ketterson, Northwestern University, Physics

Ultrasonic velocity and attenuation measurements are powerful tools to study condensed matter systems,[1] especially various phase transitions and collective phenomena. Sound waves, which can be regarded as long wavelength phonons, can couple with the electrons or other collective excitations: the resultant loss in amplitude (attenuation) and/or change in the velocity gives information about the possible physical processes taking place. Useful parameters to control are the temperature, applied magnetic field, and pressure. Our group has constructed a unique ultrasonic spectrometer specifically for the study of condensed matter systems at the intense pulsed magnetic fields at NHMFL-LANL and have used it to study the metamagnetism in UPt_3 and URu_2Si_2.

The instrument is a computer-controlled, phase-sensitive, ultrasonic spectrometer, which works as follows: the r.f. output of a synchronizer is split into two channels by a power splitter. One of these channels is gated and after passing through an r.f. power amplifier, sent to the cryostat. The r.f. pulse is converted to an acoustic pulse via a piezoelectric transducer bonded to one side of the sample under study. This generates an "echo train" which is detected via a second transducer bonded to the opposite side of the sample. This received r.f. signal is amplified and mixed with a local oscillator, the output of which is further amplified after which it is applied to a phase sensitive detector with in-phase and quadrature outputs. The reference signal for the phase sensitive detector is obtained by independently mixing the second channel from the synchronizer with the local oscillator.

The two outputs of the phase detector are averaged (by separate boxcar integrators), digitized, and stored in the computer. The amplitude and phase are then computed yielding immediately changes in the attenuation and velocity. Alternatively one can use the output of the 90° channel as a

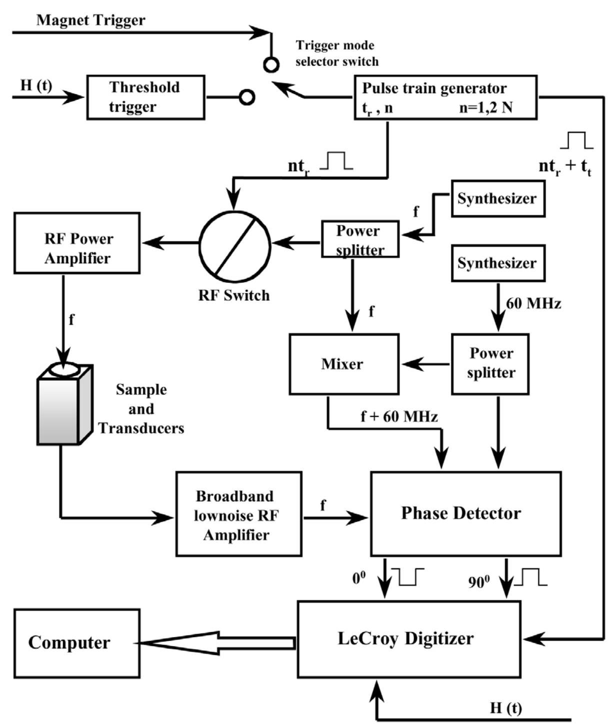


Figure 1. Block diagram of spectrometer.

feedback signal to shift the frequency of the synthesizer to null this channel. The in-phase output then gives the attenuation change directly while the velocity change can be computed from the synchronizer frequency shift. For very large velocity shifts, one must be concerned that the frequency does not stray outside the bandwidth of the transducers since this would result in a spurious attenuation and velocity (phase) shift. This problem can be addressed by periodically incrementing the synchronizer frequency such that a phase shift of 2π is introduced in the received signal and introducing the appropriate corrections into the data analysis.

The above procedures allow the detection of attenuation and velocity changes corresponding to .01 dB and a part in 10^7 respectively. A schematic of a typical electronic setup is shown in Fig. 1. Many variations are possible, the choice depending on the experiment at hand. By assembling the detector system from individual r.f. components, the instrument has maximal versatility and can adapt to a wide variety of experimental conditions. In sound velocity measurements using the cw (continuous wave) method, the resonant frequency is measured to determine changes in sound velocity. This technique enables one to achieve a very high resolution in sound velocity measurements, and since this is also a low power measurement,

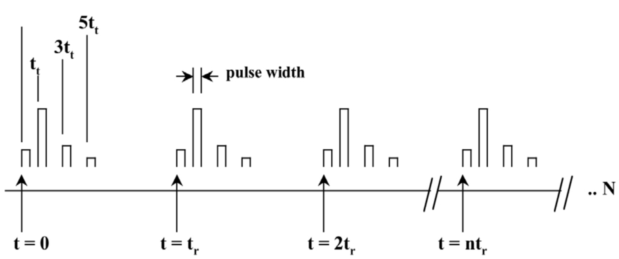


Figure 2. A train of N pulses.

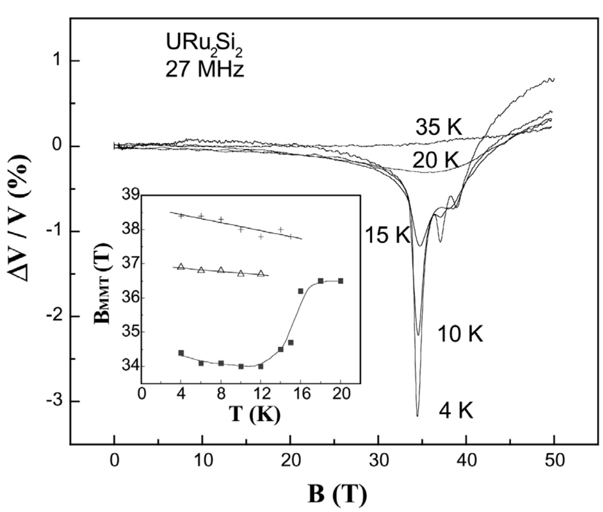


Figure 3. Longitudinal velocity at the metamagnetic transition in URu_2Si_2.

it is particularly suited for low temperature measurements. The measurement technique involves frequency modulation, and the detection is enhanced by the use of lock-in amplifiers.

For the pulsed magnetic field studies at the Los Alamos site, additional measurement features are required. The short pulse magnets (20 ms 50 T or 60 T) require approximately 30 to 40 minutes to recover between pulses. In order to collect the maximum amount of data per pulse, it is desirable to: (1) digitize and store all echoes in a given pulse train and (2) launch as many pulse trains as possible. Typically the ultrasonic pulses were sent at a frequency of 100 kHz or 10 μsec between pulses. During this time, the magnetic field changes by 50 mT.

The second requirement implies that a new pulse train will be launched as soon as the previous one has disappeared into the noise, corresponding to about 3 to 4 decay times for the pulse train. At such high, short-term duty cycles one must be concerned that the r.f. output does not fall off with successive pulses. Since typical ultrasonic pulsers draw energy from a capacitor during the pulse (which is recharged by a power supply of limited capacity) they would not be suitable for the present purpose. Hence, we require an r.f. amplifier (amplifier research has various power amplifiers covering a broad frequency range) capable of delivering a high average power. The first requirement is common to all pulsed field measurements. We use the LeCroy 6810/6310 digitizing systems at the Los Alamos site. During the magnet pulse, a rapid succession of ultrasonic pulses is sent into the sample, and all the information stored. The data is analyzed to obtain the velocity and attenuation information that is then transferred to the computer and stored, freeing up the LeCroy system for the next set of pulses.

For measurements in pulsed magnetic fields, the spectrometer has been used in a second configuration, which we call a digital spectrometer. The IF frequency of 12.5 MHz, is extracted by a low pass filter and is digitized by a GaGe-CS 1250 oscilloscope card. The sampling rate is 50 MHz, which allows us to measure four points per IF period. The signal is stored in the memory of the oscilloscope card and later transferred to the computer. The mixing and detecting functions are now performed digitally.

Typically, the high accuracy and sensitivity in the ultrasonic measurements is achieved by signal averaging over many pulses; thus, requiring a slow sweep rate of the magnetic field. Typical sweep times in the DC fields at Tallahassee were 60 minutes to full field. In the short pulse magnets no signal averaging is possible since the magnetic field changes by as much as 0.2 T between the r.f . pulses. Also, the echo train should be short, so that there is no overlap between echoes of neighboring pulses.

