EXPERIMENT GUIDE FOR SUPERCONDUCTOR DEMONSTRATIONS

WARNING: Liquid nitrogen and superconductor pellets may be dangerous if handled improperly. Do not handle liquid nitrogen or the contents of this kit without first reading and understanding the warnings and instructions contained in this manual. USE AT YOUR OWN RISK.

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INTRODUCTION

Welcome to the fascinating new world of ceramic superconductors. Colorado Superconductor's growing family of High Temperature Superconductor Kits emphasize both the underlying physics, and also the new applications of these versatile new materials. Furthermore, in an effort to keep abreast of the rapid advances in this field, we are pleased to provide several *Comparison* Kits that compare the properties of some of the leading high temperature superconductors available today.

These Kits are designed to introduce the new ceramic superconductors in a 'cookbook' fashion. This instruction manual has detailed explanations to help you implement the various experiments and to understand their physical basis. The materials in these Kits can be also be used to design and perform several additional experiments with the help of the references listed on page 44.

Colorado Superconductor is continuously introducing new Kits to demonstrate newly discovered physical phenomena and also new materials. Applications for these materials are being pursued at a rapid pace. Consequently, this manual is organized in five parts:

PART I	The fundamentals of superconductivity.
PART II	Safety and handling instructions.
PART III	Laboratory instructions for experiments.
PART IV	Appendixes.
PART V	References.

The following page lists the Demonstration Kits. In addition to the Demonstration Kits, Colorado Superconductor Inc. also makes various Kits for larger laboratory groups. If you are interested in these and other Kits please write to us or call us at the location listed on page 45 at the back of this manual.

The Kits are quite complete in that one can investigate the basic phenomena of superconductivity with very little in addition to the Kit contents themselves. The only item that the investigator needs to provide is the liquid nitrogen. For the Critical Temperature Kits, simple, inexpensive digital voltmeters are also needed. The K20 *Susceptibility* Kit, requires a function generator capable of producing a sine wave at a frequency of 1KHz, along with an AC and DC digital voltmeter, AC digital ampmeter, and an ohmmeter. The *Complete Exploration* and the *Super Exploration* Kits contain superconducting four point probe devices that can be used for the experiments described in this manual, or extended far beyond that by the interested experimenter. These Kits will require several different items of measurement apparatus depending on the particular experiment being performed.

While the various items (other than the superconductors on account of their chemical constituents) provided with the Kits pose no special safety concerns, the experimenter will need to use liquid nitrogen, which is not provided. Liquid nitrogen is widely used, but because it is extremely cold, it does possess the potential to cause harm to the user. Therefore, IT IS ESSENTIAL THAT YOU READ THE SECTION ON SAFETY ON PAGES 13 to 15 OF THIS INSTRUCTION MANUAL BEFORE PROCEEDING WITH ANY EXPERIMENTS. A SOURCE OF EMERGENCY MEDICAL INFORMATION FOR THE SUPERCONDUCTOR CHEMICALS IS LISTED ON PAGE 15.

ALL KITS MUST BE USED ONLY BY, OR UNDER THE SUPERVISION OF, ADEQUATELY TRAINED INDIVIDUALS.

Please also read the section on page 10 to ensure proper care for your superconductor sample. Read this manual carefully, and then get ready for an adventure.

THE KITS AND THEIR PURPOSE

Levitation Kit (Kit K1): Demonstrates the Meissner Effect and the low friction magnetic bearing.

Critical Temperature Kit (Kit K2): Demonstrates the determination of Critical Temperature, Meissner Effect, and the low friction magnetic bearing.

Complete Exploration Kit (Kit K5): Supports six experiments built around the electrical four point probe with its attached thermocouple. These include the measurement of Electrical Resistance as a function of Temperature, Critical Temperature, Critical Current, and Critical Magnetic Field. Also included are the Meissner Effect and low friction magnetic bearing experiments. A CSI Sand Cryostat is recommended for use with this kit.

Suspension & Levitation Kit (Kit K6): Demonstrates the phenomena of Flux Pinning through the suspension of a superconductor disk below a rare earth magnet. This Kit also demonstrates the Meissner Effect and the low friction magnetic bearing.

Levitation Comparison Kit (Kit K11): Compares the Meissner effect between American-invented $YBa_2Cu_3O_7$ and the Japanese $Bi_2Sr_2Ca_2Cu_3O_9$

Critical Temperature Comparison Kit (Kit K12): Compares the Critical Temperature and Meissner Effects between YBa₂Cu₃O₇ and Bi₂Sr₂Ca₂Cu₃O₉

Super Exploration Kit (Kit K15): Compares physical properties of YBa₂Cu₃O₇ and Bi₂Sr₂Ca₂Cu₃O₉ using the four point probe. A CSI Sand Cryostat is recommended for use with this kit.

Superconducting Energy Storage Kit - also called: Battery Kit - (Kit K18): This exciting Kit directly delves into one of the key application areas of the new superconductors. A toroidal superconductor is used to investigate the mechanics of electrical energy storage in superconductors. A disk superconductor is provided for comparison studies of the Meissner Effect.

Grand Compendium Kit (Kit K17): This kit contains one each of all elements of Kits **K1 through K18**. In one simple purchase, the investigator can study the Meissner Effect, Four Point Probe experiments, the Suspension Effect, and the Superconducting Energy Storage Device. Both $YBa_2Cu_3O_7$ and $Bi_2Sr_2Ca_2Cu_3O_9$ materials are included for the Meissner and Four Point Probe experiments for material property comparisons. A **CSI Sand Cryostat** is recommended for use with this kit.

Magnetic Susceptibility Kit (Kit K20): Developed in cooperation with a research team at Whittier College, this kit introduces a new way to measure critical temperature using a magnetic induction coil. A CSI Sand Cryostat is strongly recommended for better temperature control.

Laboratory Kits (Kits K3 and K4): Are similar to the Levitation and the Critical Temperature Kits. They are designed to provide six experimental stations for the laboratory class

While no such Kits are listed, we will be pleased to supply Kits containing $Bi_2Sr_2Ca_2Cu_3O_9$ devices alone.

LIMITED WARRANTY

Our process is optimized to produce superconductor disks that can produce a levitation of between 3 to 5 millimeters with the magnets we supply. The process is not optimized to produce disks that are necessarily cosmetically perfect. Our disks are rugged, but may have defects such as small cracks, pinholes, or dimples. Since these defects do not affect the ruggedness or performance of the superconductor manufactured by Colorado Superconductor, Incorporated is handled in strict accordance with the **Superconductor Handling Instructions** described in this booklet, and the superconductor fails within 180 days from the date of shipment, then Colorado Superconductor, Inc. will replace it free of charge. Please return the defective superconductor to us for a free replacement.

LECTURE PLAN

The material in this manual is laid out to guide you step by step through the fundamentals of high temperature superconductors. The booklet traces the history, physics, chemistry, and preparation technology of the new materials. Special emphasis has been placed on the safety aspects of the experiments.

While the manual itself is a broad introduction to superconductivity in ceramic superconductors, it is expected that a teacher of physics would choose to prepare students for the use of the Kit by means of a classroom lecture in the fundamentals.

We would suggest that a classroom lecture begin with an exposition of the physical properties of superconductors, and how they differ from normal conductors. The texts listed in the references on page 45, or any other introductory text in solid state physics will be a good source of basic information on the physics of superconductivity. Three other basic books on superconductivity and electricity & magnetism are listed in the references. The sections on page 7, and pages 16 through 44 of this manual should also be of considerable help. In addition, the American Physical Society Publication, *Physics Today*, reports current Investigations in this field.

The next step would be to trace the history of superconductivity. A brief on this subject appears on page 6. The history of the new superconductors is intricately entwined with their chemistry. We have attempted to introduce you to some of the salient points of this chemistry on page 8. The American Chemical Society publication, *Chemical & Engineering News, is* a good source of up to date information on ceramic superconductor chemistry.

At this point it would be appropriate to discuss the applications of superconductors, with an emphasis on how easy it is to attain the operating temperatures of the new superconductors. Towards the end of this booklet, "Where do we go from here?" tests these waters.

OVERHEAD PROJECTOR USE

An incontrovertible demonstration of superconductivity to a lecture class would go a long way towards obtaining their interest and curiosity. We have found that a simple overhead projector, and the materials in this Kit can be used to project a fascinating visual image of the fundamental Meissner Effect on a projection screen. Before attempting the demonstration before a class, one should read about the Meissner Effect on pages 16 through 18 of this manual.

Place an overhead projector on its side in a way that it still illuminates a screen. Prepare a superconductor sample from this Kit for the Meissner Effect. Use a shallow dish which is no deeper than the thickness of the superconductor disk. Place that sample with its levitated magnet in front of the glass plate (where one normally places the transparencies), so that its silhouette is projected onto the screen. The magnet should be less than an inch away from the glass plate for best results. Adjust the focus setting of the projector to accommodate the distance of the levitated magnet from the glass plate. The Meissner Effect sample will probably have to be placed on several books or blocks to bring it level with the center of the glass plate of the overhead projector. A more impressive result can be obtained by stacking several pellets as described on page 16.

The use of a TV camera or camcorder in conjunction with a large screen TV would of course be an even better demonstration of the aforementioned experiment.

PART I

THE FUNDAMENTALS OF SUPERCONDUCTIVITY

THE HISTORY OF SUPERCONDUCTORS

Superconductivity was discovered by H. Kamerlingh-Onnes in Holland in 1911 as a result of his investigations leading to the liquefaction of helium gas. In Onnes' time superconductors were simple metals like mercury, lead, bismuth etc. These elements become superconductors only at the very low temperatures of liquid helium During the 75 years that followed, great strides were made in the understanding of how superconductors worked. Over that time, various alloys were found that were superconductors worked at temperatures much more than 23 Kelvin (see page 10 for an explanation of the Kelvin scale of temperature measurement). Thus, liquid helium remained the only convenient refrigerant that could be employed with these superconductors.

