

# REPORTS



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## Previewing the 2000 NHMFL Annual Research Review

Biology  
Chemistry  
Geochemistry  
Superconductivity - Basic  
Superconductivity - Applied  
Quantum Solids  
Kondo/Heavy Fermion Systems  
Molecular Conductors  
Semiconductors  
Magnetism and Magnetic Materials  
Other Condensed Matter  
Magnetic Resonance Techniques  
Instrumentation  
Engineering Materials  
Magnet Technology  
Cryogenics

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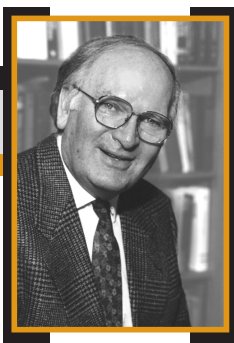
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Cover art by: Robert Burke



J. Robert Schrieffer

### Previewing the 2000 NHMFL Annual Research Review

*The laboratory's major annual report of research has recently been completed and will be available on the Internet (<http://www.magnet.fsu.edu>) and in compact disk format in the near future. We are proud to devote this issue of our newsletter to highlighting some of the laboratory's outstanding research and engineering activities that will be included in the 2000 Annual Research Review.*

*The Science Program of the NHMFL continued to grow in strength during 2000. Users and NHMFL faculty submitted 295 research reports in 16 categories, with interdisciplinary activities clearly on the rise. There also appears to be increased collaboration between in-house research staff and external users.*

*The breakout of reports by category is as follows: biology (47), chemistry (27), cryogenics (5), engineering materials (6), geochemistry (13), instrumentation (16), kondo/heavy fermions (19), magnetism and magnetic materials (36), magnetic technology (6), molecular conductors (19), magnetic resonance techniques (19), other condensed matter (10), quantum solids (3), superconductivity applied (16), superconductivity basic (26), and semiconductors (27).*

*Representative highlights from various categories follow. Many other projects of comparable excellence could have easily been chosen.*

#### Selected Reports

##### BIOLOGY

### Membrane Protein Structure Determination Using PISA Wheels

Wang, J., NHMFL/FSU, Molecular Biophysics  
Kim, S., NHMFL/FSU, Molecular Biophysics  
Kovacs, F., NHMFL/FSU, Molecular Biophysics  
Cross, T.A., NHMFL/FSU, Chemistry

**P**atterns of resonances have been recognized in solid state NMR spectra of uniformly aligned samples of alpha-helical membrane proteins. These patterns reflect helical wheels in which it is illustrated that 3.6 amino acid residues complete a full turn of the helix. In the PISA (polar index slant angle) wheel,<sup>1</sup> displayed in the NMR spectra, there are 3.6 resonances per repeat of the circular pattern. The shape, size, and position of these patterns within the spectra reflect the tilt or slant angle of the helix with respect to the bilayer normal that is aligned parallel to magnetic field. This information can be obtained without any resonance assignments. Recognition and characterization of protein structure directly from NMR spectra has not been achieved previously.

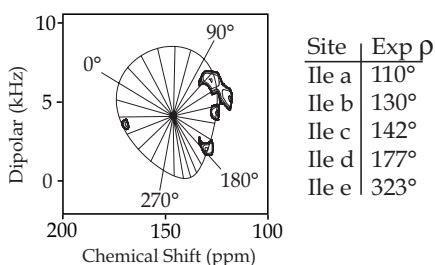
The rotational orientation of the helix can be assessed from a single resonance assignment in the helix. In other words, instead of assigning the 20 amide <sup>15</sup>N resonances along the entire length of a transmembrane helix, it is possible to make

this characterization with a single assignment. Resonance assignments often stand in the way of structure characterization by NMR because of the difficulty in making definitive assignments. However, a few assignments can be readily achieved through an amino acid specific labeling procedure. In other words, all of the isoleucine residues can be <sup>15</sup>N labeled at the same time.

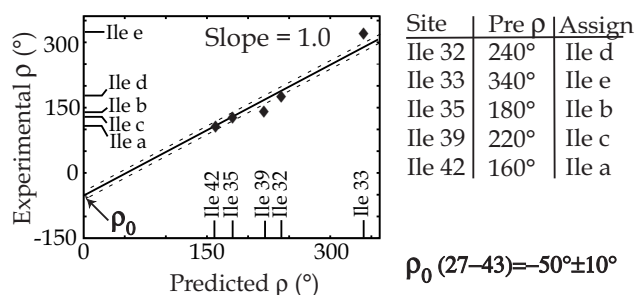
In the transmembrane helix of M2 protein, there are five isoleucines at positions 32, 33, 35, 39, and 42. These have been labeled as shown in Fig. 1, and their resonances are resolved. Experimental values of the rotational angle can be assigned to each resonance, and in Fig. 2, these experimental rho values are correlated with predicted rho values from a helical wheel. Residue 33 and resonance e are unique resulting in an unambiguous assignment. Indeed, the rest of the assignments can also be accurately made for the other four isoleucine residues. This assignment uniquely fixes the rotational orientation of the helix within the lipid bilayer. Indeed, with this information, it has been possible to determine at high precision the backbone structure of this transmembrane helix in a hydrated model membrane environment.

Today, there is great interest in high through-put structural characterization. These results suggest that solid state NMR may be able to perform such a task for the third of all proteins that are membrane bound.

<sup>1</sup> Wang, J., *et al.*, *J. Magn. Reson.*, **144**, 162-167 (2000).



**Figure 1.** Five-site isoleucine labeled transmembrane peptide from the M2 protein of Influenza A virus. The solid state NMR PISEMA spectrum correlates the anisotropic N-H dipolar interaction with chemical shift interaction. The pattern of resonances fit a PISA wheel description as shown, and the rotational orientation angle for each resonance can be quantified.



**Figure 2.** The experimental rho values are correlated with predicted values from a helical wheel. The line is restricted to a slope of 1.0, resulting in a unique correlation and a sequence specific assignment of the resonances.