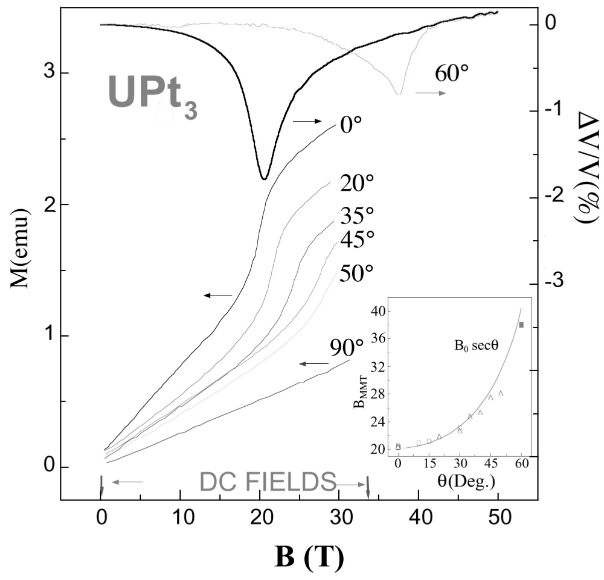


Figure 4. Anisotropy of the metamagnetic effect in UPt_3.

Ultrasonic experiments generally require large single crystals, and this gives rise to the possibility of appreciable joule heating during the magnet pulse. Care has to be taken in our sample holder design to minimize any additional joule heating. The sample is bathed in helium for maximum cooling and heat exchange during the magnet pulse.

This spectrometer has been used in the study of the heavy-fermion materials UPt_3 and URu_2Si_2 in the 20 ms 50 T pulsed magnets at NHMFL-LANL. Both of these systems show metamagnetism at high fields, an enhanced magnetization accompanied with a sharp velocity dip. Ultrasonic velocity measurements were done on a single crystal URu_2Si_2 at various temperatures. At 4 K, a three-fold splitting in the metamagnetic transition is seen (Fig. 3) agreeing with earlier work[2] on magnetization measurements. The temperature evolution of this splitting is shown in the inset. As the temperature is raised, the three-fold splitting merges to two and finally one.

Fig. 4 shows both magnetization and velocity measurements in UPt_3. Lower curves are magnetization measurements done with a Lake Shore Vibrating Sample Magnetometer on two UPt_3 samples: a ***sphere*** (open circles) and a ***rectangular piece*** (open triangles), in the ***30 to 33*** T DC fields at the NHMFL in Tallahassee. The magnetic field is applied at an angle θ° from the basal plane. The upper curves are ultrasonic velocity measurements performed on a ***20 millisecond 50*** T pulsed magnet at the NHMFL, Los Alamos facility, and at angles ***0***° and ***60***°. At the metamagnetic transition, B_{MMT}, there is an increase (a step) in the magnetization and a sharp dip in the velocity.

The magnetic field at which this occurs is anisotropic, and the anisotropy is shown in the inset. The solid line is B_0 ***sec***θ, the field required to yield a component of B_0 in the basal plane. The velocity data is shown as solid squares. The ***60***° data shows the strength of the velocity measurement technique in a pulsed field. Magnetization measurements would be very difficult because of the large dB/dt (rate of change of flux) through the pick-up coils.

This research is supported by NSF through Grant No.s DMR-9971123 and DMR-9704020, and the NHMFL, Tallahassee and Los Alamos.

1 Sarma, B.K., *et al.*, *Physical Acoustics*, **20** (Ed. by M. Levy, Academic Press, 1991).
2 Sugiyama, K., *et al.*, *J. Phys. Soc. Jpn.*, **68**, 3394, (1999).

Bruce Brandt
Director, NHMFL DC Field Facility User Program

We are delighted that the portable dilution refrigerator has been made to operate in the Hybrid magnet. Vaughn Williams designed a new stand so that the refrigerator could be aligned in and mechanically isolated from the magnet. Eric Palm and Tim Murphy extended pumping lines and control cables to move critical parts into outsert fringe fields low enough to allow them to work properly, assembled the components, and got it all to work. The refrigerator has run successfully for six weeks. It was removed in early July to make room for other experiments and to allow some work to be done to lower the base temperature. It will run again this fall in either a 33 T magnet or the Hybrid depending on the science to be done.

Hybrid users were limited to 42 T while we waited for the last part needed to build a complete set of spare insert coils. The set was completed in late July, and users have been given limited numbers of sweeps to 45 T since then. We hope to offer unlimited operation at 45 T by December when the redesigned insert is on hand.

Users of the NHMFL:

The recent successes of the NHMFL outlined in this and previous newsletters are the product of inspiration and dedication from both staff and users of the Laboratory. The users can be particularly proud of their achievements. We have performed excellent science at the NHMFL and we have made recommendations that have guided new directions and improvements in capability and infrastructure. The User Committee is elected by the user community, broadly defined, to bring your concerns to the attention of our community and to the Laboratory management, the Laboratory Advisory Committee, and the review committees of the NSF. I welcome news of your research successes and suggestions for improvements.
Send to: *w-halperin@northwestern.edu*

Bill Halperin, chair
Users Committee

Summer 2001 Research Experiences for Undergraduates and Teachers

While most education facilities slow down during the summer months, the NHMFL's Center for Integrating Research and Learning is more active than ever. Tours, outreach, and teacher workshops continue even as the Center hosts 17 students and 17 teachers through its Research Experiences for Undergraduates (REU) and Research Experiences for Teachers (RET) Programs. In addition, two high school students and one middle school student are earning credit for their work with mentors at the NHMFL. The Center is also preparing for an extremely active fall schedule of statewide teacher workshops and classes for fourth graders from local schools.

Successful Center programs depend upon the relationship among teachers, scientists, researchers, and Center staff. Without a willing and committed cadre of dedicated mentors, the REU and RET programs could not exist. Any scientist or researcher who has mentored students or teachers can tell you that it requires dedication and a great deal of time. REU mentors provide a structured research experience that can culminate in a published article with students as co-authors. NHMFL mentors at all three sites routinely provide meaningful experiences for students, spending hundreds of hours making sure that students complete their work, present their results, and learn about careers in science. RET mentors have a commitment to public education, the enhancement of science education, and the role of teachers as translators for the science community. We are extremely pleased to recognize our student REU participants, teacher RET participants, and mentors in the table (on page 12).

In addition to the REU and RET program participants, the laboratory integrated three secondary-level students into ongoing research and engineering activities: Nathan Scott, rising senior, worked with Danny Crook in MS&T; John Reilly, senior, worked with George Miller in MS&T; and Chase Pheifer, 8th grade, worked with Dianne Walker in an affiliated program, the Sensory Research Institute.

Science, Tobacco & You workshops continue nationwide and the Center is anticipating five more sessions in the State of Florida during the 2001-2002 academic year. In addition to teacher workshops during the school year, Center educators are teaming up with area teachers, the FSU Physics Department, and the Office of Science Teaching Activities at FSU to facilitate a 2-week summer institute on Motion, Forces, and Energy. NHMFL sessions will include a tour of the laboratory and hands-on activities and demonstrations on magnets, magnetism, electricity, and related concepts.

Pat Dixon, Director of CIRL, Sam Spiegel, former Director of CIRL, and Gina Lafrazza-Hickey, Education Specialist, recently published "Looking, Thinking, Asking, Learning How Your Body Works." A large-format book for students and teachers on human anatomy, the book was created to fill a gap for elementary classrooms studying the respiratory, circulatory, nervous, and muscular systems and to advance science education through NHMFL resources. Another publication, "Eroding Brick and Bureaucratic Walls: Oral and Written Interaction as Means of Curriculum Coherence," appeared in the Spring 2001 issue of the *Journal for the Art of Teaching*, co-authored by Pat Dixon.

The Center is looking forward to expanding its outreach to area students and teachers during the 2001-2002 academic year.

Table 1. Summer 2001 REU and RET participants and mentors.