Then in 1986, researchers at an IBM laboratory in Switzerland discovered that ceramics from a class of materials called perovskites, were superconductors at a temperature of about 35 Kelvin. This event sparked great excitement in the world of physics, and earned the Swiss scientists a Nobel prize in 1987. As a result of this breakthrough, scientists began to examine the various perovskite materials very carefully. In February of 1987, a perovskite ceramic material was found that was a superconductor at 90 Kelvin. This was very significant because now it became possible to use liquid nitrogen as the refrigerant. Since these materials superconduct at a significantly higher temperature, they are called High Temperature Superconductors, High T_c Superconductors, or simply: HTS materials.

There are several advantages in using liquid nitrogen instead of liquid helium. Firstly, the 77 Kelvin temperature of liquid nitrogen is far easier to attain and maintain than the chilly 4.2 Kelvin of liquid helium. Liquid nitrogen also has a much greater capacity to keep things cold than does liquid helium. Most importantly, nitrogen constitutes 78% of the air we breathe, and thus unlike liquid helium, for which there are only a few limited sources, it is relatively much cheaper.

The interest in the new superconductors continues to mount. Many Governments, Corporations and Universities are investing large sums of money in this to investigate this major breakthrough that many have hailed as important as the invention of the transistor.



THE LANGUAGE OF SUPERCONDUCTOR PHYSICS

The theoretical understanding of the phenomena of superconductivity is extremely involved. It is far beyond the scope of this manual to attempt to delve into that subject. However, in this short section, we have emphasized some of the fundamental terms and phenomena that will make it possible for you to conduct the experiments suggested in these Kits. A more detailed description of the physics of superconductors can be obtained from any of the several references in listed on page 45.

Superconductors have the ability to carry an electrical current without loss of energy. Unlike normal conductors of electricity in which the current is carried by individual electrons, in superconductors the current is carried by pairs of electrons called <u>Cooper Pairs</u>, in honor of one of the formulators of the famous 'BCS' theory of superconductivity. When the electrons move through a solid in Cooper Pairs, they are impervious to the energy absorbing interactions that normal electrons suffer. At this point, there is no resistance to the flow of electric current. To form Cooper Pairs, a superconductors made from different materials have different values of T_c. For the new ceramic superconductors in these Kits, T_c is about 90 Kelvin (See page 10 for an explanation of the Kelvin scale) for YBa₂Cu₃O₇, and for Bi₂Sr₂Ca_{n-1}Cu_nO₉, 80 Kelvin and 110 Kelvin, for n=2 or 3. The *Critical Temperature Kits*, and the *Complete Exploration*, *Super Exploration*, and Magnetic Susceptibility Kits are designed to allow you to measure T_c in several simple and elegant ways.

It is not yet clear that these ceramic superconductors indeed do conduct electricity by means of Cooper Pairs as described by the 'BCS' theory. In fact, another theory called the 'Resonant Valence Bond' theory has been advanced as being more effective. This theory may explain the gradual onset of superconductivity at a temperature around T_c in the ceramic materials.

Since there is no loss in electrical energy when superconductors carry an electrical current, relatively narrow wires made of superconducting material can be used to carry huge currents. However, there is a certain maximum current that these materials can be made to carry, above which they stop being superconductors. This maximum current flux is referred to as the <u>Critical Current Density</u>, or J_c. There has been a great deal of effort to increase the value of J_c in the new ceramic superconductors. For routine electrical measurements on the samples provided in these Kits, you must remember to use electrical currents that result in current densities that are smaller than J_c.

It has long been known that an electrical current in a wire creates a magnetic field around the wire. The strength of the magnetic field increases as the current in the wire is increased. Thus, on account of their ability to carry large electrical currents without loss of energy, superconductors are especially suited for making powerful electromagnets. Furthermore, if the electrical current travels only through a superconductor without having to pass through a normal conductor, then it will persist forever resulting in the formation of a powerful permanent electromagnet (see the *Superconducting Energy Storage - 'Battery' - Kit*). These permanent currents in a superconductor are referred to as persistent currents. The magnetic field generated by the superconductor in turn however, affects the ability of the superconductor to carry electrical currents. In fact, as the magnetic field increases, the values of both T_c and J_c decrease. When the magnetic field is greater than a certain amount, the superconductor is guenched, and can carry no superconducting current. This maximum magnetic field is called the maximum $\underline{Critical Field}$, or H_c . Again, this is a large field, and even the powerful rare earth alloy magnets we will be using in our experiments will not significantly affect our superconductors. The *Complete Exploration* and *Super Exploration Kits* can be used to determine both H_c and J_c .

The experiments in these Kits delve into some of the basic physics of superconductivity. These phenomena are explained in greater detail in the experimental sections of this manual.

THE CHEMISTRY OF CERAMIC SUPERCONDUCTORS

This section will describe some of the chemistry of the superconductors in these Kits. This discussion will also be useful in the next section which details how the superconductors are made.

The new ceramic superconductors are a class of materials collectively called perovskites. <u>Perovskites</u> are metal oxides which exhibit a stoichiometric ratio of 3 oxygen atoms for every 2 metal atoms. Perovskites are typically mixtures of several different metals. For example, in the YBa₂Cu₃O₇ superconductor in all of the Kits, the metals are yttrium (Y), barium (Ba), and copper (Cu). Using the standard valence values for these metallic elements, one would expect the chemical formula YBa₂Cu₃O₉. Surprisingly, scientists have found that this superconductor has about 2 oxygen atoms less than predicted, and instead has the approximate formula YBa₂Cu₃O₇, this is also sometimes written as YBa₂Cu₃O_{7- δ}.

One should note that the proportions of the 3 different metals in the YBa₂Cu₃O₇ superconductor are in the mole ratio of 1 to 2 to 3 for yttrium to barium to copper respectively. Thus, this particular superconductor is often referred to as the <u>1-2-3 superconductor</u>. For the bismuth-based superconductor, one chemical formula is Bi₂Sr₂Ca_{n-1}Cu_nO₉ and it is often referred to as the <u>2-2-(n-1)-n</u> material, for n=1 2 or 3. In practice two stoichiometric compositions, the n=2 and n=3, of the Bismuth-based material exist in most simply prepared samples. The n=3 (T_c corresponding to 110K) composition, or crystalline phase, dominates the samples that are prepared by Colorado Superconductor.

The 1-2-3 ratio in YBa₂Cu₃O₇ is also an indication of the simple ratio required of the elements in the constituent original chemical precursors to make this superconductor. So, for example for making YBa₂Cu₃O₇, three separate chemical compounds containing yttrium, barium, and copper respectively are mixed in proportions such that the three metals are in the molar ratio of 1-2-3. The resulting mixture is then heated and cooled several times in a kiln or electrical furnace, usually in the presence of oxygen. The amount of oxygen in the 1-2-3 compound can vary depending on the way it was made. If the sample is short on oxygen, it will be green, and will not be a superconductor. If on the other hand it has the right amount of oxygen, it will be black. This black material is a superconductor. This is the reason that in most cases the molar amount on the oxygen atom is expressed as 7- δ . Bi₂Sr₂Ca_{n-1}Cu_nO₉ can also be made in a very similar manner.

Scientists are discovering that many other metals may be substituted for the ones in our example. In fact in 1988, scientists in Arizona found a compound of thallium, barium, calcium, and copper that is perhaps an even better superconductor than $Bi_2Sr_2Ca_{n-1}Cu_nO_9$. Unfortunately, the element thallium is very toxic, and cannot be supplied for classroom work. Since then, compounds substituting lead or vanadium for copper have been discovered. The most recent versions of the CRC handbook contain tables of the various high T_c superconductors.

The perovskites are ceramics, and thus share many properties with other ceramics. One of these properties is their brittleness. This has particularly bedeviled technologists because it makes it very difficult to make, for example, the flexible wires that are needed for many practical applications.

HOW ARE THESE SUPERCONDUCTORS MADE?

The previous section detailed some of the chemistry of the new ceramic superconductors. This section uses that knowledge to outline a process by which one can make superconductor disks similar to those in the Kits. This outline is not meant to suggest that the reader should attempt to make these superconductors without adequate supervision. There are various procedural and safety considerations that are not recorded here.

Since the new superconductors are ceramics, the technique for making them is quite similar to making other ceramics. Besides the precursor chemicals, all that is needed is a mortar and pestle, a die mold, and a well-ventilated kiln or furnace that can reach a temperature of about 1700 degrees Fahrenheit (975 degrees Celsius).

Using the fabrication of $YBa_2Cu_3O_7$ as an example, the first step is to measure out the correct proportions of the precursor source chemicals. These proportions were described in the previous section, **The Chemistry of Superconductors**, where the elements yttrium, barium, and copper were shown to be in the mode ratios of 1 to 2 to 3 respectively. Oxides, nitrates, or carbonates are excellent sources of these elements. Care must be exercised in selecting laboratory grade chemicals for their purity. The chosen source chemicals are carefully weighed out so that the three elements are in the 1:2:3 mole ratios as described. The three powdered chemicals are then carefully mixed and ground together using the mortar and pestle. The well-powdered mixture that forms is a deep gray in color.

The resultant mixture of chemicals is then be poured into the type of die mold which can be found in a machine shop. A pressure of about 10,000 pounds per square inch will produce a compressed disk from the powder. It is very important to design the mold carefully, otherwise when under pressure it could fail mechanically.

Next, the pressed disk is fired in an electric furnace or kiln. The kiln should be capable of attaining a temperature of about 1700°F, and must be well ventilated. In addition, the kiln should also have a clean shelf on which to place the pressed disk.

The pressed disk is baked in the kiln at 1700°F overnight. Next, the kiln is gradually cooled to near room temperature over the course of a day. When the kiln and disk are about at room temperature, the disk can be withdrawn. It is important to circulate fresh air through the kiln during the heating and cooling cycles. Best results are obtained if the samples are slowly cooled surrounded by an oxygen atmosphere. Also, several heating and cooling cycles appear to improve the quality of the 1-2-3 ceramic superconductors. If the ceramic disk is ground into a fine powder and then pressed into a disk again between each cooling and heating cycle, an even better sample results.