## CHEMISTRY

# Reading Chemical Fine Print: Resolution and Identification of 3000 Nitrogen-Containing Aromatic Compounds from a Single Electrospray Ionization Fourier Transform Ion Cyclotron Resonance Mass Spectrum of Heavy Petroleum Crude Oil

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 Rodgers, R.P., NHMFL  
 Hendrickson, C.L., NHMFL  
 Emmett, M.R., NHMFL  
 Marshall, A.G., NHMFL/FSU, Chemistry

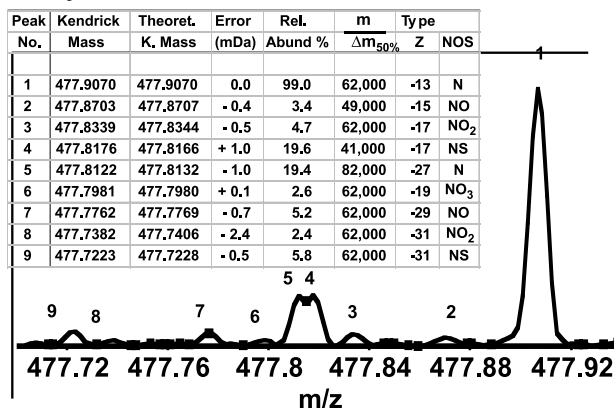
**E**xtra heavy petroleum crude oil (50% of the mixture boils at >566 °C) has been analyzed directly, without prior fractionation, by a high-field (9.4 T) Fourier transform ion cyclotron resonance mass spectrometer coupled to an external micro-electrospray ion source. At an average mass resolving power, ( $m/\Delta m_{50\%} \approx 50,000$ ), a single wideband (250-1250 Da) mass spectrum exhibited ~5000 resolved peaks with an average mass of 617 Da (e.g., up to 7-10 resolved peaks at each nominal mass). Their elemental

compositions were positively identified by accurate mass measurement with an average deviation of less than 1 mDa from each assigned elemental composition. The number of elemental compositions at each nominal mass, the number of sulfur/oxygen atoms in a molecule, and aromaticity each increase with increasing mass.

Based on elemental composition alone, we resolve more than 3000 distinct chemical formulas (excluding  $^{13}\text{C}$  isotopic species). Of the 3000 unique elemental compositions, we identify 12 major heteroatomic “classes”; (e.g., molecules containing N, NS, NS<sub>2</sub>, NO, NOS, etc.); for the various “classes”, we identify more than 100 hydrocarbon “types” (e.g., molecules with the same number of rings plus double bonds); and for each “type”, we determine the carbon number distribution (20-80 carbons) to reveal the number of alkyl carbons appended to aromatic rings. The present results represent the most complete chemical characterization ever achieved for such a complex mixture, based on a single experimental data set.

**Acknowledgements:** The authors thank Daniel McIntosh for machining all of the custom parts required for the 9.4 T instrument construction and John P. Quinn for many helpful discussions. This work was supported by the NSF National High-Field FT-ICR Mass Spectrometry Facility (CHE 99-09502), FSU, and the NHMFL. The authors thank ExxonMobil for supporting this exploratory research of heavy petroleum and the permission to publish the data.

## ESI FT-ICR MS of $\text{C}_c\text{H}_{2c+Z}\text{N}_n\text{O}_s\text{S}_s$ Heavy Crude Oil



**Figure 1.** Mass scale expanded segment of a full range heavy crude mass spectrum, allowing for resolution and elemental composition assignment (based on accurate mass measurement) of 9 chemically distinct species at a single nominal mass. The tabulated data for the 9 peaks show an average mass error of ~1.5 ppm for the proposed assignments. The chemical “type” (i.e., number of rings plus double bonds) is classified according to “Z” value in the chemical formula at the top of the diagram (see text), and the heteroatom content is also shown in the table.

<sup>1</sup> Qian, K., *et al.*, “Reading Chemical Fine Print: Resolution and Identification of 3000 Nitrogen-Containing Aromatic Compounds from a Single Electrospray Ionization Fourier Transform Ion Cyclotron Resonance Mass Spectrum of Heavy Petroleum Crude Oil,” *Energy & Fuels*, **15**, 0000-0000 (2001).

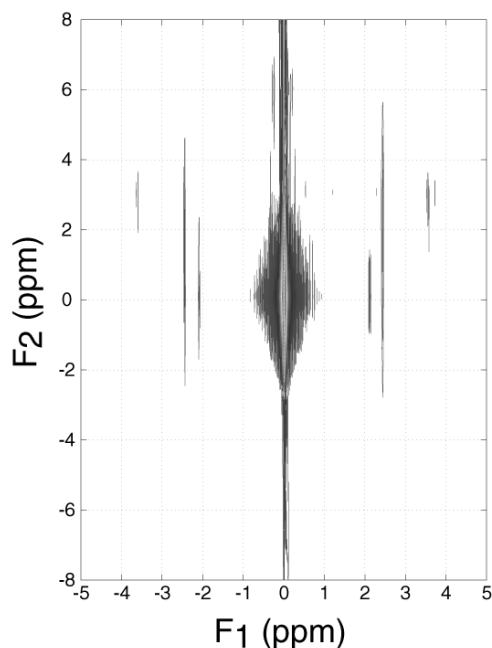
## Resolution Enhancement in Solution NMR on the Keck Magnet by Intermolecular Zero-Quantum Detection and Matrix Pencil Estimation

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 Brey, W., NHMFL  
 Murali, N., NHMFL

**S**trong magnetic fields are desirable in high-resolution NMR to enhance resolution, improve sensitivity, and simplify spectra. Resistive, or hybrid magnets can achieve substantially higher fields than those available in superconducting magnets, but their spatial homogeneity and temporal stability are unacceptable for high-resolution applications. We showed that modern stabilization and shimming technology produce a sufficiently good field that detection of intermolecular zero-quantum coherences (iZQCs),<sup>1-3</sup> using the CPMG-HOMOGENIZED sequence, can remove almost all of the rest of the inhomogeneity, while retaining the information of chemical shift differences and J couplings.<sup>4</sup> In the 25 T Keck electromagnet (1 kHz/s drift, 3 kHz linewidth over 1 cm<sup>3</sup>), preliminary results of iZQC detection of a 1:1 acetone:water solution remove >99% of the remaining inhomogeneity to achieve a resolution enhancement of ~100, thereby generating the first ever high-resolution, liquid-state NMR spectra acquired at > 1 GHz. Optimization of the pulse sequence and spectrometer promises even higher resolution, until reaching a theoretical boundary of ~4 Hz linewidth. This motivates, for example, refinement of the flux stabilizer and application of pulse shaping.

iZQC detection is a two-dimensional acquisition scheme, with the high-resolution information encoded in the indirect dimension. The final achievable resolution is restricted by Fourier transforming the heavily truncated signal in the indirect dimension due to limited capacity of cooling water and, therefore, magnet time available for uninterrupted acquisitions. This inherent limitation can be alleviated by invoking high-resolution spectral estimators as alternatives to the Fourier transform. In particular, the method proposed in Ref. 5 is especially promising for estimating and quantifying the iZQC signals. The number of exponential components in the signals is first determined by criteria derived from information theory and the spectral parameters are then estimated by the efficient matrix pencil method. Fig. 1 shows the iZQC spectrum of a 1:1 methyl-ethyl-ketone:water solution processed by the information theory and matrix pencil method. The iZQC spectrum shows the expected peaks at the precise frequency difference with correct phase alternation. More importantly, the J-splitting

structures are now resolvable. It may be concluded that the combined detection-estimation scheme provides the possibility of extracting accurate linewidth information in the presence of field instability and inhomogeneity in the Keck magnet. Such capability is crucial to our implementation of iZQC variants of the TROSY experiment to verify the theoretical prediction of destructive interference between chemical-shift-anisotropy and dipole-dipole relaxations within <sup>15</sup>N-<sup>1</sup>H moieties at very high fields.