Student	Home Institution	Mentor	Research
Adam Abate	Harvard University	Stan Tozer	Fermi measurements on the lanthinum 218s
Jill Adcox	University of Florida	John Eyler	Inductively coupled plasma Fourier transform ion cyclotron resonance
Joshua Alwood	University of Florida	Gregory Stewart	Specfic heat measurements of the $U_2 (Ni_xCO_{1-x})^2$ in system
Norman Anderson	Iowa State	Alex Lacerda	Experimental techniques in low temperature physics
Tom Bemben	University of Florida	Alex Angerhofer	The effects of bond length on g-factors with liganded chlorophyll a and Bacteriochlorophyll
Jiawen Chen	Cornell University	Scott Smith	A python interface to the “GAMMA” magnetic resonance library
Rick Clinite	Cornell University	James Brooks	Temperature dependence in organic conductors
Alisha Elsebough	Florida State University	Roy Odom	Techniques for creating specified magnetic fields with superconducting magnets
Michael Fanous	Columbia University	Cesar Luongo	Creating specified magnetic fields with superconducting magnets
Nathaniel Hammond	Florida State University	Peter Kalu	Material microstructure & characterization
Stephanie Howse	FAMU	Roy Odom	Techniques for creating specified magnetic fields with superconducting magnets
Kristen Johannessen	New College of USF	Martin Kendal-Reed	Responses to n-amylacetate in normosmics
Misha Lipatov	Harvard	Justin Schwartz	Investigation of sintering & metal-coating techniques of magnesium diboride
Kenneth Purcell	Western Kentucky	Jack Crow/Scott McCall	A study of Na doped $SrRuO_3$
Shelly Ann Ramrattan	Columbia University	Roy Odom	Techniques for creating specified magnetic fields with superconducting magnets
Haley Showman	William & Mary	Jack Crow/Gang Cao	Isotope effect on magnetic properties of $SrRuO_3$
Corinne Teeter	University of Washington	Steven Van Sciver	Heat flow in silver submerged in liquid helium
Teacher	**Grade Level**	**Mentor**	**Research**
Logan Chalfant	High School	Jack Crow/Gang Cao	Construction & testing of superconductor Y-123
Patricia Cramer	Middle School	Martin Kendall-Reed	Olfaction makes sense
Kristina Dugger	Pre-service	Patricia Dixon	Educational teacher outreach
Alison Gerry	Pre-service	Arneil Reyes/Phil Kuhns	Macroscopic model of nuclear spin
Susan Goracke	Elementary	Bob Goddard	Crystalline & chemical structure of cans
Matt Guyton	Middle School	Justin Schwartz	Superconducting tape properties
Thomas Hawkins	Elementary School	James Brooks	Granular physics
Robert Hoffman	High School	James Brooks	Granular physics
Toyka Holden	High School	Bob Goddard	Crystalline & chemical structure of cans
Dawn Housser	Pre-service	Roy Odom	Spanish moss project
Richard McHenry	High School	Stan Tozer/Eric Palm	Developing cantilevers from chemical etching process
Dan Nelson	Elementary School	James Brooks	Granular physics
David Rodriguez	Middle School	Roy Odom	Spanish moss project
Lynne Sapp	Middle School	Roy Odom	Spanish moss project
Alan Turner	Elementary School	Justin Schwartz	Superconducting tape properties
Bailey White	Pre-service	Martin Kendal-Reed	Olfaction makes sense
Linda Wolters	Elementary School	Arneil Reyes/Phil Kuhns	Macroscopic model of nuclear spin

Novel Use of Three-Dimensional Homonuclear NMR Spectra for Sequential and Stereospecific Assignment of an RNA Stem Loop

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Nancy L. Greenbaum, Florida State University, Chemistry, NHMFL

A revolution in molecular biology occurred in the mid-1980's with the radical discovery that RNA (ribonucleic acid) molecules catalyze certain biochemical reactions previously thought to be the province of protein enzymes only.[1,2] It is now recognized that RNA-based enzymes catalyze numerous cellular processes, including protein synthesis by the ribosome and RNA processing by the spliceosome. Other RNA-mediated functions include the use of small structural elements within coding and noncoding regions of messenger RNA to regulate various steps in gene expression. The notion of an "RNA World," a time when all functions traditionally attributed to proteins were carried out by RNA, has catapulted the field of RNA structural biology into a position of increasing prominence.[3]

A question that fascinates our laboratory is how an RNA polymer with a limited repertoire of four bases (possessing far less structural variability than observed among the 20 amino acid side chains) is able to fold into the myriad subtle conformations necessary to carry out its diverse biological functions. Formation of loop and bulge structures by folded RNA exposes the backbone and bases to other biomolecules, thereby presenting opportunities for specific recognition by proteins and other RNA molecules. RNA also employs several mechanisms to "fine-tune" its structure and stability, including chemical modification of bases to include functional groups having different chemical or steric properties (e.g. reference 4) and the specific binding of metal ions (e.g. reference 5).

Studying the structures formed by biologically active RNA molecules provides a powerful approach to understanding the many functions of RNA in normal cellular activities and pathological states, as well as defining potential therapeutic targets in the management of genetically based diseases. Our knowledge of RNA structure lags far behind that of protein structure, but the field is growing rapidly as a result of new biochemical and biophysical methodologies. Recent crystal structures of large supramolecular assemblies such as the ribosome provide new insight into how RNA participates in catalysis.[6] Solution NMR has become an increasingly important tool in the determination of the structure of smaller proteins and nucleic acids. Determining the structure of a molecule in solution is advantageous because it permits evaluation of molecular stability and dynamics under somewhat physiological conditions, and substantial information about structural features can be extracted in the absence of solving the entire structure. Analogous to the case in proteins, the biologically active regions of an RNA molecule are localized in specific structural domains, often of a size amenable to NMR analysis.

The proton NMR spectra of RNA, however, are notoriously difficult to assign. Substantial overlap of the resonances (at least 70 % of RNA ^{1}H-NMR signals resonate within a ~0.8 ppm range) complicates signal assignments, particularly in the ribose region, and may preclude the identification of important long-range interactions. Such difficulties are

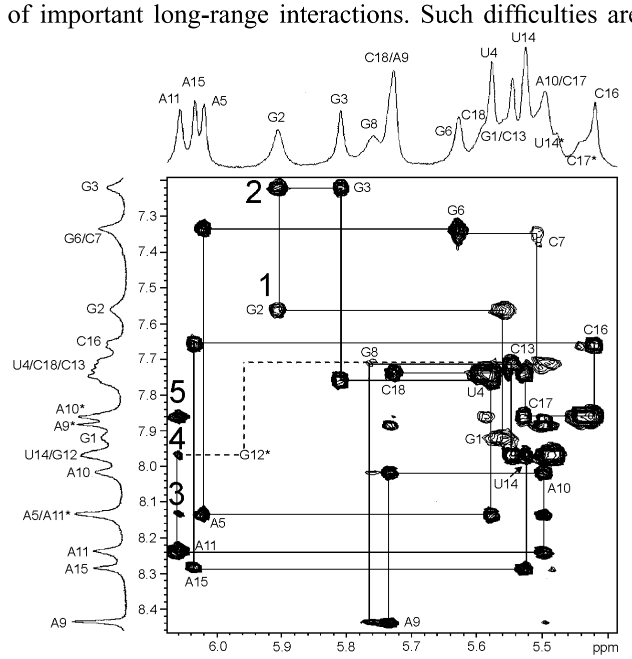

Figure 1. Anomeric-aromatic ("base-H1'") region of a two-dimensional 1H-NOESY spectrum of the GAAA tetraloop acquired at 25 °C with a mixing time of 200 ms on a 500 MHz spectrometer. The sequential H6/8-H1' walk is shown by the black lines. The G12 H1' is actually shifted far upfield (3.72 ppm) and is shown here with an asterisk and dotted line to preserve connectivities. NOE cross-peaks marked with bold numerals are identified in the text. Peaks in the 1D traces marked with an asterisk (*) belong to adenine H2 or pyrimidine H5 protons.

usually overcome by powerful new heteronuclear pulse sequences using isotopically labeled RNA samples (e.g. references 7-9). In some cases, however, it is impractical or impossible to generate an isotopically labeled sample, for instance when a chemically modified residue is included in the sequence or when an oligomer is not amenable to transcription. In addition, isotopically labeled RNA samples are exceedingly expensive and difficult to prepare. Improved homonuclear strategies would therefore be of great value under these circumstances.

Herein, we present a new strategy for RNA assignments using a combination of 3D homonuclear NMR techniques including NOESY-NOESY[10,11] and TOCSY-NOESY.[12] The target of our studies was an 18-nucleotide stem-loop sequence 5'-GGGUAGC<u>GAAA</u>GCUACCC-3' (underscored residues form the loop, and flanking regions form the Watson-Crick base paired stem), an RNA tetraloop of the "GNRA" family (where N = any base and R = any purine), which owes its unusual stability to base stacking and an extensive hydrogen bonding network.[13] Both f1f2 and f1f3 planes were used to complete the ^{1}H NMR assignments for this RNA stem-loop fragment, facilitating the determination of important structural features like the sugar pucker and the torsion angles γ and χ for each nucleotide. In addition, most of the H5'/5" protons were stereospecifically assigned using NOEs that are generally not accessible with 2D homonuclear techniques, such as the H3'–, H4'–, and H6/8→H5'/5" interactions. A manuscript addressing the use of this strategy in assigning loop regions will be submitted for publication shortly.