The resulting disk should be hard and black like the superconductor in the Kits. Any green color will indicate that the disk will be an insulator instead of a superconductor.

The bismuth-based, Bi₂Sr₂Ca_{n-1}Cu_nO₉ requires a lower baking temperature (this is believed to be due to the low melting point of Bi), but the duration can be as long as 5 to 10 days. The superconductive properties are found to improve the longer the material is baked. Furthermore, this superconductor has three distinct phases with different critical temperatures. To preferentially promote the phase with the higher critical temperature (110 Kelvin), a small amount of lead oxide may be introduced into the precursor mixture. The lead oxide acts as a catalyst to promote the n=3 crystalline phase.

TAKING CARE OF YOUR SUPERCONDUCTORS

The samples of superconductors provided with these Kits are quite rugged. However, the superconductors are ceramics and can be very brittle. It is therefore important that the samples not be dropped or hit any sharp blows. They could otherwise chip or shatter.

The superconductors in the kit are somewhat porous and also hygroscopic (they absorb water). Over long use, the absorbed water will cause the sample to become mechanically weak, and it will eventually crumble. In particular, water can be absorbed from the frost that forms when the sample warms after immersion in liquid nitrogen. After use, the superconductor disk must be carefully dried with a clean disposable wipe to remove the frost, and then held under a desk lamp or hair dryer for a few minutes to completely dry it. The sample should be stored in an airtight box with a drying agent like silica gel.

A substantial body of knowledge has been accumulated about the effect of moisture on $YBa_2Cu_3O_7$ superconductors. It is now known that the superconductivity diminishes with time. Researchers believe that this is due to an interaction with atmospheric water vapor and perhaps carbon dioxide. While we have found that our superconductors do not show any such effects over a 6 month observational period, it would be prudent for the experimenter to use and store them away from direct contact with sources of water vapor or carbon dioxide. Summarizing:

- Do not expose the superconductor disk to water.
- After immersion in nitrogen, carefully wipe the superconductor to remove frost or water. Then hold it about 6 inches away from a hair dryer for a few minutes to ensure that it is dry.
- Handle the superconductor gently. Do not drop, saw, pound, shape, or attempt to break the disk.
- Do not expose the superconductor to temperatures greater than 110°F for extended periods.
- Always store the superconductor in a box with some drying agent like silica gel. Store the box in a dry place.

4-point probes:

- With these probes do not pass more than the recommended 0.5 Amp of current through the wire leads, or else the contact points will burn out and damage the superconductor.
- Do not connect or disconnect the probe from the power supply when it is outputting more than 0 volts.
- Always make slow adjustments to the voltage and current running though the probes.
- Do not overstress the wires, they are only held in place by electrical connections to the superconductor.

THE KELVIN SCALE OF TEMPERATURE

Since it is inconvenient to use negative values when measuring low temperatures of either the Fahrenheit or the Celsius scales, in the nineteenth century, Lord Kelvin, an Englishman, invented a new temperature scale suitable for measuring low temperatures.

When a material is cooled, it looses energy in the form of heat, and its temperature decreases until a point is reached where it has no more energy left to loose. At this point it is not possible to lower the temperature any further. This low temperature is called <u>absolute zero</u>. Lord Kelvin suggested that this absolute zero temperature be the basis of a new scale which begins with the value zero at absolute zero. At sea level on this scale, water freezes at 273 Kelvin, and boils at 373 Kelvin. So, just like the Celsius scale, there is a difference of 100 degrees between the freezing and boiling points of water. This new scale is called the Kelvin scale in honor of Lord Kelvin, and has the unit "Kelvin". To convert from degrees Celsius to "Kelvin" use the formula:

Kelvin = degrees Celsius + 273.15

This scale is very convenient for recording the very low temperatures of liquid nitrogen and liquid helium. On this scale, nitrogen condenses to a liquid at 77 Kelvin, and helium, a chilly 4.2 Kelvin. $YBa_2Cu_3O_7$ has a Critical Temperature of about 90 Kelvin, and $Bi_2Sr_2Ca_2Cu_3O_9$ a Critical Temperature of 110 Kelvin. Thus when they are immersed in liquid nitrogen at 77 Kelvin, they become superconducting.

Additional notes of interest:

There is an absolute scale that uses the same degrees as those of the Fahrenheit scale. This scale is called the Rankine Scale.

Rankine = degrees Fahrenheit + 459.67

On this scale, nitrogen condenses at 138.6 Rankine, and helium at 7.56 Rankine. The conversions are fairly simple.

Fahrenheit = (9/5)*degrees Celsius + 32

While most everyone can easily understand why 0°C can be set as the freezing point of water, and 100°C the boiling point, the Fahrenheit scale is a little less obvious.

Daniel Fahrenheit determined the zero point of the Fahrenheit scale in the early 1700's. He did it by taking equal parts of ice and salt, and then set what he called 0°F as the lowest temperature the mixture would melt. He then determined the scale using what he considered as two other standard temperatures; the freezing point of water, and the temperature of a healthy human. Dividing up his scale into 12 equal parts initially (think of a ruler having 12 inches), then subdividing those parts 3 more times he arrived at 96°F as the temperature of a healthy human body and 32°F as the freezing point of water.

In this text, temperature is measured using the Kelvin scale.

THE MEASUREMENT OF TEMPERATURE

Temperature can be accurately measured with thermometers designed and calibrated for use in the temperature range of interest. For all experiments in this manual using Colorado Superconductor's family of superconductor kits, a range from room temperature to that of liquid nitrogen is of interest. Highly accurate thermometers typically do not operate over such a wide range. Thermocouple thermometers however are fairly accurate over this large temperature variance.

A thermocouple consists of a mechanical junction of two dissimilar metals. This junction generates a small electrical potential (voltage), the value of which depends upon the temperature of the junction. Thus with calibration, and an appropriate choice of metals, one can obtain a thermometer for the desired temperature range. For our range (300 Kelvin to 77 Kelvin), a type T, or Copper-Constantan thermocouple is used. A -0.16mV reading indicates room temperature (298K), and +6.43mV is 77K.

The thermocouple junction has been carefully attached to the superconductors in our kits, and thermally balanced and calibrated to be used with the table below at 70°F. A simple digital millivoltmeter attached to the leads can be used to determine the voltage of this junction. Note that thermocouple leads must be connected to the voltmeter via wires of the same material and the junction to the thermocouple leads must be at room temperature. This voltage can be converted to the equivalent temperature with the help of the conversion chart below.

٥K	0	1	2	3	4	5	6	7	8	9	10	٥K
60	7.60	7.53	7.46	7.40	7.33	7 26	7.19	7.12	7.05	6.99	6.92	60
70	6.92	6.85	6.78	6.71	6.64	6.56	6.49	6.42	6.37	6.33	6.29	70
80	6.29	6.25	6.21	6.17	6.13	6.09	6.05	6.01	5.97	5.93	5.90	80
90	5.90	5.86	5.83	5.79	5.75	5.72	5.68	5.64	5.60	5.56	5.52	90
100	5.52	5.48	5.44	5.41	5.37	5.34	5.30	5.27	5.23	5.20	5.16	100
110	5.16	5.13	5.09	5.06	5.02	4.99	4.95	4.91	4.88	4.84	4.81	110
120	4.81	4.77	4.74	4.70	4.67	4.63	4.60	4.56	4.53	4.49	4.46	120
130	4.46	4.42	4.39	4.35	4.32	4.28	4.25	4.21	4.18	4.14	4.11	130
140	4.11	4.07	4.04	4.00	3.97	3.93	3.90	3.86	3.83	3.79	3.76	140
150	3.76	3.73	3.69	3.66	3.63	3.60	3.56	3.53	3.50	3.47	3.43	150
400	0.40	0.40	0.07	0.04	0.00	0.07	0.04	0.04	0.40	0.45	0.40	400
160	3.43	3.40	3.37	3.34	3.30	3.27	3.24	3.21	3.18	3.15	3.12	160
170	3.12	3.09	3.06	3.03	3.00	2.97	2.94	2.91	2.88	2.85	2.82	170
100	2.82	2.79	2.70	2.73	2.70	2.07	2.04	2.01	2.58	2.53	2.52	100
190	2.52	2.49	2.40	2.43	2.40	2.37	2.34	2.31	2.29	2.20	2.23	190
200	2.23	2.20	2.17	2.14	2.11	2.00	2.05	2.02	1.99	1.90	1.95	200
210	1 93	1 90	1 87	1 84	1 81	1 78	1 75	1 72	1 69	1 66	1 64	210
220	1.64	1.61	1.59	1.56	1.54	1.51	1.49	1.46	1.44	1.41	1.39	220
230	1.39	1.36	1.34	1.31	1.29	1.26	1.24	1.21	1.19	1.16	1.14	230
240	1.14	1.11	1.09	1.07	1.04	1.02	0.99	0.97	0.94	0.92	0.89	240
250	0.89	0.87	0.84	0.82	0.79	0.77	0.74	0.72	0.69	0.67	0.65	250
260	0.65	0.62	0.60	0.58	0.55	0.53	0.50	0.48	0.45	0.42	0.40	260
270	0.40	0.38	0.36	0.34	0.32	0.30	0.28	0.26	0.24	0.22	0.20	270
280	0.20	0.18	0.16	0.14	0.12	0.10	0.08	0.06	0.04	0.02	0.00	280
290	0.00	-0.02	-0.04	-0.06	-0.08	-0.10	-0.12	-0.14	-0.16	-0.18	-0.20	290
300	-0.20	-0.22	-0.24	-0.26	-0.28	-0.30	-0.32	-0.34	-0.36	-0.38	-0.40	300

Conversion from mV to Kelvin

See the appendix for a more detailed explanation of how thermocouples operate, and how to use a reverence junction to make extremely accurate temperature measurements

PART II

SAFETY & HANDLING INSTRUCTIONS

GETTING STARTED SAFELY

This is an extremely important section that makes recommendations for handling liquid nitrogen and the superconductor disks. These safety recommendations are not exhaustive, and it is <u>strongly recommended</u> that all users of these kits perform the experiments under the supervision of a person trained in the relevant safety procedures.

