

# Low Temperature Measurements of Novel Low-Dimensional Systems<sup>§</sup>

James R. Maloney<sup>‡</sup> and James M. Stock<sup>†</sup>  
Department of Physics, University of Florida,  
Gainesville, FL 32611-8440

(August 2, 2000)

## ABSTRACT

In order to investigate the structural and electromagnetic properties of novel low-dimensional systems at low temperatures, we constructed a versatile sample cell for use in a cryogenic probe between 1.5 K and 300 K. More specifically, we examined the phase transitions in  $(\text{CH}_3)_2\text{NH}_2\text{CuCl}_3$  (MCCL) and aspects of conductivity in Langmuir-Blodgett (LB) films. Neutron scattering indicates the presence of structural phase transitions of MCCL at temperatures around 10 K and 150 K. Our measurements attempted to isolate the temperature regions of the MCCL transitions by monitoring capacitance, yielding a possible transition point at 154 K. In addition, we monitored the lossy contributions to conductivity of LB films and aluminum foil as a response to signal frequency at 300 K. The results indicate an increased lossy contribution from the LB film and noise coupling to the blank mylar in the sample cell. Our measurements employed an ac capacitance bridge and lock-in amplifier.

---

<sup>§</sup>Work performed in collaboration with B.C. Watson, S.E. Gerofsky,  
and M. W. Meisel (Email: [meisel@phys.ufl.edu](mailto:meisel@phys.ufl.edu))

<sup>‡</sup>Supported by NHMFL, REU Program; Email: [james@phys.ufl.edu](mailto:james@phys.ufl.edu)

<sup>†</sup>Supported by NSF, UF REU Program; DMR-9820518 Email: [jstock@stetson.edu](mailto:jstock@stetson.edu)  
Physics Department; Stetson University; DeLand, Florida

## INTRODUCTION

The discovery of high critical temperature ( $T_c$ ) superconductors, which can be modeled as low-dimensional systems, has generated a heightened interest in the investigation of these types of materials. In order to investigate the structural and electromagnetic properties of two such systems,  $(\text{CH}_3)_2\text{NH}_2\text{CuCl}_3$  (MCCL) and Langmuir-Blodgett (LB) films, we designed a versatile technique to examine these samples over a extensive temperature range. The technique required the design, construction, and operation of an experimental sample cell for a cryogenic probe. The measurement techniques involved bridge balancing and lock-in detection.

## LOW-DIMENSIONAL SYSTEMS

Low-dimensional systems may be described as one-dimensional chains or two-dimensional sheets. Under most conditions the magnetic system of MCCL is one-dimensional, making MCCL an ideal choice for our first series of measurements. Previous experiments using neutron scattering techniques indicated that MCCL has structural phase transitions in the temperature regions around 10 K and 150 K [1]. However, difficulty in the precise location of the transitions through the use of neutron scattering measurements merited the development of a different approach. We decided to use MCCL as a dielectric medium in a capacitor. Capacitance could then be monitored over the desired temperature ranges in the hopes that a structural phase transition would produce a change in the material's dielectric constant.

We also investigated LB film for its low-dimensional properties. LB films are self-assembled thin films that form as two-dimensional sheets called bilayers (a region of hydrophobic-hydrophilic components on both sides of a chemically prepared surface). See Fig. 1. The Talham Group, University of Florida, Department of Chemistry, is currently developing techniques for which LB films with desired magnetic and conducting properties can be synthesized. Previous current-voltage (I-V) studies of the LB film failed to detect any macroscopic conductivity, suggesting the LB film was not reaching its percolation threshold (the point at which the film becomes conductive rather than insulating) [2].