Two dimensional homonuclear NOESY experiments have traditionally been used to obtain the sequential assignments between the base and sugar protons in RNA. For example, the H1'– H6/8 NOE connectivity "walk" relies on both the intra-nucleotide $H1'_i \rightarrow H6/8_i$ and the sequential $H1'_i \rightarrow H6/8_{I+1}$ NOEs for successful assignments. As an illustration, the tetraloop's G2 H1' proton shows a clear interaction with its own H8 at 7.56 ppm and to the sequential G3 H8 at 7.22 ppm (Fig. 1, cross peaks 1 and 2, respectively). However, this sequential walk can not generally be traced throughout the entire molecule without some ambiguity. The H6/8-H1' cross-peaks are often overlapped in the 2D NOESY spectrum, preventing a definitive sequential assignment. For example, the A11→G12 interaction is ambiguous in the 2D NOESY spectrum; three potential cross peaks could correspond to the sequential H1'→H6/8

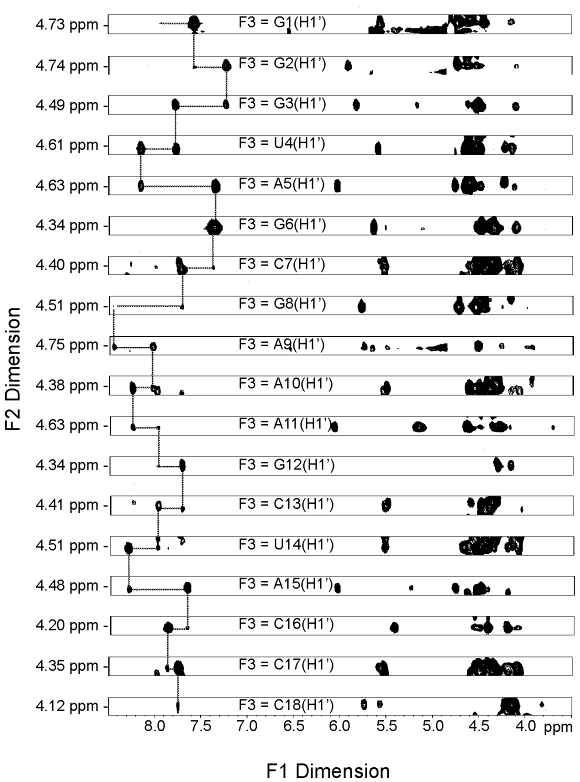

Figure 2. Selected f1f2 planes of a three-dimensional NOESY-NOESY experiment shown for the GAAA tetraloop (at the f3 values indicated). The dotted lines display an NOE connectivity "walk" through the molecule.

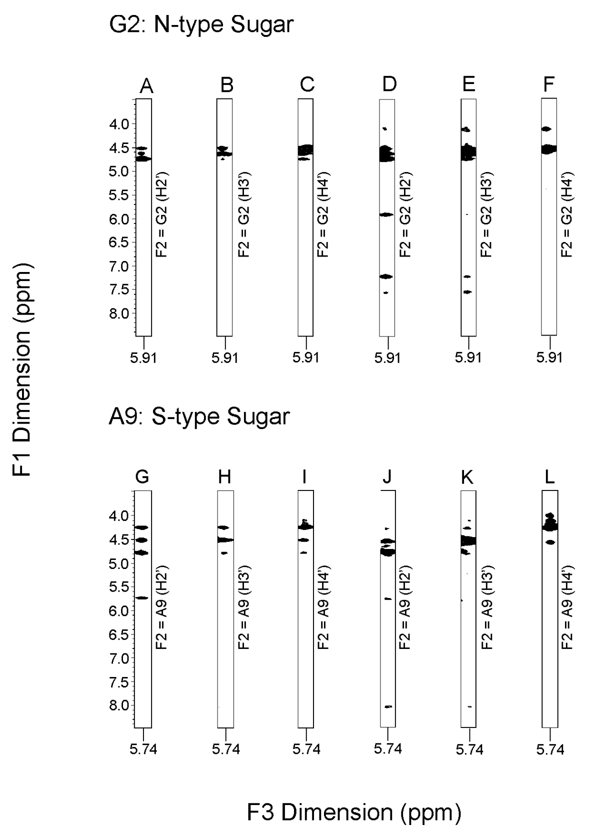

Figure 3. f1f3 planes of 3D TOCSY-NOESY (A-C and G-I) and NOESY-NOESY (D-F and J-L) spectra shown for selected values of f2. A-F correspond to an N-type sugar conformation typical of RNA A-form helices (in this case, G2), and G-L display the pattern typical of an S-type sugar in the loop region (A9). Details are described in the text.

interaction at 7.87, 7.97, and 8.12 ppm (Fig. 1, cross peaks 3-5). Fortunately, this problem can be overcome by analyzing the f1f2 planes of a 3D NOESY-NOESY experiment. Fig. 2 shows a series of f1f2 planes corresponding to a new H6/8-H2'-H1' NOE "walk", where the f3 dimension corresponds to the H1' protons as indicated in the figure. By analyzing three different types of nucleotide protons (H6/8, H2', and H1' in 3D NOESY-NOESY) instead of two (H6/8 and H1' in 2D NOESY) the ambiguous A11→G12 interaction could be resolved. The H6/8-H2'-H1' NOE walk showed only 1 sequential cross peak at 7.97 ppm (Figure 2, F3 = A11) for the same A11→G12 sequential interaction. Other areas of ambiguity that were solved using the 3D NOESY-NOESY include overlap for the A9-A11, C13-U14, and C17-C18. In fact, the entire molecule could be traced from the 5' to the 3' end of the tetraloop fragment without interruption, and thus help to assign unambiguously all of the H1', H2', and H6/8 protons (Fig. 2, dotted line).

In addition, stronger NOEs are generally noted for the sequential aromatic protons throughout the trace (Fig. 2, F3 = G1-C7, A9-A10, and G12-C18) as expected for regions of regular secondary structure where the sequential H2'$_i$ -H6/8$_{I+1}$ distance is short for A-type RNA helices. In fact, the absence of strong sequential aromatic NOEs in these f1f2 planes could provide a useful clue for determining regions of irregular secondary structure like loops and bulges (Fig. 2, F3 = G8 and A11). For example, both the G8 and A11 f1f2 planes lack strong sequential NOEs. This corroborates the large turns that occur within the loop between the G8/A9 and A11/G12 nucleotides. Even large deviations from helical structure can still be followed without interruption using this technique with moderate mixing times (150 ms).

The H2' proton assignments made from the new H6/8-H2'-H1' NOE walk were then used to find the H3' and H4' protons with a combination of TOCSY-NOESY and NOESY-NOESY experiments. For example, setting the F2 dimension equal to the G2 H2' proton (4.73 ppm) in a 3D TOCSY-NOESY experiment leads to a total of three peaks in the f1f3 plane corresponding to the G2 H2', H3', and H4' protons at 4.73, 4.63, and 4.52 ppm, respectively (Fig. 3A). In this context, the G2 H1' proton (F3 = 5.91 ppm) shows an NOE to the G2 H2' proton which subsequently shows TOCSY cross-peaks to the G2 H3' and H4' protons. No TOCSY cross-peak is observed for the H2'→H1' interaction. This is consistent with an N-type sugar conformation commonly found in A-type RNA helices that displays small $J_{1'-2'}$ coupling constants. Unfortunately,

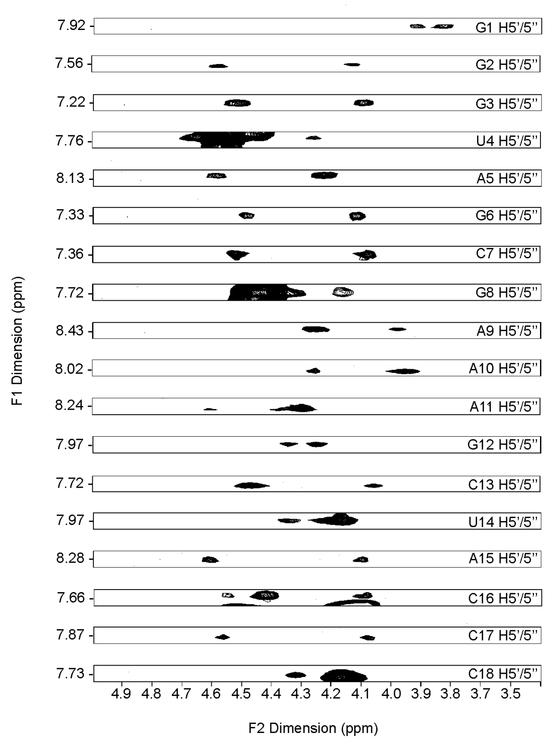

Figure 4. f1f2 planes of a 3D NOESY-NOESY used for identification of intra-nucleotide aromatic-H5' and -H5'' cross-peaks.