This section deals with the things you must know before you begin any of the experiments described in this booklet. The section outlines suggestions for the safe handling and transport of liquid nitrogen.

Liquid nitrogen is not provided with the Kit. While liquid nitrogen is non-toxic, it is extremely cold, and needs great care in handling and use. It is widely available, so you should have no difficulty obtaining it. We do strongly recommend that you ask someone with experience with it to help you. IT IS VERY IMPORTANT THAT YOU READ AND UNDERSTAND THE SECTION ON PAGES 13, 14 & 15 ON THE HANDLING & DISPOSAL OF LIQUID NITROGEN BEFORE YOU START ANY OF THESE EXPERIMENTS. These safety instructions are recommendations, and should not be construed as license by the manufacturer of the kits to use liquid nitrogen without adequate supervision.

Liquid nitrogen; Sources and Handling

Cooling air down to a temperature of 77 Kelvin makes liquid nitrogen. The nitrogen in the air condenses to form a colorless liquid. It is quite easy to produce, and it is also widely used, so that near any fair-sized city, there will be an outfit either making or selling liquid nitrogen. Factories, foundries, universities, hospitals, doctor's offices, welding shops, plumber's supply stores, animal breeding, etc, use liquid nitrogen. In fact, frozen foods at the supermarket are often frozen at the food processor's establishment by immersion in liquid nitrogen. It is transported to all of these places in specially insulated tanks that can readily keep the nitrogen cold for over a month at a time without additional refrigeration. Liquid nitrogen typically costs about \$0.80 a liter (about 3.8 liters = 1 gallon) when purchased in large quantities. When purchased in smaller quantities, the price can be expected to be somewhat more.

Small quantities of liquid nitrogen can be stored in Dewar bottles. Dewar bottles or flasks are hollowwalled glass vessels that are wonderfully insulating. An intact and clean Dewar bottle when properly handled, will keep liquid nitrogen cold for long periods of time.

We do not recommend it, however some people have found that a *Thermos* Dewar bottle with a pyrex or stainless steel shell, the kind that is used to keep coffee hot and tea cold, is adequate for transporting and storing small quantities of liquid nitrogen. These *Thermos* bottles may be purchased from a discount store for as little as \$5. The *Thermos* bottle may keep the nitrogen cold for over 24 hours at a time.

Thermos bottles however, often have screw-on plastic caps. THIS CAP MUST NOT BE SCREWED ON AT ANY TIME WHEN THERE IS LIQUID NITROGEN IN THE DEWAR BOTTLE, OR ELSE, AS THE NITROGEN GRADUALLY BOILS OFF, PRESSURE WILL BUILD INSIDE THE BOTTLE UNTIL IT EXPLODES. THIS CAN BE VERY DANGEROUS. Store-bought *Thermos* bottles come enclosed in plastic or metal cases. If this is so, then any nitrogen spilled on it could cause it to crack. For all of these safety concerns, we strongly recommend using instead, a laboratory-type Dewar bottle with a lid that cannot be screwed on. Furthermore, laboratory Dewar bottles usually come in a metal jacket which is not easy to damage. We do not recommend usage of any non-certified container for use with liquid nitrogen.

As the liquid nitrogen is first poured into the warm Dewar bottle, it will boil furiously. At this point one must be careful to keep the mouth of the bottle pointed away from people to prevent being splashed by the liquid. ONE MUST WEAR SAFETY GLASSES AT ALL TIMES WHILE OBTAINING OR USING LIQUID NITROGEN. One should not obtain more than one liter (about a quart) of liquid nitrogen at a time. When the Dewar bottle is about two-thirds full, the lid should be placed in the mouth of the bottle loosely, so that any liquid that boils off can escape without building up a pressure. We also recommend safety gloves while handling liquid nitrogen or its container.

The **CSI Sand Cryostat** is also an effective device for cooling and controlling the temperature of Four Point Probes and Magnetic Susceptibility Probes in Kits K5 (K5b for Bismuth), K15, K17 and K20. When using the **Sand Cryostat**, we recommend also using a thermocouple reference junction for more precise temperature measurements when necessary.

Some suggested practices for the safe use of the liquid nitrogen:

- Wear safety glasses at all times while in the presence of liquid nitrogen.
- Wear insulating gloves at all times when handling liquid nitrogen.
- Do not allow any liquid nitrogen to touch your body.
- Do not touch anything that has been immersed in liquid nitrogen until that item warms up to room temperature. Use the provided tweezers to remove and place items in liquid nitrogen.
- Do not move liquid nitrogen containers in a manner that causes splashing.
- Be extremely careful not to overfill or spill the liquid from any vessel.
- Never obtain more liquid nitrogen than you need immediately. We suggest that you never have more than about 1 liter on hand.
- The gas while non-toxic can asphyxiate through the displacement of oxygen in the air in an enclosed space. Thus use it only in very well ventilated areas.
- Do not store the liquid nitrogen in a container with a tight-fitting lid.
- Do not pour the liquid nitrogen on any surface except those designed for it. Most surfaces will be damaged by the extreme cold.

Disposal of liquid nitrogen.

The best way to dispose of liquid nitrogen is to allow it to evaporate slowly in its open container. During this time, the container should be in a safe, well-ventilated place where it will not be disturbed. One can also dispose of liquid nitrogen by carefully pouring it out into the soft soil of, for example, an unused part of an outdoors flowerbed. There it will rapidly evaporate and return to the atmosphere.

Superconductor Handling Instructions, and the safe use of kit materials.

The kit contains one or more superconductor samples. These samples are made of oxides of various metals. Some of these constituent metal oxides are toxic. We recommend the following guidelines for handling the materials in the kit:

- Wear protective gloves.
- Wear an apron or lab coat while performing experiments.
- Wear safety glasses at all times.
- Never handle the superconductors with your hands. Use the provided tweezers.
- Wash your hands with soap and water after the experiment is completed.
- Do not under any circumstances ingest the materials.
- Do not leave the materials in an unsupervised situation.
- This is not a toy. Do not let children handle this kit.

Disposal of the superconductor disk.

Since the superconductor disk contains various metals, it should be disposed of at a toxic waste disposal site. Consult your school or university waste disposal expert for further information regarding the disposal of small quantities of the constituent metal oxides.

EMERGENCY MEDICAL INFORMATION

The superconductor disk is made either from the salts or oxides of yttrium, barium, and copper, or from those of bismuth, strontium, calcium, copper, and lead for the bismuth-based superconductor. The salts of these metals are toxic if ingested. If the disk is swallowed, or portions are inhaled or enter the eye, immediately contact a physician. Physicians note that our product is registered with the **Rocky Mountain Poison Index.** The Regional Poison Control Centers also have information regarding treatment for these materials and their constituents.

LABORATORY INSTRUCTIONS

THE MEISSNER EFFECT

One of the properties of superconductors most easy to demonstrate, and also the most dazzling, is the Meissner Effect. Superconductors are strongly diamagnetic. That is to say that they will repel a magnet. Imagine a 'perfect' conductor of electricity that simply has no resistance to the flow of an electric current. If a conductor of electricity is moved into a magnetic field, Faraday's Law of Induction would lead us to expect an induced electrical current in the conductor and its associated magnetic field which would oppose the applied field. The induced electrical current would not dissipate in a 'perfect' conductor was already in a magnetic field, and then that applied field would indicate that an electrical current and its associated magnetic field would appear in the conductor which would attempt to oppose the removal of the applied field. If we were to do an experiment in which we placed a magnet on top of a material that by some process then became a 'perfect' conductor, we would see no physical effect on the magnet. However, were we to attempt to remove the magnet, only then would we feel an opposing force.

A superconductor is fundamentally different from our imaginary 'perfect' conductor. Contrary to popular belief, Faraday's Law of induction alone does not explain magnetic repulsion by a superconductor. At a temperature below its Critical Temperature, T_c, a superconductor will not allow any magnetic field to freely enter it. This is because microscopic magnetic dipoles are induced in the superconductor that oppose the applied field. This induced field then repels the source of the applied field, and will consequently repel the magnet associated with that field. This implies that if a magnet was placed on top of the superconductor when the superconductor was above its Critical Temperature, and then it was cooled down to below T_c, the superconductor would then exclude the magnetic field of the magnet. This can be seen guite clearly since Magnet itself is repelled, and thus is levitated above the superconductor. For this experiment to be successful, the force of repulsion must exceed the magnet's weight. This is indeed the case for the powerful rare earth magnets supplied with our kits. One must keep in mind that this phenomenon will occur only if the strength of the applied magnetic field does not exceed the value of the Critical Magnetic Field, H_c for that superconductor material. This magnetic repulsion phenomenon is called the Meissner Effect and is named after the person who first discovered it in 1933. It remains today as the most unique and dramatic demonstration of the phenomena of superconductivity.

On account of the polycrystalline nature of a typical ceramic superconductor, the Meissner Effect appears to be a bulk phenomenon. This can be demonstrated by stacking two or more superconductor disks. With the addition of each disk, the magnet will be levitated higher. This result is particularly advantageous if the Meissner Effect is being demonstrated to an audience with the help of an overhead projector as described on page 5.

Another interesting observation is that the levitated magnet does not easily slide off the superconductor. This seemingly stable equilibrium is actually a manifestation of <u>Flux Pinning</u>; a phenomenon uniquely associated with Type II superconductors, of which our high temperature ceramic superconductors are examples. Here lines of magnetic flux associated with a magnet can penetrate the bulk of the superconductor in the form of 'magnetic flux tubes'. These flux tubes are then 'pinned' to imperfections or impurities in the crystalline matrix of the superconductor thereby 'pinning' the magnet.