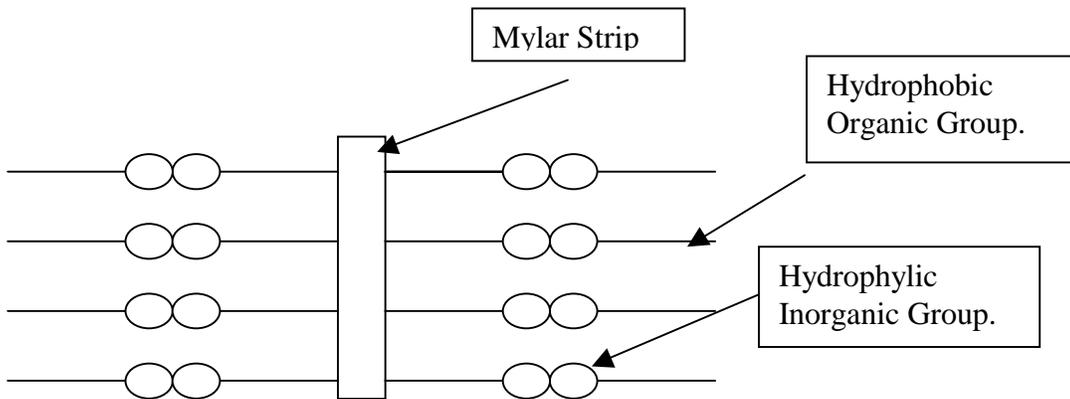


Fig. 1. A single bilayer of LB film.

While the former studies implied that the percolation threshold was not attained, our motive for developing the capacitive measurement technique was to investigate whether microscopic islands of conductivity still existed. We analyzed the lossy (real) or resistive ( $\rho$ ) term of a signal in response to the frequency at which the signal was

applied to our sample. We then monitored the conductivity ( $\sigma \equiv \frac{1}{\rho}$ ) in an attempt to quantify the LB film's conductivity, albeit on a microscopic scale.

## SAMPLE CELL

We housed the materials under investigation in a sample cell capable of an assortment of measurements. The sample cell was designed with TURBO CAD for use with a pre-constructed cryogenic probe capable of temperatures ranging from 1.5 K to 300 K. The cavities of the sample cell become two identical parallel plate capacitors when assembled as illustrated in Fig. 2 (a). Their wiring schematic as seen in Fig 2 (b) allows for (i) a single three-wire measurement or (ii) two independent two-wire measurements.

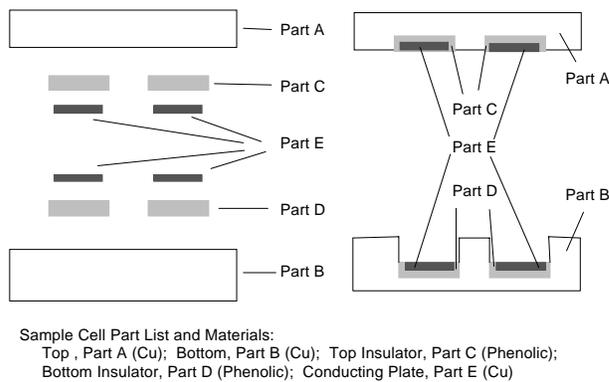


Fig 2. (a) Layered model of the sample cell.

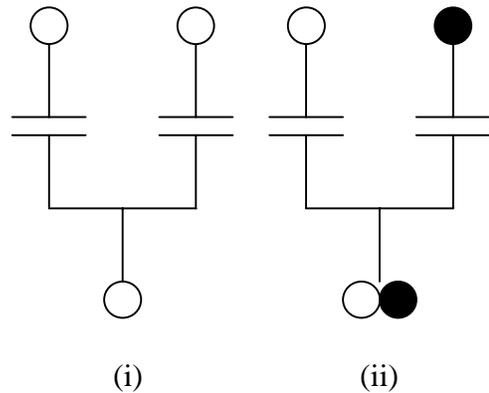


Fig 2. (b) Electrical schematics of the sample cell.

The initial two-wire analysis of MCCL involved only one cavity of the sample cell. MCCL chunks were crushed into small pieces, which still maintained the structural

integrity of their crystal lattices. These pieces were placed between the conducting plates of one cavity to act as a dielectric medium.

The LB film was deposited in bilayers onto both sides of a mylar sheet and oxidized. This composite material necessitated the use of both cavities of the sample cell in taking conductivity measurements. An LB film and mylar disk were each cut to fit into a cavity of the sample cell. Two-wire measurements were taken across each cavity so that the mylar background could be subtracted.

Conductivity measurements were also taken from an aluminum foil model, which attempted to simulate the islands of conductivity expected in the LB film. One small disk, three small disks, and one large disk were each, in turn, placed between mylar disks in one cavity and measured in reference with two mylar disks in the other cavity.