**Figure 1.** Experimental iZQC spectrum of a 1:1 methyl-ethyl-ketone:water solution detected by CPMG-HOMOGENIZED sequence, and estimated by information theory and matrix pencil method.

**Acknowledgements:** This work was supported by the NIH under grant GM35253 and the NHMFL.

- <sup>1</sup> Warren, W.S., *et al.*, *Science*, **262**, 2005 (1993).
- <sup>2</sup> Lee, S., *et al.*, *J. Chem. Phys.*, **105**, 874 (1996).
- <sup>3</sup> Vathyam, S., *et al.*, *Science*, **272**, 92 (1996).
- <sup>4</sup> Lin, Y.-Y., *et al.*, *Phys. Rev. Lett.*, **85**, 3732 (2000).
- <sup>5</sup> Lin, Y.-Y., *et al.*, *J. Magn. Reson.*, **128**, 30 (1997).

## Carbon Isotopic Evidence for the Source and Fate of Dissolved Organic Matter in the Florida Everglades

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**T**he natural carbon isotopic ratios of dissolved organic compounds (DOC) reflect the sources and fates of DOC in an ecosystem. In the surface waters in the Florida Everglades, DOC can come from the historic peat deposits, “modern” wetland vegetation, and sugarcane (the dominant agricultural crop in the Everglades Agricultural Area). Stable carbon isotope analyses of DOC, plants, and soils collected from the northern Everglades indicate that less than about 23% of the DOC was derived from sugarcane, and the amount of DOC from sugarcane was greater under dry conditions. Water samples containing higher proportions of sugarcane-derived DOC also had higher amounts of soluble reactive phosphate and lower amounts of DOC and DOP (dissolved organic phosphorus). Most of the DOC (>50%) in the northern Everglades were in the low molecular weight (<1000 Dalton) fraction. The relative amount of high molecular weight (>1000 Dalton) DOC was higher in the wet period than in the dry period.

Radiocarbon ages of DOC in the northern Everglades ranged from “>modern” to about 2400 years B.P., indicating that DOC was derived from both historic peat deposits and modern vegetation. The very old radiocarbon ages of DOC in canal discharges indicate that the historic peat deposits in the Everglades Agricultural Area were a predominant source of DOC in the inflow waters to the Everglades Water Conservation Areas and the Everglades Nutrient Removal Project. In contrast, the DOC in the pristine area in the northern Everglades had “young” radiocarbon signatures and was primarily derived from recent photosynthate. At each site, the high molecular weight DOC (>1000 Dalton) had older radiocarbon ages than the low molecular weight DOC (<1000 Dalton), and therefore contained a greater fraction of DOC derived from the historic peat deposits. It appears that at least some of the old DOC from the historic peat deposits were decomposed by microbes during their residence in the surface water system in the northern Everglades, and the low molecular weight DOC was more microbially labile than the high molecular weight DOC. Our analysis suggests that accelerated decomposition of organic matter in the historic peat deposits (due to land-use change) could provide a significant source of DOC and nutrients for aquatic organisms in the northern Everglades. Our data

also suggest that the radiocarbon signature of DOC could be used as a sensitive indicator of the overall effectiveness of a wetland restoration project.

## Heat Capacity Measurements in NHMFL 60 T Quasi-Continuous Magnet

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**W**e have successfully demonstrated the feasibility of the specific heat measurements in pulsed magnetic fields up to 60 T in the NHMFL/LANL 60 T Long Pulse (60 T LP) Magnet, the primary objective of our project.

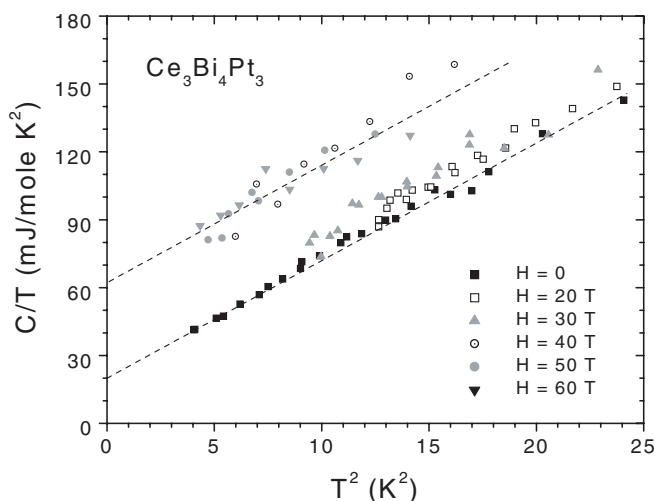
We used a quasiadiabatic heat pulse method that is based on delivering a known amount of heat to the sample, and measured its temperature before and after the heat pulse. It requires that the complete assembly of the sample, heater, and thermometer comes to thermal equilibrium before and after the heat pulse. This condition was impossible to satisfy in the short pulse (capacitor bank driven) magnets, where the *total* magnetic field pulse was on the order of ten milliseconds. The development of the 60 T long-pulse magnet at LANL, with the 100 millisecond *flat top*, opened the door for the equilibrium measurements in pulsed magnetic fields.

Working in a pulsed field environment necessitated development of a specialized probe. Made of plastic materials to avoid eddy current heating, it allows us to perform heat capacity measurements at temperatures between 1.6 K and 20 K (at present we are battling the systematic error in temperature above 4 K, increasing up to 20 % at 20 K) in fields up to 60 T. To maximize the available experimental space, a novel vacuum tapered seal was developed. The conical plug part of the joint is made out of G-10, and the matching vacuum can is made out of 1266 Stycast epoxy. The differential thermal contraction between the parts aids in producing a superfluid-tight joint. The simple construction of the joint resulted in a comparatively large (16 mm diameter) experimental region.

The quasiadiabatic method turns out to be applicable to a rather large variety of metallic and insulated compounds. The primary scientific result from that effort was observation of the insulator-to-metal transition in the Kondo Insulator,  $\text{Ce}_3\text{Bi}_4\text{Pt}_3$ , published in *Nature*, vol. **405**, 160 (2000), where we saw a threefold increase in the zero temperature Sommerfeld coefficient  $\gamma_{\text{H}} = C_{\text{H}}/T|_{T \rightarrow 0}$ , between the fields of

30 and 40 T. Other systems that were shown to be amenable to investigation with the heat-pulse method include valence-transition compound, YbInCu<sub>4</sub>, heavy fermions (such as UCd<sub>11</sub>, UBe<sub>13</sub>), and insulating compounds.