The procedure below will guide the experimenter through a demonstration of the Meissner Effect in a cookbook fashion, step by step. This procedure can also be used for the overhead projector-based classroom demonstration described on page 5.

A Pyrex petri dish, or a third of an inch high portion of the bottom of a Styrofoam coffee cup, can be used for holding liquid nitrogen for the experiment. To project a sharp image of the Meissner Effect with an overhead projector, use a very small dish so that the levitated magnet is less than an inch from the projector's glass plate.

Procedure

- 1. <u>ACTION</u>: Using the provided tweezers, carefully place the black superconductor disk carefully in a Pyrex dish, or in a appropriately shaped Styrofoam cup.
- <u>ACTION</u>: Carefully pour liquid nitrogen into the dish or Styrofoam cup until the liquid is about a quarter of an inch deep, and completely covers the superconductor disk; the top of the disk should be flush with the surface of the liquid nitrogen.
 - RESULT: The nitrogen boils around the disk. Wait until this boiling stops.
- <u>ACTION</u>: After ensuring that the disk is completely (and just) covered by the liquid nitrogen, use the tweezers to pick up the provided magnet, and attempt to balance it on top of the superconductor disk.
 - <u>RESULT</u>: Instead of settling down onto the surface of the superconductor, the magnet will simply 'float' a few millimeters above the superconductor.

This is a demonstration of the Meissner Effect.

Precautions

- 1. When pouring liquid nitrogen please be careful to prevent any splashing. Please read the section on handling and safety (pages 13 to 15), before beginning this experiment.
- 2. Conduct the experiments in a well-ventilated room.
- 3. Do not touch any items immersed in the liquid nitrogen with your hand until they have warmed to room temperature. Use the provided tweezers to add and remove items from the liquid nitrogen.

This experiment can also be conducted by placing the magnet on top of the superconductor before it is cooled in liquid nitrogen. As predicted by the Meissner Effect, the magnet will levitate when the temperature of the superconductor falls below its Critical Temperature. As explained earlier, there is no material other than a superconductor that could have shown this effect.

If you carefully set the magnet rotating, you will observe that the magnet continues to rotate for a long time. This is a crude demonstration of a <u>frictionless magnetic bearing</u> using the Meissner Effect. The rotational speed of a cube-shaped magnet can be increased by using a plastic drinking straw to blow a stream of air at one of the edges or corners of the cube. Another way to increase the magnet's rotational speed is to cut out a small rectangular hole in a piece of paper. The hole is positioned over the levitated magnet such that half of the magnet projects above the plane of the paper. A stream of air directed along the upper surface of the paper will cause the magnet to rotate rapidly.

The resistance of air slows the rotating cubical magnet. Consequently, it can be expected to stop after a while. A cylindrical magnet will rotate for much longer, since it is rotationally streamlined. However, the cubical magnet makes this demonstration much more graphic. A research group at Cornell University has demonstrated a frictionless superconducting bearing that can turn at a rate of one million rotations per minute. A bearing using the Meissner Effect is much more convenient and safe than a conventional magnetic bearing because of the 'self-centering' nature of the Meissner Effect on account of flux pinning.

All Kits from Colorado Superconductor, Inc. are equipped to demonstrate the Meissner Effect. The **Comparison Kits** contain both a yttrium-based ($YBa_2Cu_3O_7$) and a bismuth-based ($Bi_2Sr_2Ca_2Cu_3O_9$) superconductor. Both superconductors exhibit the Meissner Effect, however, if the disks are carefully removed from the liquid nitrogen bath while the magnet is still levitated, the bismuth-based material will continue levitating the magnet for a considerably longer time than the yttrium-based superconductor. This is because the bismuth-based superconductor has a significantly higher Critical Temperature than the yttrium-based one.

The **Critical Temperature Kit** and the **Critical Temperature Comparison Kit** both use the Meissner Effect to measure the Critical Temperature of superconductors.

Some questions

- 1. Why does the liquid nitrogen boil when you pour it into the dish? Why does it boil around the superconductor disk?
- 2. When the nitrogen has evaporated, the magnet stays levitated for a short while longer. Why is this so? Can you think of any other experiments using this fact?
- 3. If you push the levitated magnet with the tweezers so as to move it across the superconductor, it will resist movement. Why does this happen?
- 4. How can you improve the operation of the model frictionless bearing in your Kit?

There are many potential applications of the Meissner Effect, for example, magnetically levitated transport vehicles, frictionless bearings, low vibration mounts, etc. Can you think of other applications?



The Meissner Effect

MEASURING THE CRITICAL TEMPERATURE USING THE MEISSNER EFFECT

We have discussed the concept of Critical Temperature on page 7. There are several ways that it can be measured. One effective and elegant way is to use the Meissner Effect. The superconducting devices with attached thermocouple probes in both the *Critical Temperature Kit* and the *Critical Tem*

The superconductor and thermocouple device are encapsulated in a metal casing. We have designed this casing to impart greater thermal and mechanical stability to the device. The top of the device is the brass portion that shows a flat surface of the black superconductor disk. See figure 1 on the following page for details.

The procedure below will guide you through the measurement of the Critical Temperature of the superconductor step by step.

Procedure

- 1. <u>ACTION</u>: Carefully straighten the thermocouple leads and attach them to a voltmeter that can measure and display in the 0.01 milliVolt range.
- <u>ACTION</u>: Immerse the device completely in liquid nitrogen. Allow the boiling of the liquid to subside. The thermocouple should read about +6.43 milliVolts, corresponding to the liquid nitrogen temperature of 77K.
- 3. <u>ACTION</u>: Remove the device from the liquid nitrogen and place it flat on a non-conducting surface with the black superconductor exposed on the top surface.
- 4. <u>ACTION</u>: Carefully balance the small cubical magnet so that it 'floats' via the Meissner Effect over the center of the disk.
- <u>ACTION</u>: Keep the magnet under careful observation while recording the voltmeter reading at 5-second intervals. This part is best performed with the aid of a lab partner. You may have to center the magnet periodically with the tweezers.
 - <u>RESULT</u>: For several minutes the magnet stays levitated. During this time the voltmeter reading begins to show a gradual increase in temperature. After a while, the magnet begins to drop, and finally comes to rest on the surface of the superconductor. The temperature as measured by the voltmeter at the time when the magnet has just come to a complete rest on the surface of the superconducting device, is the Critical Temperature, T_c, of the superconductor.

One of the mysteries of these new superconductors is that they do not have sharply defined Critical Temperatures. Typically, the transition from normal to superconducting state takes place over a range of about 5 Kelvin. The 'Critical Temperature' that you measure falls in this range, with a reading of about 95 Kelvin for YBa₂Cu₃O₇, and about 110 Kelvin for Bi₂Sr₂Ca₂Cu₃O₉.

We suggest that you use clean alligator clips to attach the thermocouple leads to the voltmeter leads. These connection points should be kept dry and at room temperature. The thermocouple has been carefully attached and packed inside the metal device casing. Please do not attempt to open the casing, or else the thermocouple junction will no longer be in good thermal contact with the superconductor.

Precautions.

- 1. Be careful not to let the liquid nitrogen splash or spill when you pour it. Read the handling guidelines (page 14) before using liquid nitrogen.
- 2. Use the provided non-magnetic tweezers when handling the device or magnet.
- The electrical leads of the thermometer are delicate. Do not pull them, or twist or bend them unnecessarily. Bend the wires only before the device is cooled in the liquid nitrogen. Remember to keep the thermocouple-to-voltmeter lead connection at room temperature.

It appears that in ceramic superconductors, the Meissner Effect is a bulk phenomenon. Consequently, if any portion of the superconductor is below its Critical Temperature, the resultant Meissner Effect for that portion of the material will repel the magnet. The top surface of the superconductor disk warms first and looses its superconductivity as the liquid nitrogen evaporates. Other parts of the superconductor disk are still below the Critical Temperature, and thus continue to repel the magnet. However, since these parts are further from the magnet, it is levitated less. As the disk warms further, the magnet floats lower and lower, until the bottom of the disk is finally warmer than the Critical Temperature, at this point the magnet finally comes to rest on the top surface of the disk. Therefore, when the magnet comes to a complete rest on the surface of the superconductor, the bottom part of the disk, which is thermally attached to the thermocouple, is at the Critical Temperature.

Some Questions.

- Under some circumstances, the magnet will abruptly scoot to one side of the device as it warms. Can you think of an explanation for this?
- The device develops a layer of frost only after the liquid nitrogen has all boiled away. Why is this?
- Try the experiment by first placing the magnet on the superconducting device, and then cooling it down in liquid nitrogen. Do you observe any differences in the Critical Temperature? If so, why?
- The application of the Meissner Effect to measure the Critical Temperature was just one possible application of this effect. Can you think of other, elegant applications of this unique Effect?



Figure 1: The Superconducting Thermocouple Device

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THE FOUR POINT ELECTRICAL PROBE

The four point electrical probe is a very versatile device used widely in physics for the investigation of electrical phenomena. Colorado Superconductor Inc. has especially designed two four point superconducting devices from the $YBa_2Cu_3O_7$ and the $Bi_2Sr_2Ca_2Cu_3O_9$ materials for such investigations. The **Complete Exploration Kit** and the **Super Exploration** Kit contain four point electrical probes.

When a simple measurement of the electrical resistance of a test sample is performed by attaching two wires to it, one inadvertently also measures the resistance of the contact point of the wires to the sample. Typically the resistance of the point of contact (called contact resistance) is far smaller than the resistance of the sample, and can thus be ignored. However, when one is measuring a very small sample resistance, especially under variable temperature conditions, the contact resistance can dominate and completely obscure changes in the resistance of the sample itself. This is the situation that exists for superconductors.