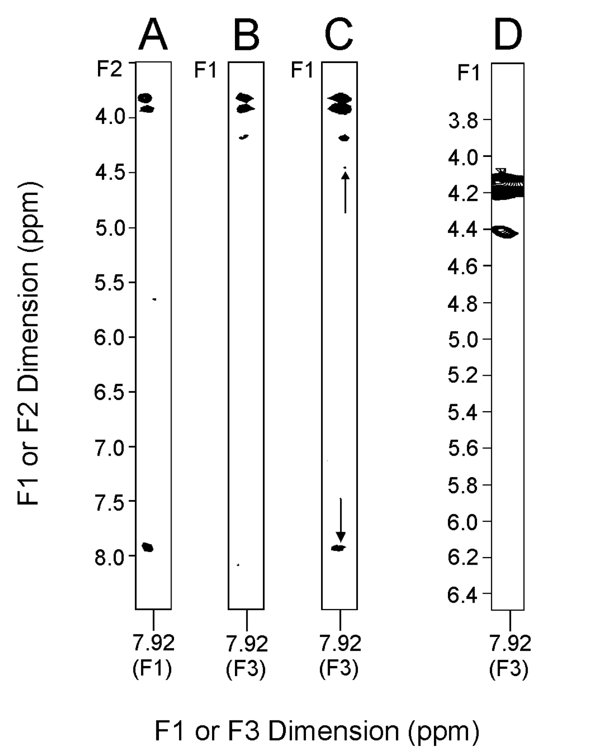

Figure 5. Stereospecific assignment of H5' and H5'' using f1f2 and f1f3 planes of NOESY-NOESY (A-C) and TOCSY-NOESY (D) spectra. See text for details.

the H3’ can not easily be differentiated from the H4’ or even the H2’ protons using this experiment (compare Fig. 3 A-C). Although an analysis of the intensity of the spin-lock lines for sequential and non-sequential bases has been used successfully to differentiate these sugar protons,[12] overlap in this region is common and the intensity of the spin-lock lines can often be misleading.

An easier approach is to compare the same f1f3 planes from a 3D NOESY-NOESY experiment. In this case, the H2’ shows strong NOE interactions to the base (~7-8 ppm) and anomeric (~5-6 ppm) protons (Fig. 3D), the H3’ shows interactions to only the base protons (Fig. 3E), and the H4’ protons generally do not show strong interactions to either base or anomeric protons (Fig. 3F). Furthermore, the H2’ generally shows a stronger interaction to the sequential base while the H3’ proton shows a stronger interaction to its own base for N-type sugars (compare aromatic region of 3D to 3E). Finally, we note that the H4’ proton generally shows strong intra-residue NOEs to its own H5’/5”, while the H2’ proton generally shows weaker NOEs to the sequential H5’/5” in the 3’ direction.

An S-type sugar, A9, is also shown for comparison (Fig. 3 G-L). Unlike G2, the A9 H2’ shows a strong TOCSY interaction to the A9 H1’ in addition to the H3’ and H4’ protons in the 3D TOCSY-NOESY experiment (compare Fig. 3G with Fig. 3A), consistent with the larger $J_{1'\text{-}2'}$ coupling constants for an S-type sugar. The A9 H3’ and H4’ protons do not show an interaction to the H1’ (Fig. 3H and 3I, respectively) and can thus help differentiate the H2’ protons from the H3’ and H4’ protons. The NOESY-NOESY can also help to distinguish the ribose protons of an S-type sugar. Again, the H2’ proton shows a strong interaction to both its own H1’ and its H6/8 (Fig. 3J). However, the strong sequential H2’-H6/8 interaction is missing, since A9 is found in the loop and not in the A-type helical stem. The A9 H3’ shows only a moderately strong interaction to its own base and the H4’ does not show a strong interaction to either its own base or its own anomeric proton as expected (Fig. 3K and L).

The f1f2 planes of the 3D NOESY-NOESY experiment also proved to be quite helpful in identifying all of the H5’/5” protons that are normally buried in the overcrowded ribose region (Fig. 4). This method relies on the strong geminal NOEs and the interaction of these protons with their own base through an H5’↔H5”→H6/8 pathway. Interactions with the H3’ and H4’ protons can also be seen *via* H3’/H4’→H5’/H5”→H6/8, but are generally weaker since the first NOE transfer is usually smaller and can be avoided with shorter mixing times or higher contour levels. This should prove to be an excellent method, for example, to monitor the interactions of metal ions with the phosphate backbone. For example, we observe that paramagnetic metal ions bound directly to the phosphate backbone alter the chemical shifts and relaxation properties of the adjacent H5’/5” protons (data not shown here).

Finally, the torsion angle γ and the stereospecific assignments of the H5’/5” could also be achieved in most cases using the f1f2 and f1f3 planes of both the TOCSY-NOESY and NOESY-NOESY spectra. Fig. 5A shows an f1f2 plane with the f3 dimension set equal to the G1 H5’ proton (same spectrum for either H5’ or H5”). Both geminal protons can be clearly seen without other complicating interactions. On the other hand, the f1f3 planes of the NOESY-NOESY going through F2, equal to 3.91, or 3.81 ppm (Fig. 5B and C, respectively), show interactions to the other G1 ribose protons. The signal at 4.16 ppm found in both Fig. 5B and 5C corresponds to the G1 H4’ proton. This is consistent with a torsion angle γ equal to g^+ where the H4’→H5’ and H4’→H5” distances are equal. This result is further corroborated by the f1f3 plane of a TOCSY-NOESY experiment with the f2 dimension set equal to the G1 H4’ proton at 4.16 ppm (Fig. 5 d). A g^+ torsion angle places the H5’/5” in a staggered configuration about the H4’ proton leading to only small H4’→H5’/5” coupling constants (2 to 3 Hz). Since no strong H4’→H5’/5” interactions are seen in the TOCSY-NOESY, a g^+ torsion angle can again be concluded. Furthermore, the signal at 3.81 ppm also shows interactions to the G1 H3’ and G1 H8 protons (marked by arrows in Fig. 3C). This allows the 3.81 ppm signal to be stereospecifically assigned to the H5”, leaving the signal at 3.91 ppm to be assigned to the H5’. There is a slightly stronger H4’→H5” interaction than H4’→H5’, suggesting a mixture of g^+ and t torsion angles.

Acknowledgment: The authors thank David Gorenstein for providing the pulse sequence for the 3D NOESY-NOESY experiment.

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AMRIS Progress, July 2001.

750 MHz Magnet for NMR/MRI: As reported last time, the 750 MHz instrument had been corrected with the addition of a second shim power supply train so that the shims didn't have to be disconnected when the gradients were removed. Although this certainly alleviated the problem, we are still struggling to get good high resolution spectra repeatably. Reloaded shim files seem unstable and un-reproducible. The situation is being investigated by the return of a Bruker engineer in July. The instrument, however, continues to image extremely well.

11.7 T, 40 cm Magnet for MRI: The situation has changed little since the last report. Gas flow has been used to clear water out of the magnet and the Magnex engineer returns mid-July to install a new top plate and begin the process of cooling and re-energization. Fingers crossed. In the meantime it was also discovered that the r.f. shielded room had degenerated after addition of the roof extension and the access plates. At no cost to us, Lindgren Inc. came back and stripped the system down (a long job including taking up concrete to access floor seals) and have successfully returned the room to specifications. Thanks Lindgren, that was very professional.

Other Magnets: Additionally, Bruker engineers returned over the last month and fixed nearly all of the remaining issues on our 500 and 600 narrow bore installations. Both instruments are now performing up to scratch. As a consequence, we have decommissioned our old 300 MHz system. This old magnet is being maintained and run by medicinal chemistry, presumably until it dies.

Staff: We have hired Dr. Cherian Zachariah, an NMR spectroscopist, to assist outside users in spectroscopic data acquisition and analysis in the AMRIS facility, complementing and assisting Jim Rocca. Welcome Cherian. Additionally, Kelly Jenkins has accepted a position as an r.f. technician and will spend half her time supporting the NHMFL outside users program. Welcome Kelly. With the hiring of Cherian and Kelly, we now have for the first time a full staff complement for instrument operation and maintenance in the facility.

Funding: We are very pleased to announce that our P41 NIH Resource grant application was funded and began on June 1st at the full amount requested of over $5 million total over 5 years. This grant represents a strong collaboration between many of us at UF, the NHMFL (Bill Brey, Tim Logan and Tim Moerland) and colleagues at Hershey (Mike Smith), University of Queensland (Stuart Crozier), University of Nottingham (Richard Bowtell), University of Alabama (John Forder), Emory (Ioannis Constantinidis), and Duke (Helene Benveniste, now at Brookhaven National Labs). The grant consists primarily of three cores: Core 1 concerns the development of small animal r.f. coils for the 4.7 and 11.7 T instruments, focusing on cavity volume coils and high frequency phased array coils; Core 2 concerns the development of microcoils and probes for microimaging and the development of high field imaging sequences; Core 3 concerns the development of multi coil arrays for spectroscopy and a superconducting probe for the 750 MHz instrument. Again, we thank all involved for making this possible and look forward to a strong research program centered around the AMRIS facility.

As of June 1st, Art Edison is now directing the AMRIS facility for a two-year term and will be your primary contact with respect to AMRIS issues. The following is a short summary of recent diffusion imaging studies by Dr. Mareci and colleagues.