During magnetic field sweep, the temperature of the heat capacity stage does not stay constant even in the total absence of eddy current heating, due to the magnetocaloric effect. The heat capacity stage is thermally isolated from the bath, and remains in adiabatic condition during the magnetic field pulse. Such a system warms as the field is ramped up, and cools during the ramp down portion of the magnetic field pulse *reversibly*. However, magnetization in general can also increase with temperature. The sample then would cool during the ramp up, and warm *reversibly* during the ramp down of the magnetic field. We are using this effect to analyze the specific heat data collected UBe<sub>13</sub>, a possible electric quadrupolar Kondo system.



**Figure 1.** Specific heat divided by the temperature vs.  $T^2$  for  $\text{Ce}_3\text{Bi}_4\text{Pt}_3$  single crystal in magnetic fields between zero and 60 T. Dashed lines are guides to the eye.

## MOLECULAR CONDUCTORS

### Proof of Interplane Coherence Using Cyclotron Harmonics in the Organic Superconductor $\beta''\text{-(BEDT-TTF)}_2\text{SF}_5\text{CH}_2\text{CF}_2\text{SO}_3$

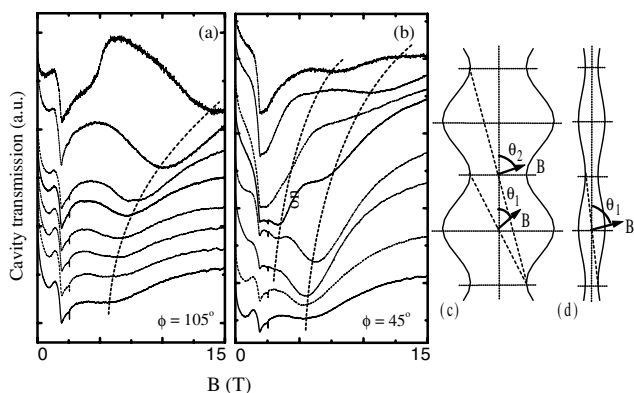
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**M**any correlated electron systems that are of fundamental or technological interest have very anisotropic electronic bandstructure. Examples include the “high- $T_c$ ”

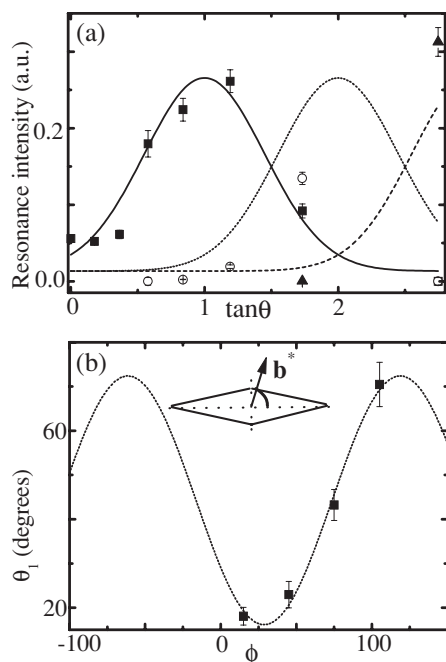
cuprates,<sup>1</sup> layered phases of the manganites<sup>2</sup> and ruthenates,<sup>3</sup> semiconductor superlattices,<sup>4</sup> and crystalline organic metals.<sup>5,6</sup> Such systems are often characterized by a tight-binding Hamiltonian in which the ratio of the interlayer transfer integral to the average intralayer transfer integral is much less than 1. The question arises as to whether these systems possess coherent or incoherent interlayer charge transfer;<sup>1,5-7</sup> i.e., whether or not their Fermi surface (FS) extends in the interlayer direction. Experimental tests for incoherence have thus far proved inconclusive; e.g., semiclassical models can reproduce angle-dependent magnetoresistance oscillation (AMRO) data equally well when the interlayer transport is coherent or “weakly coherent.”<sup>6,7</sup>

We report cyclotron resonance (CR), along with its second and third harmonics, in the quasi-two-dimensional organic superconductor  $\beta''\text{-(BEDT-TTF)}_2\text{SF}_5\text{CH}_2\text{CF}_2\text{SO}_3$ .<sup>8</sup> The magnetic field orientation dependence of the intensities of the fundamental CR and its harmonics very strongly suggests that the Fermi surface is extended in the interplane direction (i.e., that the interlayer charge transfer is coherent), in contradiction with recent proposals. Fig. 1 shows the magnetic field dependence of the transmission through a resonant cavity loaded with a sample for several values of  $\theta$  ( $\theta$  is the angle between the normal to the Q2D planes and the magnetic field); the cavity can rotate with respect to the applied field (i.e., change the angle  $\theta$ ).<sup>8,9</sup> Data for two values of  $\phi$  are shown ( $\phi$  defines the plane of rotation of the field). The feature at  $\sim 2$  T on all sweeps is a background of the apparatus.<sup>8</sup> At intermediate fields, broad resonances can be seen. For  $105^\circ$ , one resonance moves to higher fields with increasing angle. The position of the resonance in magnetic field for different angles is consistent with CR.<sup>6,8</sup> At  $\phi=45^\circ$ , another resonance appears for higher  $\theta$  at approximately half the field of the main CR. This resonance is also seen for  $\phi=75^\circ$  and  $15^\circ$ ; it is the second harmonic of the main CR. At some  $\theta, \phi$ , a third harmonic of the CR becomes visible.

Fig. 1(c) shows the cross section of a warped Q2D FS section (warping greatly exaggerated). For a general orientation of the field, quasiparticles follow orbits about the FS such that the  $z$ -component of their real space velocity,  $v_z$ , oscillates. In our measurement geometry, the dissipation caused by the sample is dominated by interplane currents, i.e., by the behavior of  $v_z$ . If  $\theta$  is small, the orbits remain within the length of one Brillouin zone (BZ) in the  $k_z$ -direction and  $v_z$  oscillates at the cyclotron frequency  $\omega_c$ . As  $\theta$  increases, the orbits extend over several BZs in the  $k_z$ -direction and  $v_z$  acquires oscillatory components at harmonics of  $\omega_c$ . In this way, by assuming an extended Fermi surface in the  $k_z$  direction, the angular behavior of the second and third harmonics can be predicted (Fig. 2a), as can the value of  $\theta, \phi$  at which the maximum amplitude of a particular harmonic occurs (Fig. 2b).<sup>8</sup>



**Figure 1.** (a) Transmission of the resonant cavity loaded with a single crystal of  $\beta''$ -(BEDT-TTF) $_2$ SF $_5$ CH $_2$ CF $_2$ SO $_3$  versus magnetic field for  $\theta = 0$  (lowest trace) to  $70^\circ$  (uppermost trace) and  $\phi = 105^\circ$  ( $T = 1.5$  K); the frequency is 70.2 GHz. (b) Equivalent data for  $\phi = 45^\circ$ . (c) and (d) represent two different cross-sections of a warped cylindrical FS; for clarity the warping has been very greatly exaggerated. Cyclotron orbits about the FS are shown schematically as dotted lines for two inclinations of the magnetic field  $B$  to the cylinder axis,  $\theta_1$  and  $\theta_2$ .