The effects of contact resistance can be eliminated with the use of a four point probe. A schematic of a four point probe is shown in figure 2. In this diagram, four wires (or probes) have been attached to the test sample. A constant current is made to flow the length of the sample through probes labeled 1 and 4 in the figure. This can be done using a current source or a power supply as shown. Many power supplies have a current output readout built into them. If not, an ammeter in series with this circuit can be used to obtain the value of the current. A 5-Watt power supply capable of producing about ½ Amp is required for the experiments described for our superconducting devices.

If the sample has any resistance to the flow of electrical current, then there will be a drop of potential (or voltage) as the current flows along the sample, as for example between the two wires (or probes) labeled 2 and 3 in the figure. The voltage drop between probes 2 and 3 can be measured by a digital voltmeter. The resistance of the sample between probes 2 and 3 is the ratio of the voltage registering on the digital voltmeter to the value of the output current of the power supply. The high impedance of the digital voltmeter minimizes the current flow through the portion of the circuit comprising the voltmeter and probes 2 and 3, the resistance associated with only the superconductor between probes 2 and 3 is measured.



Figure 2: Schematic of Four Point Probe

The four point probe devices in the *Complete Exploration Kit* and the *Super Exploration Kit* are both encapsulated In rugged brass casings. On one side of the casing, the superconductor disk is visible. An aluminum end cap has been inserted into the backside of the brass casing to seal and to protect the probe connections with the superconductor. Please do not attempt to remove the end cap. A matched thermocouple has also been attached to the superconductor in this casing. This thermocouple is a type 'T', and has been described in detail on page 11 and in the appendix (page 42).

The $Bi_2Sr_2Ca_2Cu_3O_9$ superconductor four point electrical probe casing is larger than the YBa_2Cu_3O_7 casing. The former has BSCCO printed on the aluminum cap, and the latter with YBCO for further identification.

The illustration in figure 3 below shows the salient features of the four point probe devices. The pair of black wires are current leads for the input of current from the power supply, and have been labeled probes 1 and 4 in figure 2. The pair of yellow wires are the voltage measurement probes for measuring the voltage drop across the superconductor with the help of a digital voltmeter, and have been labeled probes 2 and 3 in figure 2. The red and blue wires are leads for the thermocouple.



Figure 3: The Superconducting Four Point Probe

Measuring Resistance versus Temperature and Critical Temperature

The measurement of electrical Resistance as a function of the superconductor's Temperature yields fundamental insights into its properties. The <u>Critical Temperature</u>, <u>Critical Current Density</u>, and the <u>Critical Magnetic Field</u>, can all be obtained through variations of this basic experiment.

This experiment requires the following pieces of equipment:

- A constant current source, or a power supply operating in the current limited mode. The output should not exceed 0.5 Amp. This is connected between the black current probes (probes 1 and 4). An ammeter placed in series with this circuit will measure the current. This current will be referred to as I₁₄.
- A digital voltmeter with a 0.01-millivolt resolution to measure the voltage drop across the yellow voltage probes (probes 2 and 3). This voltage will be referred to as V₂₃.
- 3. Use of a **CSI Sand Cryostat (see appendix, page 38)** is suggested for optimal results. Alternatively, a container of liquid nitrogen deep enough to completely immerse the four point probe device may be used.

The voltmeters should be connected as shown in figure 2. Alternatively, a strip chart recorder with a 10-milliVolt full-scale range and a resolution of 10-microvolt may be connected between probes 2 & 3. This will provide a continuous record of the voltage drop. If a two-channel recorder or x-y plotter is used, then the thermocouple reading can also be measured simultaneously. The output from the voltmeters connected to probes 2 & 3, and to the thermocouple, may be sent directly to a computer to store and further analyze the data. The following is a step-by-step guide for measuring the device's Resistance versus its Temperature:

Procedure.

- 1. <u>ACTION</u>: Set up the measurement equipment as described above, but do not as yet immerse the device (four point probe) in liquid nitrogen.
- <u>ACTION</u>: Insert the device into the CSI Sand Cryostat or other certified container and carefully fill it with liquid nitrogen. Ensure that the current (I₁₄) remains constant at less than 0.5 Amp.
 - RESULT: The nitrogen boils furiously. Wait until the boiling subsides.
- 3. <u>ACTION</u>: Record the voltage V₂₃, and across the thermocouple junction.
 - RESULT: V23 should equal zero. The thermocouple temperature reading should be 77 K.
- <u>ACTION</u>: If you are not using the CSI Sand Cryostat, remove the device from the liquid nitrogen. As the device warms, continuously monitor the value of V₂₃. Record the thermocouple temperature each time V₂₃ is recorded.
 - <u>RESULT</u>: Initially, V₂₃ remains constant even as the thermocouple temperature increases. Then the voltage between the probes (V₂₃) abruptly increases, the thermocouple reading corresponding to this jump in voltage is the <u>Critical Temperature</u>, or T_c of the superconductor. The ratio of the voltage between probes 2 & 3 (V₂₃) to current flowing between probes 1 & 4 (I₁₄) is the instantaneous resistance of the superconductor between probes 2 & 3. The probe voltage, and the thermocouple reading could be input directly into a computer or chart recorder for more accurate results. This latter approach also provides a permanent record of the data. This result is shown in figure 4 on page 24.

Precautions.

- 1. When pouring liquid nitrogen be careful to prevent any splashing. Read the section on safety & handling starting page 13 before beginning this experiment.
- 2. Be careful not to touch the device or wires when they are cold. Follow the safety directions.
- 3. No more than 0.5 Amp of current should pass through the device or wires at any time.
- If using a cryostat, slowly pour the sand out first, then remove the probe. Do not try to pull the probe out by the wires
- 5. Use a hair dryer to carefully dry the Four Point Probe device after use. Store it with a desiccant.
- 6. The probe and thermocouple wires are very brittle when cold. Please handle them with care.

Some Questions.

- 1. What effect would one expect if the Critical Temperature is measured with the device placed inside a functioning electromagnet?
- 2. Why is the transition in resistance gradual at the Critical Temperature?
- 3. A simple two-probe measurement of device resistance below its Critical Temperature exhibits a non-zero value. Why?

Determination of the Critical Temperature

The Critical Temperature, T_c is obtained during the measurement of the electrical Resistance as function of the Temperature of the superconductor on the previous page. The Critical Temperature of the Bi₂Sr₂Ca₂Cu₃O₉ superconductor is about 110 Kelvin versus about 92 Kelvin for the YBa₂Cu₃O₇ material. These results are shown below in figure 4.



Figure 4: Resistance versus Temperature

Determining the Critical Current Density

The four point probe device can be used to measure the Critical Current Density, J_c , of the superconductor materials in your kit. Theoretically, one could measure J_c of the probe immersed in liquid nitrogen, by boosting the applied current I_{14} until a transition to non-superconducting state occurs. Practically, this procedure would damage the probe permanently. The following procedure will help preserve the integrity of your superconducting four point probe device. This procedure also has the added advantage of obtaining Critical Current values at different operating temperatures.

For this experiment, a power supply capable of up to 0.5 Amp output is required. Connect the device to the digital voltmeters and power supply as explained on page 22 of this Instruction Manual (describing the measurement of the device's Critical Temperature, T_c). A constant current source that can be set to output a range of current values up to 0.5 Amp will make the execution of this experiment considerably easier. Proceed with the following directions:

- 1. <u>ACTION</u>: Set the current through probes 1 and 4 at 0.1 Amp, and measure the <u>Critical</u> <u>Temperature</u> as described on page 23 of the Instruction Manual. Record the measured T_c versus the value of current used.
- <u>ACTION</u>: Now increase the set current to 0.2 Amp, and repeat the process in action Item 1, above. Keep repeating the process with 0.1 Amp increments in current, taking care not to exceed a maximum of 0.5 Amp.
 - <u>RESULT</u>: Five data points will be obtained, each at a Critical Temperature, T_c, versus the set current, I₁₄. An appropriate extrapolation (curve fit) to 77 Kelvin will result in the Critical Current for the superconductor. The Critical Current Density, J_c, can then be estimated from the probe geometry listed in the table below. Figure 5, below shows an example of the result with a Bi₂Sr₂Ca₂Cu₃O₉ based four point probe.

This is a difficult experiment. The data is electrically `noisy'. Some improvement in the signal-to-noise ratio may be achieved by making several independent measurements at each current setting.

Material	Diameter	Thickness	probe 1&4 spacing	probe 2&3 spacing	probe depth
Yba ₂ Cu ₃ O ₇	24 mm	4 mm	17.5 mm	11 mm	Surface contact
Bi ₂ Sr ₂ Ca ₂ Cu ₃ O ₉	30 mm	5 mm	17.5 mm	11 mm	1.75 mm



Figure 5: The evaluation of Critical Current

Determining the Critical Magnetic Field

This experiment measures the Critical Magnetic Field, Hc_2 of a ceramic superconductor using the four point probe device. Equipment to measure Hc_1 , the lower Critical Field is beyond the scope of our approach.

For this experiment you will need an electromagnetic coil and associated power supply. The value of the field can be obtained using the geometry of the coil and a knowledge of the current flowing through it. The cavity in the middle of the coil needs to be large enough to accommodate the four point probe device and the liquid nitrogen container in which it is immersed. The four point probe device has been designed without any ferromagnetic parts to eliminate any potential interference.

Assemble the experiment as on page 25 in preparation of the measurement of Critical Current Density. However, this time place the four point device and its container of liquid nitrogen inside the cavity of the electromagnet. Gradually increase the current flowing through the electromagnet thus increasing the magnetic field strength through the superconductor. The value of V₂₃ will show an abrupt increase at some value of applied magnetic field strength. This value of magnetic field is the upper Critical Magnetic Field, H₂₂ for the superconductor sample at the temperature of liquid nitrogen, 77 Kelvin.

The value of the Critical Field, Hc_2 can be obtained at other temperatures by either placing the device in a cryostat while performing this experiment, or by removing the device from the liquid nitrogen container and monitoring the output of the thermocouple thermometer while measuring Hc_2 .