## MEASUREMENT TECHNIQUES

Whenever a measurement is made in the laboratory, it has a corresponding uncertainty. Since this uncertainty places a limit on the accuracy of a measurement, experimental physics pays great attention to this subject. We utilized several voltage techniques to achieve a high level of accuracy and reduce unwanted noise.

The most basic voltage measurement would be a two-wire dc measurement as shown in Fig. 3(a). A dc current ( $I$ ) sent through the sample from lead A to lead B creates a voltage drop across leads A and B ( $V_{AB}$ ). From Ohm's Law  $V_{AB} = I (R_S + R_A + R_B)$  where  $R_S$ ,  $R_A$ , and  $R_B$  represent the resistances of the sample, lead A, and lead B respectively [3]. However, lead resistances always influence two-wire

measurements. To overcome this complication, four-wire measurements are employed whenever possible.

A four-wire measurement, as seen in Fig. 3(b), also sends a current ( $I$ ) through the sample along leads A and B. The voltage drop measured across C and D no longer takes into account lead resistances because no current is flowing through C and D [4]. Now,  $V_{CD} = I R_S$ .

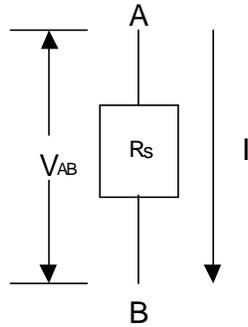


Fig. 3(a) Two-wire measurement.

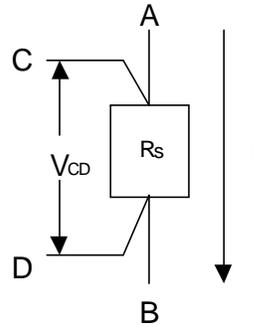


Fig. 3(b) Four-wire measurement.

These techniques still have a considerable drawback if one is monitoring small changes in voltage ( $\delta V$ ). A large constant voltage ( $V$ ) will considerably mask the signal of interest,  $\delta V = (V + \delta V) - V$ . At first glance this equation may appear to be equivalent to  $\delta V$ . However, uncertainty ( $E$ ) in voltage is propagated in quadrature, so that the resulting uncertainty ( $E_{\delta V}$ ) is  $E_{\delta V} = \sqrt{2E^2 + E_{\delta V}^2}$ . Due to this propagation of error the "Golden Rule of Experimental Science" is to always take a measurement about null (zero). A bridge circuit accomplishes this task.

The Wheatstone bridge is a model example of this class of circuits. A meter detector (M) measures the potential difference across the bridge. A derivation using Kirchhoff's Laws illustrates that the potential across the bridge ( $V_{AB} = V_A - V_B$ ) can be nulled via the variable element ( $R_3$ ). The bridge is then balanced, allowing for any change in these conditions to give rise to a voltage signal with the least amount of uncertainty [5].

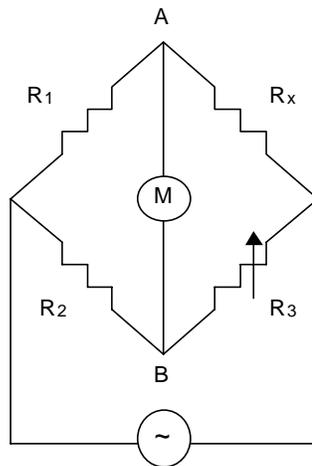


Fig. 4. Schematic of a Wheatstone Bridge.  
 $R_x$  is the sample's resistive component;  $R_1$  and  $R_2$  are selected to create an acceptable balancing range; and  $R_3$  has been tuned so that  $V_{AB} = 0$ .

However, noise from dc schemes can arise from thermal electromotive forces (TEMFs), potential differences caused by temperature gradients. These gradients were of considerable concern for the design of our probe's temperature ranges. TEMFs may be accounted for through the use of an ac scheme [6]. Thus, we chose an ac bridge, which consisted of impedance elements and an ac current source to take our measurements. The

immense reduction in noise through the use of ac schemes is facilitated by a lock-in amplifier, which measures very small ac signals.