**Figure 2.** (a) Intensity of CRs versus  $\tan\theta$  for  $\phi = 75^\circ$ . Solid curve: predicted intensity of the fundamental CR (data: square points). Dotted curve: predicted intensity of the second harmonic, (data: hollow circles). Dashed curve: predicted intensity of the third harmonic (data: triangles). (b) Predicted  $\theta, \phi$  position of the maximum intensity of the fundamental CR (curve); points are experimental values. Inset: the orientation of the FS cross-section to the crystal's  $\mathbf{b}^*$  axis derived from the theory.

## Pressure Dependence of the Quantum Hall Effect of $(\text{TMTSF})_2\text{ReO}_4$

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Jo, Y.J., Ewha Womans Univ., Korea, Physics

Kang, W., Ewha Womans Univ., Korea, Physics

Quantum Hall effects are readily observed in the quasi-one-dimensional organic conductors such as  $(\text{TMTSF})_2\text{ClO}_4$ ,<sup>1</sup>  $(\text{TMTSF})_2\text{PF}_6$ ,<sup>2</sup> and  $(\text{TMTSF})_2\text{ReO}_4$ .<sup>3</sup> However, contrary to the conventional QHE in semiconductors, unusual features such as Hall sign reversal,<sup>4</sup> missing quantum indices, etc., are commonly observed in organic conductors. Even in  $(\text{TMTSF})_2\text{PF}_6$ , which is believed to be the simplest in the series, Hall sign reversal has been observed over a certain pressure range. Although the standard model of quantized nesting theory<sup>5</sup> has offered a firm basis for the QHE, there is no consistent understanding for these additional features.  $(\text{TMTSF})_2\text{ReO}_4$  is the least studied probably because of its highest critical pressure necessary to stabilize the metallic state at low temperature.

We have performed the Hall resistance measurement of  $(\text{TMTSF})_2\text{ReO}_4$  under various hydrostatic pressures. Several new features drew our attention in the present study. First, in addition to the previously known negative Hall step around 15 T, at least two more new negative indexed quantized states have been observed. Interestingly enough, the negatively indexed states seem to form their own quantum Hall sequence independently from the positively indexed states at high field. Second, low field negatively indexed states are better developed at low pressure while the positively indexed states favor high pressure. When the field is normalized, only the transition fields for low field states could be scaled, to which the standard model can be applied. Third, an isolated Hall resistance peak appears for the narrow pressure interval around 14 kbar.

Most of these experimental findings cannot be explained with the quantized nesting model. We believe that a superlattice potential due to anion ordering plays an important role. Further analysis in consideration of possible mixed anion ordering and in comparison with other compounds will be reported elsewhere.

<sup>1</sup> Ioffe, L.B., *et al.*, Science, **285**, 1241 (2000).

<sup>2</sup> Rao, C.N.R., J. Mater. Chem., **9**, 1 (1999).

<sup>3</sup> Bergemann C., *et al.*, Phys. Rev. Lett., **84**, 2662 (2000).

<sup>4</sup> Kelly, M.J., Low Dimensional Semiconductors (Oxford University Press 1995).

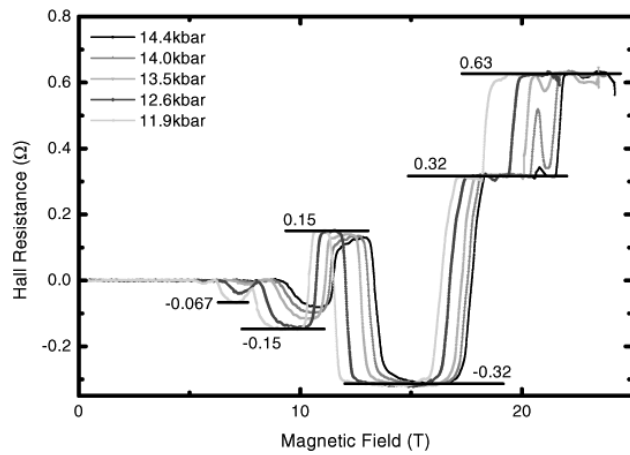
<sup>5</sup> Strong, S.P., *et al.*, Phys. Rev. Lett., **73**, 1007 (1994).

<sup>6</sup> Singleton, J., Reports on Progress in Physics, **63**, 1111 (2000).

<sup>7</sup> McKenzie, R.H., *et al.*, Phys. Rev. Lett., **81**, 4492 (1998).

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**Figure 1.** The pressure dependence of Hall resistance at around 200 mK. All the values of pressure were measured at room temperature by manganin resistance inside the cell, and the real pressure at low temperature was lower than that by 2 kbar. The Hall resistance data for  $N = 0$  were intentionally removed for clarity.

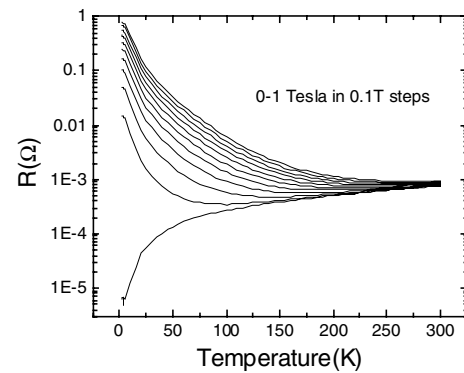
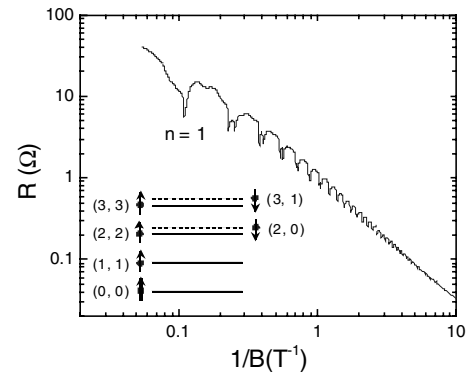
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- <sup>2</sup> Cooper, J. R., *et al.*, *Phys. Rev. Lett.*, **63**, 1984 (1989).
- <sup>3</sup> Kang, W., *et al.*, *Phys. Rev. B*, **43**, 11467 (1991).
- <sup>4</sup> Balicas, L., *et al.*, *Phys. Rev. Lett.*, **75**, 2000 (1995).
- <sup>5</sup> Gor'kov, L. P., *et al.*, *J. Phys. Lett.*, **45**, L433 (1984).