Another interesting experiment is the measurement of the Critical Temperature at different applied magnetic field strengths. The result of such an experiment for $YBa_2Cu_3O_7$ is shown schematically in figure 6. The value of H_{C_2} has been extrapolated to 0 Kelvin. It is very instructive to perform a subset of this experiment using the square neodymium magnet provided with your kit instead of an electromagnet. As the magnet is brought close to the surface of the superconductor device (which has been prepared as on page 25), the value of V_{23} will slowly increase for a given value of I_{14} . This phenomenon could potentially be used to construct a superconductor-based magnetic field detector.





THE SUPERCONDUCTING SUSPENSION EFFECT

The levitation of a magnet above a superconductor, the Meissner Effect, is well known. In October 1988, Huang and Peters of Lockheed and NASA respectively announced a startling and almost accidental discovery that they had made while investigating high temperature ceramic superconductors. This suspension phenomenon can be demonstrated with the help of the *Suspension & Levitation Kit* from Colorado Superconductor Inc.

The Kit contains a special superconductor disk which we shall call the Enhanced Flux Pinning (EFP) disk. A large, powerful neodymium rare earth magnet has been provided to suspend the EFP disk. A parallel set of materials has also been provided with the Kit to demonstrate the Meissner Effect described on page 16, for comparison. The experimenter requires only a source of liquid nitrogen for this experiment.

Procedure

- 1. <u>ACTION</u>: Completely immerse the EFP disk in a flat dish containing liquid nitrogen.
 - <u>RESULT</u>: The liquid nitrogen boils around the EFP disk. When the boiling subsides, proceed.
- 2. <u>ACTION</u>: Examine the large neodymium magnet, and find the face through which the magnetic axis passes (the magnet has the strongest attraction on this face).
- <u>ACTION</u>: Holding the magnet with the provided non-magnetic tweezers such that the magnetic axis is vertical, slowly lower the magnet so that it just touches the top of the immersed EFP disk.
 - <u>RESULT</u>: As the magnet approaches the EFP disk, there will be a momentary resistance to its continued downward motion. This will cease when the magnet is in contact with the EFP disk. This resistance is a manifestation of the well-known Meissner Effect.
- 4. ACTION: Gradually withdraw the magnet upwards out of the liquid nitrogen.
 - <u>RESULT</u>: The EFP disk should follow the magnet as it moves upwards and out of the liquid nitrogen. Observe that there is a gap between the EFP disk and the magnet. This is a gap that one would not observe in normal magnetic attraction.
 - <u>RESULT</u>: As the EFP disk warms, it will lose its superconductivity, and can no longer be suspended under the magnet. It will drop.

This was a demonstration of the <u>Suspension Effect</u> in the new high temperature ceramic superconductors. Figure 8 on the following page illustrates the salient features of this demonstration. A picture of the Suspension Effect is also shown on the cover of this Instruction Manual.

Precautions

- 1. The EFP disk is particularly sensitive to moisture. Please dry the disk immediately after use as described on page 10.
- The large neodymium rare earth magnet is very brittle. This powerful magnet will abruptly adhere to ferromagnetic materials, or to the small neodymium magnet provided with kit. This impact can easily chip or shatter the magnet(s).
- 3. The EFP disk may shatter if it falls on a hard surface after the Suspension Effect ceases. Please take care to suspend the disk over a soft surface, or over the liquid nitrogen bath.
- 4. The EFP disk is very cold. Do not attempt to handle it when it has been withdrawn out of the liquid nitrogen bath. Also, do not attempt to catch it in your hand when it falls when suspension ceases.

Some questions

- 1. Can you think of any way that the Suspension Effect can be prolonged?
- 2. Why do you think that the Suspension Effect was not observed with the low temperature superconductors of yesteryear?
- 3. What are some potential applications of the Suspension Effect?

The EFP disk was fabricated using a different temperature cycle than that used for making the normal superconductor disks. The EFP disk can also be made by doping (alloying) the regular superconductor disk with a small amount of silver metal.

The Meissner Effect cannot be used to explain the Suspension Effect. Several alternative explanations have been advanced to explain this phenomenon. One such explanation invokes the pinning of magnetic flux lines by the superconductor (hence our name, the Enhanced Flux Pinning disk). The magnetic field flux lines are pinned to impurities or imperfections within the superconductor, and they thus prevent the superconductor from moving in any direction relative to the flux lines.



Figure 8: Schematic showing the Suspension Effect

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THE SUPERCONDUCTING ENERGY STORAGE ('BATTERY') RING

Various properties of superconductors were described on page 7, the 'Language of Superconductor Physics', and then later explored in the preceding sections of PART III, the laboratory experiments. One of the most basic applications of the fundamental properties of a superconductor is exploiting its complete absence of resistance to an electrical current flow. This property has been used in a superconducting energy storage ring designed by the U.S. Navy in the SMES (Superconducting Magnetic Energy Storage) project, and also in potential applications by electric power utilities for base load power storage for commercial electric power generation.

The Superconducting Energy Storage (Battery) Kit from Colorado Superconductor demonstrates the fundamentals of energy storage in superconducting rings.

The basis of this Kit is a toroidal ring made from a high temperature superconductor. A current can be induced in the toroid, and because of its superconducting nature, the current can potentially persist for an extremely long time. A powerful cylindrical magnet is provided with the kit for induction of a persistent current. A large magnetic compass is provided as a detector of persistent currents. The compass is in four parts, but can be reassembled following the directions provided with it.

The experiment described in the following pages can be performed with no additional equipment except for liquid nitrogen and a suitably deep container for immersing the toroid. Should one be available, a Hall-probe Gaussmeter can substantially enhance the quality of the results of this experiment. Please see page 31 for a listing of some suggested Gaussmeters.

- 1. <u>ACTION</u>: Place the toroidal ring in a 1½ inch high section of the bottom of a Styrofoam coffee cup, or a Pyrex petri dish. The cavity should be deep enough that the toroid can be completely immersed even when it is standing on its edge.
- 2. <u>ACTION</u>: With the toroid flat at the bottom of the container, place the pole of the cylindrical magnet in close proximity, above or below the hole of the toroidal ring.
- 3. ACTION: Immerse the superconducting toroid in a bath of liquid nitrogen.
 - <u>**RESULT</u>**: The liquid nitrogen boils furiously. Wait until the boiling subsides.</u>
- 4. <u>ACTION</u>: Carefully move the magnet, crossing any one side of the toroid. Either the magnet or toroid may be moved relative to each other. Take care not to withdraw the toroid for more than a few seconds out of the liquid nitrogen bath while performing this a action. Remove and store the magnet in a far corner of the room. Several other techniques for moving the magnet relative to the toroid have been found to work. We encourage you to experiment.
- 5. <u>ACTION</u>: Using the provided non-magnetic tweezers, carefully turn the toroid on edge so that the axis of the toroid is horizontal. Orient the liquid nitrogen container so that the axis of the toroid is in the earth's magnetic east-west direction. Again take care that no part of the toroid emerges above the surface of the liquid nitrogen.
- 6. <u>ACTION</u>: After establishing an identification of the faces of the toroid, carefully bring a needle tip of the provided compass close to the axis of the toroid. The liquid nitrogen container with the toroid may need to be elevated to facilitate this action. Also, great care must be exercised that there are no magnetic or ferromagnetic objects near the compass or toroid. The liquid nitrogen container walls will be in between the toroid and the compass, but this should pose no problems since the walls are thin, and are made from a non-magnetic material.

RESULT: The compass needle will be either repelled or attracted by the toroid.

- 7. ACTION: Present the other pole of the compass needle to the same face of the toroid.
 - <u>RESULT</u>: This time the repulsion/attraction action of the previous result will be reversed. If the Meissner Effect or the Suspension Effect were responsible for the repulsion or attraction of the compass needle, this reversal would not have been observed.

The amount of deflection of the compass needle can be used to obtain a rough measurement of the current that has been induced in the toroid. The provided scale with compass will allow one to obtain the angle of the deflection. The deflection will have to be corrected for the Meissner Effect by successively measuring the deflection for either the two different poles of the compass needle, or the deflection caused by the two faces of the toroid for the same pole of the compass needle. Please see figure 9 below for the positioning of the experimental components. Figure 10 shows the different variables and the configuration of the compass needle and toroid.



Figure 9: Schematic showing toroid and compass position



Figure 10: Illustration of the different variables

Estimation of the value of the Induced current

The induced superconducting current represents a stored electric current, and can be shown to persist for extremely long periods of time as long as the toroidal ring is kept at liquid nitrogen temperatures (page 32). If this experiment were to be scaled up in size, the stored electrical energy could potentially be used at any later time. This is the principle of the U. S. Navy's SMES experiment.

This semi-quantitative measurement of the persistent current in the toroid requires an estimation of the deflection of the needle (shown as the angle ϕ in figure 10 on the previous page), and the distance of the needle tip from the center of the toroid (distance x in figure 10). The deflection of the compass needle at distance 'x' from the toroid is the result of a combination of forces exerted by the horizontal component of earth's magnetic field (B_{earth}) and the magnetic field due to the toroid (B_{toroid}). The relation between these quantities is simply:

$B_{toroid} = B_{earth} tan(\emptyset)$

The horizontal component of the earth's magnetic field is about 2 X 10^{-4} Tesla (2 Gauss). This value varies slightly with one's position on the earth. From this estimation of the magnetic field of the toroid, the Biot-Savart Law or Ampere's can be used to calculate the current induced (I_{toroid}) extant in the toroid.

$$I_{toroid} = (2B_{toroid}(x^2 + r^2)^{3/2})/\mu_0 r^2$$

Here 'r' is the average radius of the toroid and is approximately 0.0095 meters (see figure 10), and μ_0 is the permeability of free space and is equal to $4\pi \times 10^{-7}$ Newton/Amp².

r = r_{average} = (Outer diameter + Inner diameter)/4

All units in this form of the Biot-Savart Law are SI (Standard International) units. Distances x and r are in meters, magnetic field strength B is in Tesla, and Current I is in Amperes.