AMRIS Research Report #7
Fiber Tract Mapping with Magnetic Resonance Diffusion Tensor Imaging

Evren Ozarslan, UF, Physics, UF McKnight Brain Institute, and NHMFL
Baba Vemuri, UF, Computer Science and Engineering
Xeve Silver, UF McKnight Brain Institute
Michelle DeFord, UF McKnight Brain Institute, UF, Neuroscience
Tom Mareci, UF McKnight Brain Institute, NHMFL, UF, Biochemistry and Molecular Biology

Fundamental advances in understanding biology require detailed knowledge of structural and functional organization in the living system. This is particularly important in the nervous system where anatomical connections determine the information pathways and how this information is processed. Our current understanding of the nervous system is incomplete because of a lack of fundamental structural information necessary to understand function.

Recently measurements of water translational self-diffusion with magnetic resonance imaging methods have been used to study the structural connectivity within whole living organisms. Water movement through tissue is restricted by the microscopic structure of the cellular environment, where the cell membrane is the most effective boundary to motion. In nervous tissue, axonal membranes mainly restrict the diffusion-driven motion of water resulting in anisotropic diffusion. In highly organized nervous tissue, like white matter in brain, diffusion anisotropy can be used to visualize fiber tracts. Recently, MR methods[1] have been developed to measure the tensor of water diffusional motion. We have applied these methods to the study of the spinal cord[2] and have extended this work to image the spatial distribution of two rates of diffusion[3] reflecting intracellular and extracellular processes. These methods provide a complete characterization of the restricted motion of water through the tissue that can be used to infer tissue structure and hence fiber tracts. By tracking the direction of fastest diffusion, non-invasive fiber tracking of the brain and spinal cord can be accomplished. Fibers tracks may be constructed by repeatedly stepping in the direction of fastest diffusion. The direction along which the diffusion is dominant corresponds to the direction of tensor eigenvector corresponding to the largest eigenvalue.

An example of white matter fiber track mapping in rat brain is shown in the figure. The results were obtained using the 17.6 T, 89 cm bore magnet Bruker Avance system at the UF McKnight Brain Institute. The specimen is an excised fixed normal rat brain. Because of the effect of the fixative (4 % formaldehyde solution) on the T_2 relaxation of water,[4] the specimen was first washed then imaged in a phosphate buffered saline solution. The diffusion tensor was calculated from a data set of 28 images; four diffusion-weighting gradient strengths in seven directions (x, y, z, xy, xz, yz, xyz). Part A is a three-dimensional MR image visualization of the rat brain. Part B shows the white matter fiber tracts derived from a measure of the diffusion tensor of water motion through the brain. More detailed views of the fibers relative to the MR image of the brain are shown in Parts C (viewed from the side) and D (viewed from below). The fiber-tracking algorithm starts from a voxel center and proceeds in the direction of the major axis of the diffusion ellipsoid. Tracking continues along the direction defined by the weighted-average of the diffusion directions around the fiber direction. Tracking stops when a measure of diffusion crosses a threshold, i.e., a loss of anisotropy in diffusion.

Editors note: The images are available in color on the laboratory's web site at *www.magnet.fsu.edu.*

This scheme for fiber tracking, however, is resolution dependent since the MR data only reflects average fiber orientation within a voxel. Small fibers adjacent to each other may not be distinguished. Moreover, this fiber-tract-mapping scheme does not handle branching fiber structures. More recently, there have been some attempts at changing the standard diffusion tensor model by using a high angular resolution diffusion weighted acquisition to investigate voxels containing multi-directional fibers.[5] This type of spherical sampling addresses the issue of how fiber populations add and may provide a method for discriminating crossing fiber structures. This is a promising approach for addressing the effects of resolution in these MR images and we are currently investigating this in our work.

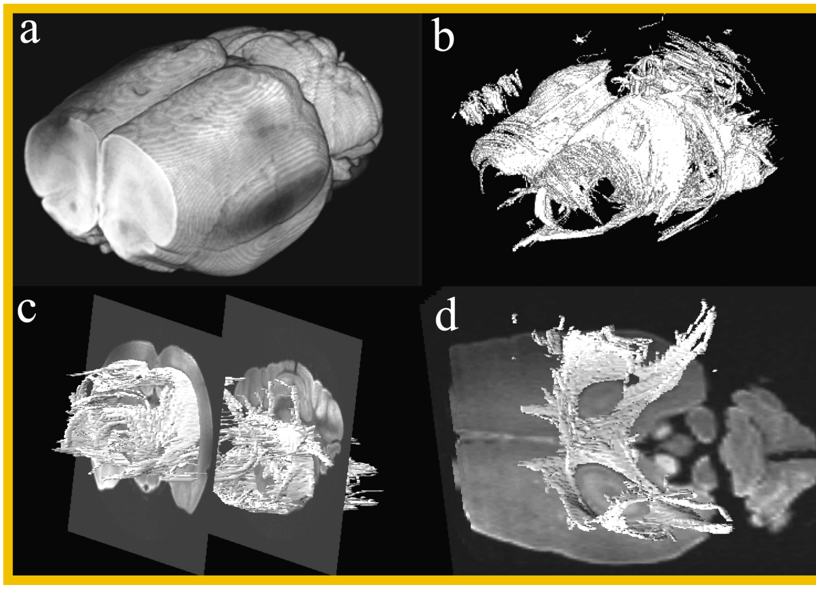


Figure A. Three-dimensional MR image visualization of the rat brain.
Figure B. White matter fiber tracts derived from a measure of the diffusion tensor of water motion through the brain.
Figure C. Detailed view of the fibers relative to the MR image of the brain (viewed from the side).
Figure D. Detailed view of the fibers relative to the MR image of the brain (viewed from below).

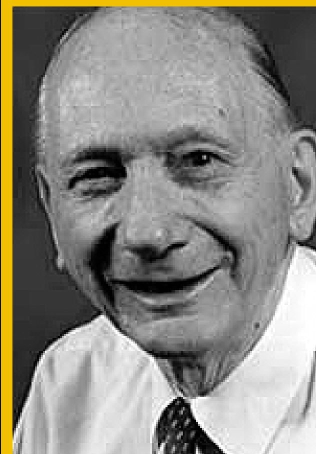
1 Basser, P.J., Pajevic, S., Pierpaoli, C., Duda, J. and Aldroubi, A. "In vivo fiber tractography using DT-MRI data," *Magn. Reson. Med.*, **44**, 625-632, (2000).

2 Inglis, B.A., Yang, L., Wirth, III, E.D., Plant, D., and Mareci, T. H., "Diffusion Anisotropy in Excised Normal Rat Spinal Cord Measured by NMR Microscopy," *Magn. Reson. Imag.*, **15**, 441-450, (1997).

3 Inglis, B.A., Bossart, E.L., Buckley, D. L., Wirth III, E.D. and Mareci, T.H., "Visualization of Neural Tissue Water Compartments using Biexponential Diffusion Tenor MRI," *Magn. Reson. Med.*, **45**, 580-587, (2001).

4 Bossart, E.L., Inglis, B.A., Silver, X.S., and Mareci, T.H., "The Effect of Fixative Solutions in Magnetic Resonance Imaging," In preparation for submission to *Magn. Reson. Med.* (2001).

5 Frank, L.R., "Anisotropy in high angular resolution diffusion-weighted MRI," *Magn. Reson. Med.*, **45**, 935-939 (2001).



N MEMORIAM

E. Raymond Andrew

June 27, 1921 - May 26, 2001

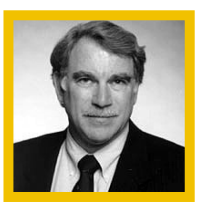
World-renowned physicist, E. Raymond Andrew, passed away on Sunday, May 26 at his home in Gainesville. Andrew, who was Graduate Research Professor Emeritus at UF, was well known for his work on magnetic resonance and his pioneering contributions to medicine through NMR and MRI. Many of his experiments are classics in today's textbooks. While at the University of Wales in the late 1950s and '60s, he made one of his most significant discoveries, the narrowing of NMR lines by magic angle spinning, which has been the foundation of modern high resolution NMR studies for chemical structures. In 1983, Andrew joined the NMR physics group at UF and later played a major role in establishing the vision for the NHMFL in 1990. Andrew is survived by his wife, Eunice; twin daughters, Patricia Andrew and Charmian Hopkins; and grandchildren, Heather and Holly Hopkins. Neil Sullivan, Dean of Arts and Sciences at UF and co-principal investigator at the NHMFL, says "although Andrew was truly one of the greats in his field of science, he always had time to talk to and encourage young scholars and students. He was a statesman and a gentleman in all his interactions with fellow scientists, staff, and students. He will be dearly missed by all who had the fortune to know him."