## OTHER CONDENSED MATTER

### Magnetotransport in High Purity Bismuth Crystals

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**M**agnetotransport has been investigated in high purity bismuth crystals in fields as high as 20 T and temperatures as low as 20 mK. This high  $B/T$  ratio permits observation of pronounced Shubnikov-de Haas oscillations (Fig. 1, top panel) up to fields where most of the carriers are in the lowest Landau level. The doublet splittings centered on each Shubnikov-de Haas oscillation exhibit a quadratic dependence on field and disappear before the last ( $n = 1$ ) oscillation. The inset of Fig. 1 is a schematic of the energy levels showing the spin direction and identifications ( $n, \nu$ ) of the order of oscillation  $n$  and the Landau level  $\nu$ . These observations allow us to conclude unambiguously that, for  $\nu \leq 2$ , the carriers are fully polarized and the  $g$ -factor for holes with the field in the trigonal direction is 35.3(4).<sup>1</sup>



**Figure 1.** Longitudinal resistance plotted as a function of reciprocal field at 25 mK (top panel) and as a function of temperature at the indicated fields (bottom panel).

By sweeping the temperature in fixed fields (0 to 1.0 T in 0.1 T steps), the family of curves shown in Fig. 1 (bottom panel) is obtained. The magnetic field has a pronounced effect, giving a more than five-decade change in the resistance at 2 K. The magnetoresistivity data,  $\rho(T, B) - \rho(T, 0)$ , are well described for two separate samples by the functional dependence,  $A_1 B^{2-\eta} / (1 + A_2 T^2 B^{-\eta})$  where  $A_1$ ,  $A_2$ , and  $\eta$  are constants. At low fields, the expected quadratic dependence,  $B^2$ , is recovered. However, the quadratic temperature dependence in the denominator is inconsistent with the linear temperature dependence predicted by the two-band model for a perfectly compensated metal, thereby necessitating a reexamination of our understanding of the magnetoresistance of bismuth.

**Acknowledgements:** The authors would like to thank Professor R. Goodrich for the loan of high quality bismuth crystals, and the In-House Research Program of the NHMFL at Tallahassee for supporting this work.

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<sup>1</sup> Bompadre, S.G., *et al.*, submitted to *Phys. Rev B* (cond-mat/0006241).

## Anomalous Behavior of Spin Fluctuations in Polycrystalline $\text{NdBa}_2\text{Cu}_3\text{O}_7$

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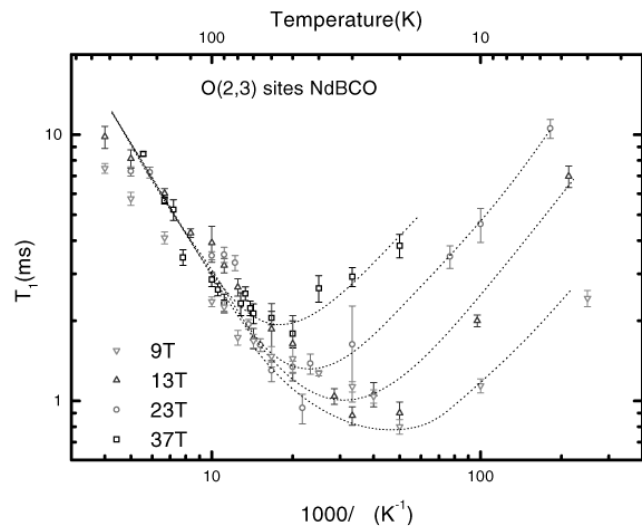
It is well known that substitution of a rare-earth (RE) magnetic ion to Y in  $\text{YBa}_2\text{Cu}_3\text{O}_7$  produces little or no effect in transition temperature  $T_c$ . The interplay between magnetism and superconductivity in these materials have been controversial and NMR have played an important role in its theoretical understanding. In this light, an  $^{17}\text{O}$  NMR study of  $\text{NdBa}_2\text{Cu}_3\text{O}_7$  was recently undertaken with some unexpected results. The Nd ions have localized moments and orders antiferromagnetically at  $T_N \sim 0.5$  K, but the  $T_c$  of  $\text{NdBa}_2\text{Cu}_3\text{O}_7$  remains at 92 K. Unlike most RE, the Nd ion is too large to fit in the lattice and tends to occupy the Ba site; the only other case being  $\text{PrBa}_2\text{Cu}_3\text{O}_7$ . The  $^{17}\text{O}$  NMR spectra and spin-lattice relaxation time  $T_1$  of magnetically aligned polycrystals were measured for two different O sites as a function of temperature at different magnetic fields, including 37 T in the hybrid magnet. Fig. 1 shows  $T_1$  vs.  $1/T$  at various fields for the planar O(2,3) sites.

There are several features to note: (a) extremely fast relaxation—the  $T_1$  is nearly 2 orders of magnitude shorter than that of  $\text{YBa}_2\text{Cu}_3\text{O}_7$ ; (b) there is no evidence of the “pseudogap” above  $T_c$  nor a signature of superconducting transition at  $T_c$ ; and (c) there is an anomalous field-dependent minimum in  $T_1$  which lies well below  $T_c$  and much above  $T_N$ . The Cu NMR in this system is not observable due to short  $T_1$ . This fast relaxation is almost certainly due to Nd spin fluctuations which, in addition to Cu, contributes significantly and possibly dominates and masks the pseudo-gap because of its large moment.

The minimum in  $T_1$  below  $T_c$  has not been observed in other cuprate superconductors. The physical origin of this behavior is unexpected considering the fact that the antiferromagnetic ordering of the Nd occurs more than an order of magnitude lower in temperature. Typically,  $T_1$  minimum (or a peak in rate  $1/T_1$ ) is understood as a signature of critical slowing down, and possibly “freezing,” of the spin fluctuations. The minimum therefore corresponds to  $\omega\tau_c \sim 1$ , where  $\tau_c$  is the correlation time of the Nd spins and  $\omega$  is the NMR frequency. In this picture, the field dependence originates from the behavior of spin-fluctuation

power spectrum beyond the  $1/\tau_c$  cutoff, which goes as  $\propto \omega^2$ . The magnitudes of  $T_1$ , at temperatures below the minimum qualitatively scale with field as expected from this speculation. However, data on the apical  $\text{O}_4$  site (not shown) also shows a minimum in  $T_1$  at about 20 K which, however, is somewhat less field independent, and thus appears inconsistent with the speculations as to the mechanism. On the other hand, the O(4) is much more weakly coupled to the Nd than the O(2,3), as indicated by the almost one order of magnitude longer  $T_1$ , and may possibly have considerable contribution from Cu.

This phenomenon, while probably unimportant to the superconductivity, raises interesting questions as to how, in spite of a very tight coupling of the charge and spin in this system, the critical balance with magnetism is maintained to preserve superconductivity.