Two important features of the lock-in amplifier are the improvements of signal-to-noise ratio in weak ac signals and phase sensitive detection (PSD), or the ability to separate the real and imaginary components of a signal. The lock-in amplifier increases the signal-to-noise ratio by creating a narrow bandwidth tuned to the sample signal's frequency and then filtering out unwanted noise. The sample's signal is then passed through an amplifier capable of increasing its order of magnitude by  $10^9$ . The PSD uses an internal oscillator to generate a constant voltage sine wave, which acts as a reference for the lock-in. The PSD compares the reference signal to itself after it is passed through the sample, after which the PSD is able to separate the signal into components.

## **LOW TEMPERATURE TECHNIQUES**

Careful consideration was required for the cryogenic probe and sample cell to attain the temperature ranges of interest. The probe contained copper baffles as shields to suppress heat leaks due to thermal radiation. Heat leaks were also attributed to Cu-Ni wires that ran from room temperature (300 K) to the 1 K pot (a storage unit for liquid He).

The wires were thermally grounded via anchoring to a Cu mount and GE varnished to reduce the temperature gradient. Twisted pairs of wires were also used to suppress the magnetically coupled interference between the wires [6]. We also took into

account the thermal contraction of our cell and constructed it of Cu to match the base material of the probe.

The thermometers were mounted to the probe base using Apiezon Grease, which allowed for thermal contact without electrical grounding. The heater was made of two parallel  $1\text{ k}\Omega$  metal film resistors to keep constant resistances over a wide temperature range.

An evaporative cooling technique is used to reach the probe's lowest attainable temperatures. Liquid He continuously flows into the 1 K pot, and boils off due to the high vacuum (100 millitorr) maintained upon the pot. This evaporation process is an endothermic reaction enabling us to generate temperatures close to 1.5 K, significantly cooler than that of liquid He (4 K).

The Inner Vacuum Can (IVC) housing the sample cell, 1 K pot, and thermometry also required high vacuum conditions to retain homogenous atmospheric conditions. High vacuum conditions required the use of special seals to maintain isolated systems. We used an Indium O-ring seal for the IVC to thwart any attempt of liquid He to flow into the IVC. The IVC pressure was monitored with a thermocouple.

At low temperatures, vibration of sample cell components contribute to the noise level in electrical signals. To prevent such random noise, GE varnish and dental floss were used to secure any loose items free to vibrate.

## RESULTS

Preliminary capacitance data taken from 77 K up included a slight peak relative to the background curve between 153 K and 157 K. See Fig. 5. This temperature range corresponds to the predicted region for structural transition indicated by the neutron scattering experiments [1]. However, a second run from 150 K up with the same sample did not indicate such a transition, suggesting the MCCL to be hysteretic or that the crystal lattice was damaged in such a manner as not to produce the same transition more than once. Further experiments will be conducted to test the reproducibility in the data. The smooth capacitance transitions represented by the curve marked on either side of the peak in Fig. 5. can be attributed to the thermal expansion of the sample cell and its contents.

This may be shown dimensionally with  $C = \frac{K \cdot A}{d} = K \left[ \frac{L^2}{L} \right] = K \cdot L$ , where  $C$ ,  $d$ ,  $A$ ,  $L$ ,

and  $K$  are the capacitance, separation distance, area, length unit, and dielectric constant, respectively. Any changes in the dielectric constant of the sample should be noticed as deviations away from this trend. We intend to use a three-wire ratio transformer measurement technique for future experimental runs. We hope this technique will reduce even more background noise by taking a ratio of the two cavities, which are expected to expand at the same rates.

The LB film conductivity demonstrated a small increase to the lossy term due to the presence of the LB film. This increase is demonstrated at each frequency level by the higher conductivity values for the mylar with LB film bilayers as opposed to the blank mylar strips. See Fig. 6. However, our measurements using aluminum foil indicate the need for caution when analyzing this data. The aluminum foil conductivity models

demonstrate the expected increase in conductivity; more aluminum, more conductivity. However, the blank mylar reference also increased in conductivity based on the amount of aluminum foil in the opposing cavity or the sample cell. See Fig. 7. Our reference mylar should have remained the same throughout due to its isolation in another cavity, yet the data shows a different curve for the blank mylar reference for each of the aluminum foil samples. The only way we can envision the two supposedly isolated cavities, as having an affect on each other is if our isolation techniques have failed. A source of noise must be coupled to the system. If future data support this hypothesis, we may have to reconsider the electrical scheme used in these measurements.