Pat Dixon, former Assistant Director of the Center for Research and Learning (CIRL) has been promoted to Director, filling the vacancy left when Sam Spiegel joined Training Solutions, Inc. Dixon joined the NHMFL in 1996 to work on a grant to create curriculum materials for the State Department of Education. Soon, thereafter, she assumed expanded duties as Assistant Director with primary responsibility for the laboratory's teacher education and curriculum development activities. Dixon's educational research interests include teacher practice and how professional development opportunities affect classroom practice. Dixon has taught classes at the FSU College of Education and is presently teaching at Flagler College. CIRL has expanded its outreach to area classrooms, statewide teacher workshop opportunities, and continues to provide, teachers, students, and the general public with meaningful experiences related to a science research facility.

Stephen Hill, a former postdoc at the NHMFL-Tallahassee, is a new faculty member at UF. He will be bringing with him, a postdoc, graduate student, and two NSF-funded projects focusing on the electrodynamic properties of highly anisotropic superconductors in high magnetic fields and a Nanoscale Interdisciplinary Research Team (NIRT) project dealing with quantum effects in single molecule magnets. Hill received his B.A. and Ph.D. degrees in Physics from the University of Oxford, where his Ph.D. adviser was John Singleton, a prominent user of the NHMFL. In Tallahassee, Hill worked as a postdoc for two years (1995 to 1997) with Jim Brooks before taking a faculty position at Montana State University four years ago. Hill, a major user of the Tallahassee facility during the past four years, has served as a member of the NHMFL Users' Advisory Committee and the NHMFL Condensed Matter Research Program Committee. The majority of Hill's research involves the use of millimeter and sub-millimeter wave (GHz/THz) spectroscopic techniques, in combination with strong magnetic fields, to probe electronic/magnetic structure and dynamics in various molecular conductors, superconductors, and magnets. Recent publications have focused on vortices in highly anisotropic organic superconductors and Electron Paramagnetic Resonance studies of magnetization dynamics in molecular nanomagnets.

Hiroshi Maeda will be visiting the NHMFL and CAPS for an extended stay of six to nine months to continue his study on the development of conductors with low AC loss and the effects of magnetic fields on the textured growth of HTS materials. Maeda was the discoverer of the the Bi-Sr-Ca-Cu-O superconducting materials while he was a Group Leader of the Superconducting Materials Group at the National Research Institute for Metals. This material system is now the basis for the fledgling high temperature superconductor industry.

Christine Hughey, FSU Ph.D. candidate and graduate research assistant in the NHMFL ICR Program, has been selected to receive the 2001 Society for Applied Spectroscopy Student Award, to be presented on October 10, 2001 at the annual meeting of the Federation of Analytical and Chemical Spectroscopy Societies in Detroit. "This (national) award is given to a graduate student in recognition of outstanding research in the area of spectroscopy. Any full-time graduate student doing research in the field of spectroscopy is eligible for the award. The award consists of a plaque or scroll and an expense-paid trip to FACSS to accept the award." The award was based on Hughey's recent successful resolution and identification of the elemental compositions of up to several thousand compounds in petroleum heavy crude oils and petroleum distillates by electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry. Hughey is the second NHMFL/FSU graduate student to receive this award (Stone Shi was the 1998 winner).



Alan Marshall, director of the NHMFL ICR Program, has been awarded the 2002 Pittsburgh Spectroscopy Award, the first of its type for an FSU faculty member. Presented at the Pittsburgh Conference by the Spectroscopy Society of Pittsburgh, this prestigious award honors an individual who has established a career of accomplishments toward the advancement and understanding of spectroscopy. Marshall is being recognized for his co-invention with Melvin Comisarow of the Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) technique and other advancements stimulated in mass spectrometry. He is currently pioneering mass-based structural analysis of biomacromolecules and complex chemical mixtures. Marshall has mentored about 80 graduate students and postdoctoral fellows, published over 300 refereed papers on spectroscopy, and received numerous awards and honors. Prior winners of this award include three Nobelists: Herzberg, Siegbahn, and Zewail.

Justin Schwartz, leader of the NHMFL High Temperature Superconductor Magnets and Materials group and professor of mechanical engineering, has been appointed as senior research adviser to the FSU Vice President for Research for a one-year, quarter-time term. Schwartz will assist in strengthening communications with faculty on research-related policy issues, coordinating major interdisciplinary research initiatives, representing FSU with local and national scientific groups and organizations, and developing and implementing new research activities. Schwartz, who was involved in the development of the Master program in Magnets and Magnet Materials at the FAMU-FSU joint College of Engineering, says "this position will be an excellent opportunity for me to contribute to the continuously improving research environment at FSU and to become better acquainted with other departments. Moving FSU into the top tier of the nation's academic and research institutions is clearly an achievable goal. I relish the opportunity."

FSU Faculty Promotions

Congratulations to five NHMFL colleagues who were promoted recently:

Vladimir Dobrosavljevic
Condensed Matter Science, Associate Professor

Mark R. Emmett
ICR, Associate Scholar/Scientist

Lloyd W. Engel
Condensed Matter Science, Associate Scholar/Scientist

Christopher L. Hendrickson
ICR, Associate Scholar/Scientist

Vincent J. Salters
Geochemistry, Scholar/Scientist

Charles A. Swenson
Magnet Science & Technology, Research Associate

Conference & Workshop Activity

24th International EPR Symposium

July 29-August 2, 2001
Denver, Colorado
http://www.du.edu/~seaton/eprsym.html
http://www.milestoneshows.com/rmcac/

NMR Symposium at the 43rd Rocky Mountain Conference on Analytical Chemistry

July 30-August 2, 2001
Denver, Colorado
http://www.milestoneshows.com/rmcac/RMCAC_NMR.htm

Both the EPR and the NMR symposiums will be held in conjunction with the **43rd Annual Rocky Mountain Conference.** Approximately 1,000 people attend the Rocky Mountain Conference, which also includes an instrument exhibit. The EPR symposium covers all aspects of EPR spectroscopy. Sessions will emphasize the wide range of frequencies at which EPR is now performed including, for example, *in vivo* experiments at 250 MHz and high field EPR. The NHMFL is helping to sponsor the high field sessions. There also will be a special session on industrial applications of EPR. The NMR symposium will feature sessions on topics such as *in-situ* NMR, nanoparticles and interfaces, obtaining structure from multiple-spin systems, theory, polymers and dynamics, quadropolar nuclei, and inorganics.

The Future of Materials Physics: A Festschrift for Zachary Fisk

August 13-15, 2001
Oppenheimer Study Center
Los Alamos National Laboratory

Recent discoveries of complex materials and phenomena with extraordinarily interesting properties are challenging conventional wisdom and are forging an entirely new style of inquiry that links chemistry, materials science, and physics. This workshop, held as a celebration of Zachary Fisk's 60th birthday, brings together international leaders in materials physics to explore the frontiers in theoretical and experimental research of the complexity of solids. Dr. Fisk is an NHMFL faculty member, a Fellow of LANL and APS, and a member of the National Academy of Sciences. Seating for this workshop is limited. For further information, contact Rose B. Romero, workshop administrator, at *rbromero@lanl.gov*.

Fifth Latin American Workshop on Magnetism and Magnetic Materials and their Applications

September 3-7, 2001
San Carlos de Bariloche, Argentina
http://www.cab.cnea.gov.ar/law3m/

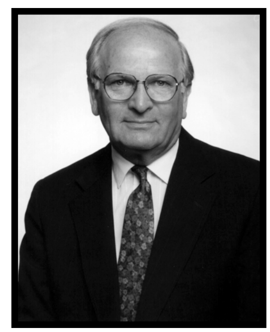
This will be the first meeting of this workshop of the new millennium and the fifth of a series initiated in La Habana, Cuba in 1991; other workshops were held in Guanajuato, Mexico (1993), Merida, Venezuela (1995), and Sao Paulo, Brazil (1998). LAW3M is designed to bring together recent developments in both fundamental and applied magnetism, on topics such as thin films, giant magnetoresistance and magnetoimpedance, nanocrystalline materials, superconducting oxides, and magneto-optics. The program will consist of invited and contributed papers, tutorial in nature, as well as reviews of recent work in specialized fields.

6th International Symposium on Magnetic Suspension Technology

October 7-11, 2001
Turin, Italy
http://www.lim.polito.it/ISMST6

The 6th ISMST will be hosted by the Technical University of Turin with support from the NHMFL. It will cover a wide range of magnetic suspension topics, including magnetic bearings, electromagnetic launch, sensors, controls, related high- and low-temperature superconducting magnet technology, wind tunnel model suspension, and design and implementation practices. Industrial applications include magnetic bearings for high-speed rotating machinery, levitated trains, vibration isolation, pointing and control, and guiding systems. An exciting new area is materials processing and biological studies in low or zero gravity by magnetic suspension.