**Figure 1.** Plot of  $T_1$  vs.  $1/T$  for  $^{17}\text{O}$  NMR at the O(2,3) sites in  $\text{NdBa}_2\text{Cu}_3\text{O}_7$  for various fields. Lines are guides to the eye.

# Closing the Pseudogap by Zeeman Splitting in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$ at High Magnetic Fields

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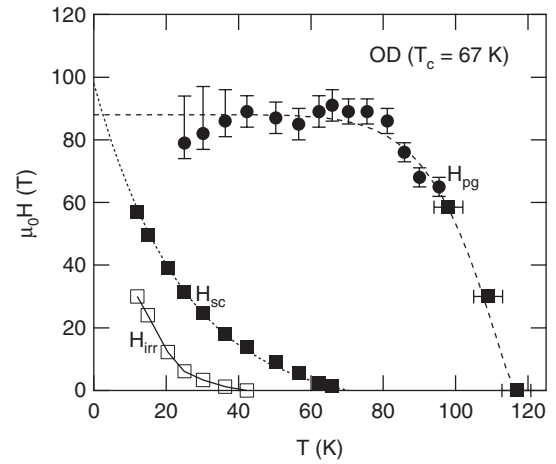
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**C**urrent knowledge about the field dependence of the pseudogap in the normal state of high temperature superconductors is partly limited by the available DC field range. More importantly, there is no systematic doping dependence in a single family of cuprates. We report the interlayer (*c*-axis) resistivity  $\rho_c$  measurements in fields up to 60 T using a long pulse (LP) system and a 33 T DC system at the NHMFL in  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  (BSCCO) crystals in a wide range of doping. From these experiments, we make a first systematic evaluation of the pseudogap closing field  $H_{pg}$  that restores low-energy density of state (DOS) to its ungapped state. Our results indicate a pronounced difference between field-temperature diagrams of the pseudogap and the superconducting states and a simple Zeeman scaling between  $H_{pg}(0)$  and the pseudogap temperature  $T^*$ .

In slightly underdoped BSCCO at temperatures below  $T_c$ , the field dependence of  $\rho_c$  exhibits a peak that we have previously demonstrated to arise from a competition between two tunneling conduction channels: Cooper pairs (at low fields) and quasiparticles (mainly at high fields).<sup>1</sup> The peak position  $H_{sc}$  marks the field (in the superconducting state) where the quasiparticle contribution overtakes the Cooper pair tunneling current. The doping dependence of  $\rho_c$  clearly shows that the peak field  $H_{sc}$  in the highly underdoped crystal, where interlayer (Josephson) coupling is the weakest, is most easily suppressed. Magnetoresistance (MR) above  $H_{sc}$  is negative and remains so above  $T_c$ . In the overdoped samples, negative MR eventually disappears. This occurs at the same temperature at which the zero-field  $\rho_c(T)$  develops a characteristic upturn from the T-linear dependence of the metallic state. This temperature is identified as the pseudogap temperature  $T^*$ . The negative MR below  $T^*$  is naturally understood by the suppression of the pseudogap by magnetic field. In our most overdoped crystal with  $T_c = 67$  K, a magnetic field of 60 T downshifts the  $\rho_c(T)$  upturn and the associated  $T^*$  by about 20 K. In other words, at this doping level, the 60 T field at  $\sim 100$  K closes the pseudogap. To track the pseudogap closing field at lower temperatures, we consider the excess resistivity  $\Delta\rho_c$  due to the DOS depletion by subtracting the metallic contribution. The field at which  $\Delta\rho_c$  vanishes is the

pseudogap closing field  $H_{pg}(T)$ . A fit to the power-law field dependence of  $\Delta\rho_c(H)$  at different temperatures allows us to evaluate  $H_{pg}(T)$  beyond 60 T.

The entire H-T diagram of the pseudogap in the overdoped crystal is shown in Fig. 1. At low temperatures  $H_{pg}$  is essentially flat with the limiting value of  $\sim 90$  T. This is in marked contrast with the characteristic fields of the superconducting state: the peak field  $H_{sc}(T)$  and the irreversibility field  $H_{irr}(T)$ . This difference may indicate different origins of the pseudo- and superconducting gaps. The doping dependence of the low-temperature  $H_{pg}$  and the zero-field  $T^*$  leads to a strikingly simple conclusion. The pseudogap closing field scales with  $T^*$  as  $g\mu_B H_{pg} \sim k_B T^*$ . Here  $g=2.0$ ,  $\mu_B$  is the Bohr magneton, and  $k_B$  is the Boltzmann constant. This immediately suggests that magnetic field couples to the pseudogap by the Zeeman energy of the spin degrees of freedom. Our finding that Zeeman splitting closes the pseudogap implies that the triplet spin excitation at high fields overcomes the singlet pair correlations responsible for the gap in the spin spectrum, and that the orbital contribution is very small.



**Figure 1.** H-T diagram showing the pseudogap closing field  $H_{pg}(T)$ , the peak field  $H_{sc}(T)$ , and the irreversibility field  $H_{irr}(T)$  in an overdoped BSCCO. Up to 60 T,  $H_{pg}(T)$  is directly determined from the down-shifting upturn of  $\rho_c(T)$  (squares). At lower temperatures,  $H_{pg}(T)$  is obtained by extrapolating  $\Delta\rho_c(H)$  to zero. The two procedures consistently produce a seamless  $H_{pg}(T)$  within the error bars.

**Acknowledgments:** We thank N. Morozov, F.F. Balakirev, J. Betts, C.H. Mielke, and B.L. Brandt for technical assistance, and L.N. Bulaevskii, N. Nagaosa, V.B. Geshkenbein, L.B. Ioffe, C.C. Tsuei, and D.M. Newns for helpful discussions. This work was supported in part by NSF through NHMFL by the Contract No. AL99424-A009.

<sup>1</sup> Morozov, N., *et al.*, Phys. Rev. Lett, **84**, 1784-1787 (2000).

## Reorientation of Anisotropy at $\nu=9/2$ and $11/2$ in a Square Well Quantum Hall Sample Under a Tilted Magnetic Field

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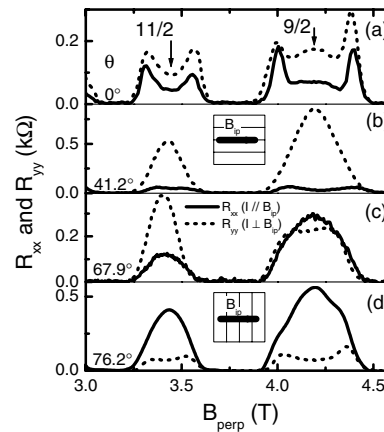
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Most recently, strongly anisotropic transport has been observed in high quality GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As samples at filling factors  $\nu=9/2$ ,  $11/2$ , etc. In these experiments, the magnetoresistance shows a strong peak in one current direction and a deep minimum in the perpendicular current direction. Tilting the magnetic field,  $\mathbf{B}$ , away from the sample normal causes the high resistance direction to change from its original orientation to the in-plane magnetic field ( $B_{ip}$ ) direction. The most appealing interpretation of this anisotropy suggests that the 2D electron gas spontaneously breaks the translational symmetry by forming a unidirectional charge density wave (UCDW).