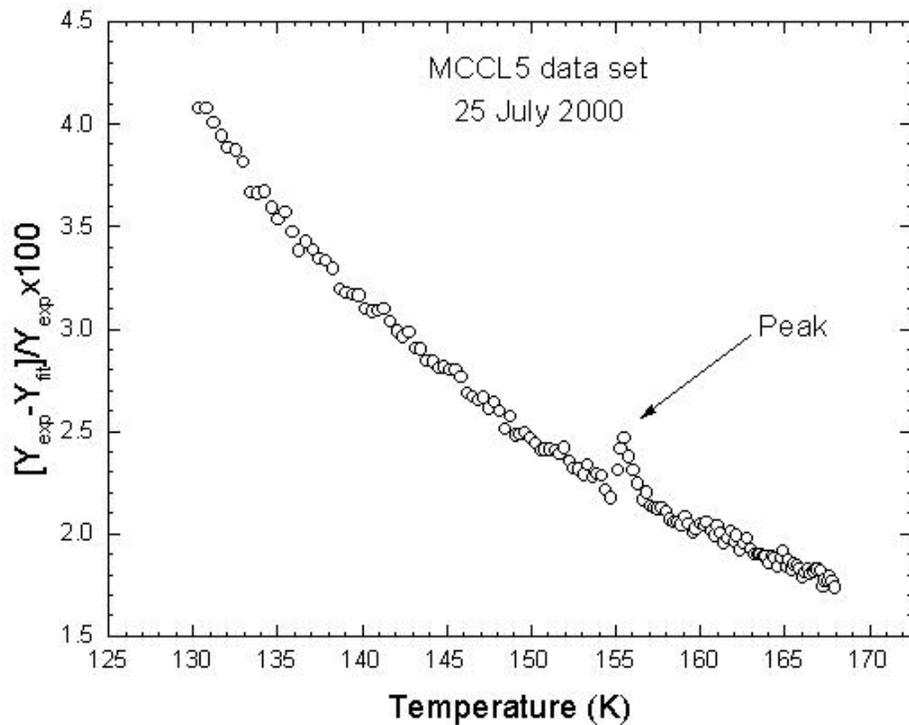


Fig. 5. Percent Difference in Capacitance from Fit vs. Temperature.

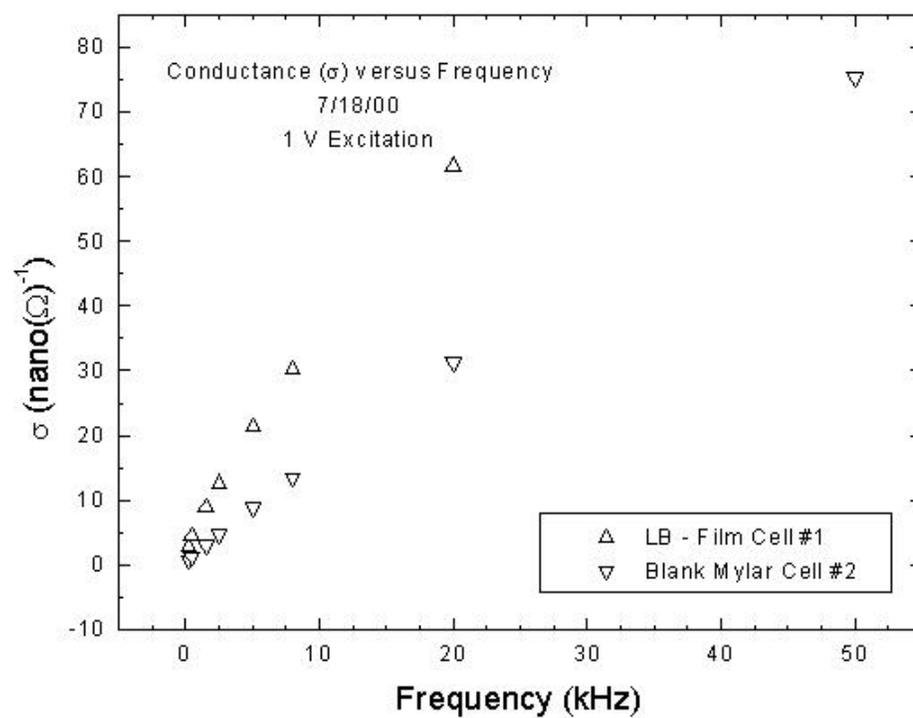


Fig.6. Conductance vs. Frequency.