Conference co-chairs are Giancarlo Genta of the Technical University of Turin and NHMFL Deputy Director Hans Schneider-Muntau. Complete information is available on the conference Web site, but interested parties may also direct inquiries by e-mail to *ismst6@magnet.fsu.edu* (NHMFL in Tallahassee) or *ismst6@polito.it* (Politecnico di Torin



J. Robert Schrieffer Symposium

October 19, 2001
Los Alamos National Laboratory
Santa Fe, New Mexico
Pre-Conference Registration Deadline: September 19, 2001
www.magnet.fsu.edu/

Nobel Laureate and NHMFL Chief Scientist Bob Schrieffer will be honored for his lifelong commitment to physics research and the scientific community. The commemorative symposium will be held in conjunction with PPHMF-IV, which he has guided since its inception at the NHMFL in Tallahassee in 1991. The special one-day meeting will feature other Nobel Prize winners and scientists who are distinguished in condensed matter physics in high magnetic fields. For more information, see the laboratory's Web site or contact Mary Layne at *symposium@magnet.fsu.edu*.

Physical Phenomena at High Magnetic Fields (PPHMF-IV)

October 19-25, 2001
Santa Fe, New Mexico
Hotel Headquarters: Hilton and Radisson of Santa Fe
Abstract Deadline: August 10, 2001
http://www.lanl.gov/mst/nhmfl/PPHMF4/

This major international conference brings together experts to discuss recent advances in areas of science and applications in which high magnetic fields play an important role. Topics will include: semiconductors, magnetic materials, superconductivity, organic solids, the quantum Hall effect, chemical and biological systems, and the technological use of high magnetic fields. Initiated by the NHMFL in 1991, this conference is held every three years.

2001 Southeast Magnetic Resonance Conference (SEMRC)

October 26-28, 2001
UF McKnight Brain Institute, Gainesville, Florida

This two-day meeting provides an ideal opportunity for scientists in all areas of magnetic resonance to come together and share new applications and technique developments. There will be a banquet on Saturday, October 27 in memory of Dr. Raymond Andrew. For full conference details, please see the sidebar. For more information, contact Art Edison at *art@ascaris.ufbi.ufl.edu*

It is time once again for the Southeast Magnetic Resonance Conference (SEMRC). The SEMRC provides an ideal opportunity for scientists in all areas of magnetic resonance to come together and share new applications and technique developments. The Southeast has had tremendous growth in magnetic resonance research over the recent years, and some of the major national and international centers are now located in this region of the country.

The 2001 SEMRC will be held from October 26 through October 28 in Gainesville, Florida at the McKnight Brain Institute of the University of Florida (MBI-UF). MBI-UF is the home of the Advanced Magnetic Resonance Imaging and Spectroscopy (AMRIS) facility, which has many state-of-the-art spectrometers for a variety of applications. MBI-UF has excellent technological and audiovisual capabilities to support the meeting.

The magnetic resonance community and the University of Florida lost a great colleague this year, Dr. Raymond Andrew. Among his many scientific accomplishments, Raymond was the inventor of magic angle spinning and was influential in the development of magnetic resonance imaging. There will be a banquet on Saturday, October 27 in memory of Raymond. We are pleased and honored that Raymond's good friend and colleague, Professor Charles Slichter, will be delivering a memorial lecture after the banquet.

We are also fortunate to have two great plenary speakers for the 2001 SEMRC, Lewis Kay and James Hyde. Other scientific talks will be picked from submitted abstracts. Abstract and registration information will be available shortly. Please contact me for further details and to have your name added to e-mail notification lists.

See you at the SEMRC,
Dr. Arthur S. Edison
Chair 2001 SEMRC

4th International Conference on the Scientific and Clinical Applications of Magnetic Carriers

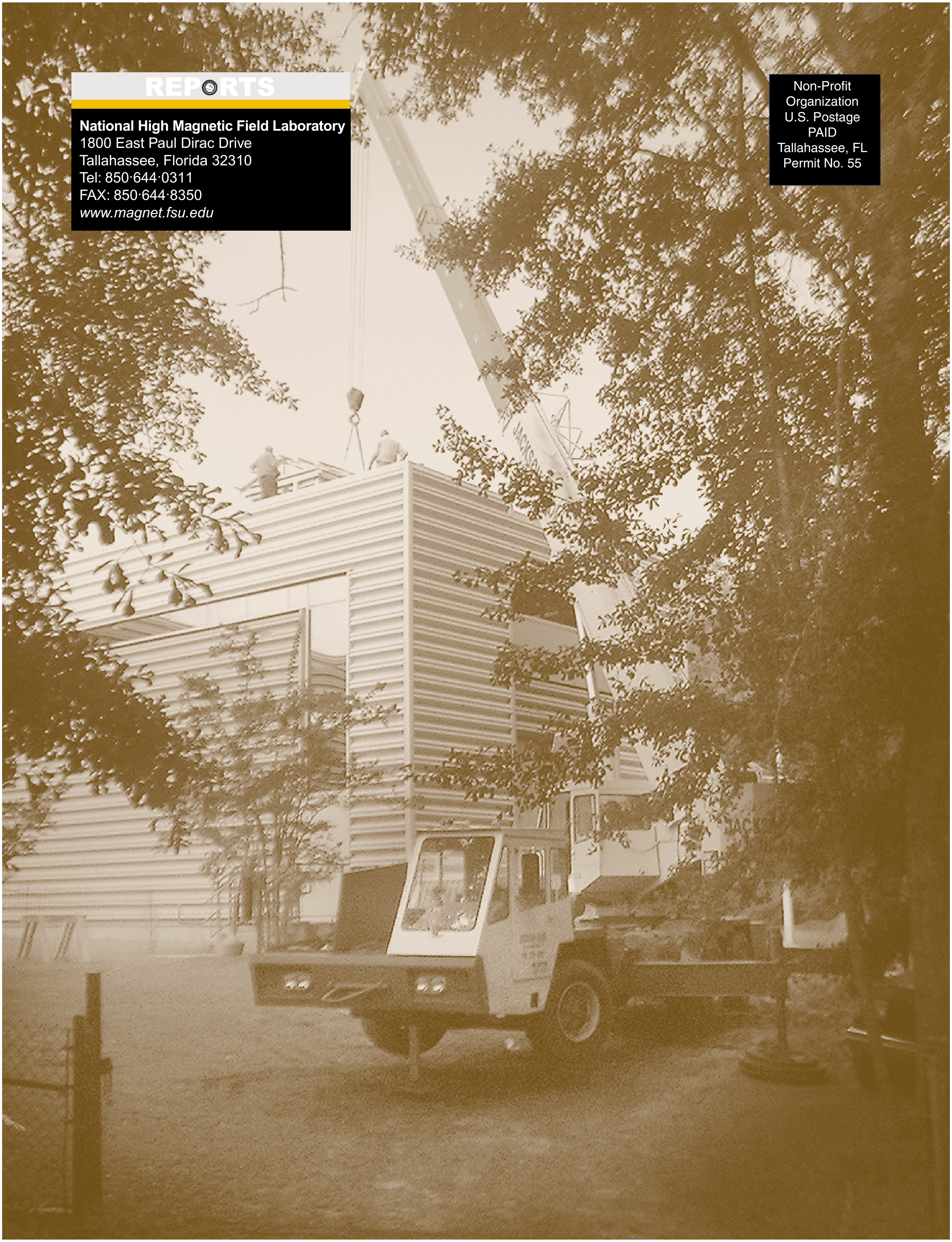
May 9-11, 2002
Tallahassee, Florida
Hotel Headquarters: Doubletree
Pre-Conference Registration
Deadline: March 8, 2002
http://www.magneticmicrosphere.com/

The laboratory is pleased to support this biennial conference being organized by the Cleveland Clinic Foundation in collaboration with IFAB, Rostock, Germany, and FSU. The meeting will be devoted to the development and application of magnetic microspheres in the basic and clinical sciences and will include a visit to the NHMFL. Preparation and modification of biodegradable magnetic particles, characterization of magnetic particles, application in cell separation and analysis, applications in molecular biology, and targeted drug delivery will be discussed. Clinical applications of magnetic nano- and microspheres for cancer treatment, hyperthermia, and magnetic resonance contrast enhancement will also be covered. For more information, contact Urs Hafeli at *hafeliu@ccf.org*. Abstract deadline is March 8, 2002.

Applied Superconductivity Conference (ASC04)

October 4-8, 2004
Jacksonville, Florida

This important international conference is held every two years and typically attracts approximately 1,800 participants. In September 2000, ASC00 was held in Virginia Beach, Virginia; in August 2002, ASC02 will be in Houston, Texas, and in October 2004, ASC04 comes to Jacksonville, Florida. For information, contact ASC04 Conference Chair Justin Schwartz in NHMFL's Magnet Science & Technology program, 850-644-0874, fax 850-644-0867; *schwartz@magnet.fsu.edu.*



REPORTS

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