A theoretical study,<sup>1</sup> in samples with two subbands occupied in zero magnetic field, has predicted much more complex behavior of the UCDW state, including stripe states induced by an in-plane field and rotation of stripe orientation at critical in-plane field strengths. A comparison between theory and experiment in a geometry for which this intricate behavior occurs, constitutes an excellent test of the UCDW explanation of anisotropic transport in higher Landau levels. We have thus chosen a square quantum well structure with two occupied electric subbands and measured magnetotransport at half-filled high Landau levels.<sup>2</sup> We find resistivities that are practically *isotropic* in perpendicular magnetic field, but become strongly *anisotropic* at  $\nu=9/2$  and  $11/2$  on tilting the field. The anisotropy appears at an in-plane field,  $B_{ip} \sim 2.5$  T, with the easy-current direction *parallel* to  $B_{ip}$ , but rotates by  $90^\circ$  at  $B_{ip} \sim 10$  T and points now in the “normal” direction (*perpendicular* to  $B_{ip}$ ). This complex behavior is in quantitative agreement with theoretical calculations based on a unidirectional charge density wave state model.

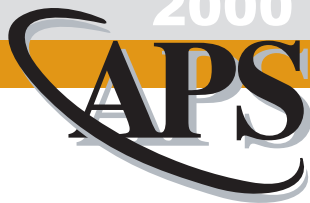


**Figure 1.**  $R_{xx}$  (solid line) and  $R_{yy}$  (dotted line) between  $4 < \nu < 6$  at four tilt angles in a square quantum well with two occupied electric subbands.

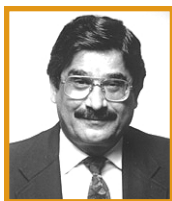
<sup>1</sup> Jungwirth, T., *et al.*, Phys. Rev. B, **60**, 15574 (1999).

<sup>2</sup> Pan, W., *et al.*, Phys. Rev. Lett., **85**, 3257 (2000).

## 2000 APS Fellows



**A**merican Physical Society Fellowships recognize members who make “outstanding contributions to physics.” The NHMFL is pleased to spotlight three colleagues and friends of the laboratory who were inducted at the spring APS meeting held in Seattle on March 12-16, 2001:



**Naresh Dalal**, professor and chair of chemistry at the Florida State University, is recognized “for his development of electron and nuclear multiple resonance methods with much enhanced time scales and spectral resolution, and especially for their application to measure molecular dynamics and to elucidate mechanisms of ferroelectric phase transitions.” He is the fourth FSU chemist to gain this distinction in physics, succeeding Michael Kasha, Alan Marshall, and Geoffrey Bodenhausen. (APS Division of Chemical Physics Fellow).



**Meigan Aronson**, associate professor of physics at the University of Michigan, is recognized “for investigation of collective phenomena in strongly correlated electron systems using neutron scattering and high pressure techniques.” Her research focuses on collective phenomena in low carrier density systems and in quantum magnets. She is a frequent user of NHMFL facilities and has served on the NHMFL Users’ Committee for several years. (APS Division of Condensed Matter).



**Michael Coey**, professor of physics at Trinity College, Dublin, is recognized “for contributions to magnetism including discovery of rare-earth iron nitrogen permanent magnets, classification of magnetic order in amorphous solids, and innovative applications of permanent magnets.” He is an NHMFL user and was featured on the cover of the Spring 1999 NHMFL Reports in an article entitled “Mysteries of Magnetochemistry.” (APS Division of Magnetism and Its Applications).

## Conference & Workshop Activity

### **Fifth MRS/ISTEC Joint Workshop on High Tc Superconductivity**

June 24-27, 2001

Honolulu, Hawaii

The NHMFL is a partial sponsor for this workshop. The general theme is Processing and Applications of High Tc Conductors, with a special focus on HTS Coated Conductors. Other U.S. sponsors include Oak Ridge National Laboratory, Los Alamos National Laboratory, Argonne National Laboratory, and the University of Wisconsin. ISTEC is a Japanese organization; MRS is the Materials Research Society.

### **24<sup>th</sup> International EPR Symposium**

July 29-August 2, 2001

Denver, Colorado

Pre-Conference Registration Deadline: July 1, 2001

<http://www.du.edu/~seaton/eprsym.html>

<http://www.milestoneshow.com/rmcac/>

The 24<sup>th</sup> International EPR Symposium will be held in conjunction with the 43<sup>rd</sup> Annual Rocky Mountain Conference. About 150 people participate in the EPR Symposium each year, presenting over 100 papers. Approximately 1,000 people attend the Rocky Mountain Conference, which also includes an NMR Symposium and instrument exhibit.

The International EPR Symposium covers all aspects of EPR spectroscopy. This year there will be sessions emphasizing 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.

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

September 3-7, 2001

San Carlos de Bariloche, Argentina

Pre-Conference Registration Deadline: June 30, 2001

<http://www.cab.cnea.gov.ar/calendario/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 the Latin American community of scientists and engineers interested in 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.

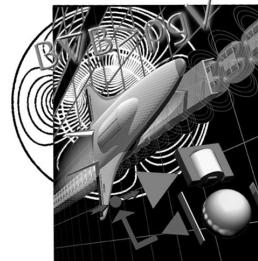
## 6<sup>th</sup> International Symposium on Magnetic Suspension Technology

October 7-11, 2001

Turin, Italy

<http://www.lim.polito.it/ISMST6>

The 6<sup>th</sup> 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](mailto:ismst6@magnet.fsu.edu) (NHMFL in Tallahassee) or [ismst6@polito.it](mailto:ismst6@polito.it) (Politecnico di Torino).

## Physical Phenomena at High Magnetic Fields (PPHMF-IV)

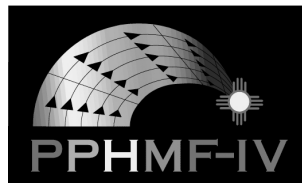
October 19-25, 2001

Santa Fe, New Mexico

Hotel Headquarters: Hilton and Radisson of Santa Fe

Pre-Conference Registration Deadline: June 1, 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. The abstract deadline is June 1, 2001.

## Applied Superconductivity Conference (ASC04)

October 4-8, 2004

Site: 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](mailto:schwartz@magnet.fsu.edu).

# REPORTS

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