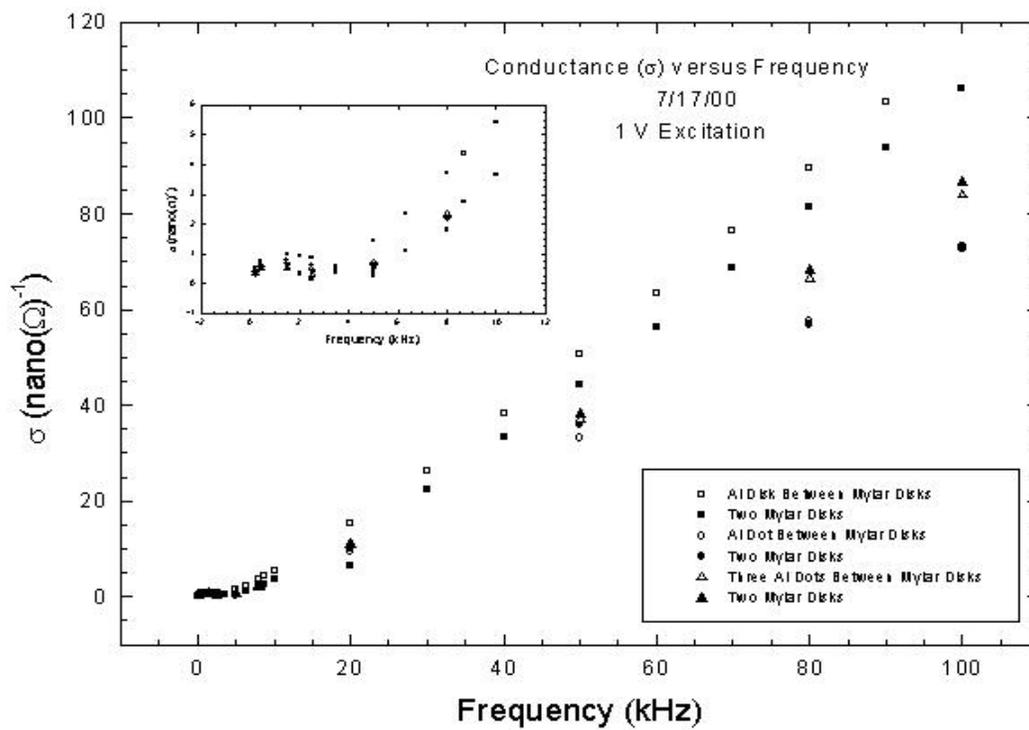


Fig. 7. Conductance vs. Frequency.

## REFERENCES

- [1] G.E. Granroth, B.C. Watson, M.W. Meisel, et al., unpublished (2000).
- [2] M.A. Petruska, *Organic/Inorganic Langmuir-Blodgett Films Based on Metal Phosphonates* (University of Florida dissertation, 2000).
- [3] D. Halliday, R. Resnick, and J. Walker, *Fundamentals of Physics Volume 2* (John Wiley & Sons, New York, 1993).
- [4] F.R. Leon and A.F. Hebard, *Low-Current Noise Measurement Techniques*, unpublished (University of Florida, 1999).
- [5] R.E. Simpson, *Introductory Electronics for Scientists and Engineers*, (Allyn and Bacon, Inc., Boston, 1974).
- [6] R.C. Richardson and E.N. Smith, *Experimental Techniques in Condensed Matter Physics at Low Temperatures* (Addison-Wesley, New York, 1988).

## ACKNOWLEDGEMENTS

We would like to sincerely thank the National Science Foundation and the National High Magnetic Field Laboratory for funding both summer research experience programs. We would also like to express our appreciation to Dr. M.W. Meisel for giving us the opportunity to collaborate with him this summer and having the time and patience to work with us. Many of the technical aspects of our project would not have been completed in time without the assistance of B.C. Watson, whose experience helped us considerably. For this we would like to thank him. Thanks to Ed Scorch for a wonderful job in producing our sample cell, and to Stephen Gerofsky for the production of our bath detector. We offer special thanks to Dr. Pat Dixon, Dr. Kevin Ingersent, and Dr. Alan Dorsey for doing such a remarkable job in making this summer as beneficial as possible.