The amount of current in the toroid (I_{toroid}), can be increased by increasing the strength of the magnet used to induce the current. However, the magnet that has been provided is already one of the most powerful commercially available permanent magnets. Several passes in the same direction with a magnet should also work. Use of an electromagnet for inducing the current in the toroid may offer the best solution.

Instead of using a compass to determine the magnetic field of the toroid, a Hall-probe Gaussmeter can be used for a much more accurate measurement. This approach has the added advantage that the toroidal ring superconductor can remain in a horizontal position throughout the experiment. An *F.W. Bell Co. Model 120 Gaussmeter* is an excellent instrument for this purpose. A less expensive alternative is a battery operated digital Gaussmeter from *Applied Magnetics Laboratory, Model GM1A*.

Estimation of the lifetime of the persistent current.

The existence of a <u>persistent current</u> in the toroidal ring has already been amply demonstrated by our compass needle deflection experiment on page 29. However, it would be instructive to actually attempt to measure the decay of the electrical current as a function of time.

The initial expectation is that there should simply be no decay of the persistent electrical current over time since, after all, the toroid is a superconductor, and exhibits no electrical resistance, and hence no energy loss. Practically, however, on account of the phenomena of <u>flux creep</u> and <u>flux flow</u>, one will see a very small exponential decay of the stored electrical current. It would be very instructive to investigate this decay because it provides an insight into the effective 'resistance' of the superconducting toroid.

The simplest approach is to first induce an electrical current in the toroid as per the procedure described on page 29. It is, however, recommended that you should use a Gaussmeter for this experiment on account of the precise measurements that are required. The toroid with its induced persistent current will then have to be stored at liquid nitrogen temperatures over a period of several weeks. The current in the toroid is measured using the method on page 31 at periodic intervals over this time. Care will need to be exercised to prevent heating any portion of the toroid during the duration of the investigation.

If the results of measurement of the electrical current in the toroid were plotted versus the time intervals over which these measurements were made, it will be seen that the current decays exponentially as a function of time as described the following relationship:

$e^{-(R/L)t} = F$

Where R is the electrical resistance of toroid, L is the inductance of toroid, and F is the fraction of the remaining electrical current at time t. Our own measurements indicate that for the toroidal ring in the Kit, the value of L is about 5 nH (nano Henry). Using this number we have obtained a value of the toroid ring electrical resistance equal to $10^{-15} \Omega$.

Working backwards, using the measured value of R above, the time required for the current to decrease to 50% of its original value is:

t_{I =0.5} ≈10²³ years

This indicates that for most practical purposes, the current is permanently stored in the toroidal ring superconductor.

The experiments and descriptions above have drawn from experimental procedures described by Dr. Donald L. Shirer of the Physics Department at Yale University and Dr. Peter Heller and his colleagues at Brandeis University. An excellent treatment of these experiments can be found in the article entitled *Nitrogen Temperature Storage Ring Experiment*' by F. Liu, R. R. Tucker and P. Heller appearing in the American Journal of Physics, March 1990.

Precautions

- 1. The toroid has a large surface area, and is thus sensitive to moisture. Please dry the disk immediately after use as described on page 10.
- The large neodymium rare earth magnet is very brittle. This powerful magnet will abruptly attach itself to ferromagnetic materials, or to the small neodymium magnet provided with kit. This impact can easily chip or shatter the magnet.
- The toroid may shatter if it falls on a hard surface if it Is removed from the liquid nitrogen bath to bring it near the compass. We do not recommend this approach.
- Exercise great caution in moving the toroid in the nitrogen bath. Use the provided non-magnetic tweezers. Do not lift the liquid nitrogen container holding the toroid to position it since the nitrogen can spill this way.
- 5. This experiment is very sensitive to the magnetic field emanating from a stray magnet or even the interactions of a magnet being used in this experiment with any ferromagnetic objects in the vicinity.

Some questions

- 1. What happens when you remove the toroid from the liquid nitrogen bath and repeat the attempt to deflect the compass needle?
- 2. Is one application of this effect a powerful permanent magnet? If so, what are its advantages and limitations over existing permanent magnets?
- 3. If you repeat this experiment but do not use a magnet to induce an electrical current in the toroid, what are the compass needle deflection results that you would expect to see?
- 4. Could you use this phenomenon to estimate the Critical Temperature? If so, how?
- Can you design a system for withdrawing the electrical energy out of our storage ring toroid for use?
- 6. What are some other potential applications of the persistent electrical current in a toroidal coil?

DETERMINING THE CRITICAL TEMPERATURE WITH THE SUSCEPTIBILITY PROBE

The procedure that follows provides a step-by-step method for determining the critical temperature of the superconductor sample contained within the superconducting susceptibility probe included in the **Superconducting Magnetic Susceptibility Kit**. The experimenter will simultaneously measure the magnetic susceptibility of the sample as well. This probe consists of a 0.5 inch long coil of wire (approximately 400 turns) around a 0.5 inch diameter superconductor rod. A thermocouple is also attached for temperature measurement.

This experiment is based on the expulsion of a magnetic field by the superconducting sample (the Meissner Effect). A current introduced into the coil will generate a magnetic field. When cooled below the Critical Temperature, the sample expels the induced field that can be seen by a distinct change in the inductance of the coil. Analysis of the data collected in this procedure reveals a sharp transition point at the Critical Temperature, T_c, of the sample, and can also be used to compute the magnetic susceptibility of the superconductor.

Procedure

- <u>ACTION</u>: Set up the apparatus as shown in figure 11. A 50-ohm resistor should be connected in series with the signal generator to prevent any damage that might otherwise occur due to the low impedance of the susceptibility probe. Set the signal generator output a sine wave (ac) at a frequency of approximately 1 kHz (integer multiples of 60 Hz should be avoided).
- <u>ACTION</u>: Carefully straighten the thermocouple leads. Attach them to a dc voltmeter with a precision of at least 0.01 mV. Use the table on page 12 or 44 to compute the temperature from the thermocouple voltage.
- 3. <u>ACTION:</u> Insert the susceptibility probe into the **CSI Sand Cryostat™**. Fill the cryostat with enough 'sand' to completely cover the probe. The amount of 'sand' can be used to vary the rate of warming of the sample.
- 4. <u>ACTION:</u> Add liquid nitrogen slowly until the temperature is below 80 K.
- <u>ACTION:</u> Allow the sample to warm slowly. Periodically record the current (I), the voltage across the coil (V_L), and the thermocouple temperature, until the sample warms to about 120 K.
 - <u>RESULT:</u> As the sample reaches the critical temperature, the induction voltage (V_L) will increase dramatically. A slower warming rate at this point is advantageous in order to obtain accurate measurements.



Figure 11: Schematic of apparatus configuration

The resistance of the coil (R_L) is temperature dependent; therefore we now need to determine the value of R_L as a function of temperature. The following procedure will allow the experimenter to accurately measure R_L as a function of temperature:

- 1. <u>ACTION:</u> Connect an ohmmeter to the inductor leads to record the resistance (R_I), and a dc voltmeter to the thermocouple leads to record the temperature of the coil.
- <u>ACTION:</u> Using the same procedures as in the first part of the experiment, immerse the probe in the Sand Cryostat and cool it with liquid nitrogen to below 80 K.
- <u>ACTION:</u> Allow the probe to warm gradually. Record both R_L and the thermocouple temperature continuously until the probe warms to about 120 K.
 - <u>RESULT</u>: You now have a measurement of the resistance of the coil as a function of temperature. You will need this information to accurately compute the inductance of the coil, and magnetic susceptibility of the sample.

Note: For both this and the previous experiment, the connections of the thermocouple leads should remain at room temperature if the conversion chart on page 12 is used. Vapors from the liquid nitrogen can cool the connections, resulting in incorrect temperature readings. Since these vapors collect near the apparatus, the thermocouple leads should be kept as far away from the apparatus as possible, and should be elevated a few inches above the surface upon which the apparatus rests.

Precautions:

- Students should not touch any object cooled in liquid nitrogen. If the apparatus needs to be moved, tongs or protective gloves should be used.
- If a sensitive lab surface is being used, a block of wood or thick foam should be placed between the bench and apparatus in order to prevent damage.

ANALYSIS OF DATA

The critical temperature (T_c) at which the superconducting transition takes place can be seen clearly by plotting the inductance of the coil vs. temperature. An example plot is given in figure 12 on the following page. The inductance of the coil (ωL) can be computed using the following equation:

$$\omega L = \sqrt{\left(\frac{V_L}{I}\right)^2 - R_L^2}$$

Where V_L is the voltage of inductance (the ac voltage measured across the black coil leads), and R_L is the resistance of the coil. Since ceramic superconductors do not have sharply defined critical temperatures (the transition is visible over a range of 5K in figure 12), the midpoint of the range is usually taken as T_c.

An ideal superconductor screens the B-field completely at B-fields lower than the critical field. This makes a superconductor perfectly diamagnetic and thus the magnetic susceptibility (χ) is equal to -1.

If the coil contains a sample with χ not equal to -1, the magnetic flux through the coil will change, resulting in a change in the inductance of the coil. The inductance of a coil measured in a medium of susceptibility χ is given by:

$$L = L_{\circ}(1 + \chi)$$

Where L_{\circ} is the inductance of the coil in a vacuum. This equation holds for any sample provided that the sample occupies all the space in which the coil produces a field. Since our sample occupies most of the volume we can approximate the inductance (L) by adjusting the above equation to account for this difference:

Material	Core diameter	Coil diameter (38 gauge wire is used for the coil)				
		Inner	outer			
Yba ₂ Cu ₃ O ₇	1.18 cm	1.28 cm	1.32 cm			
Bi ₂ Sr ₂ Ca ₂ Cu ₃ O ₉	1.40 cm	1.50 cm	1.54 cm			

```
L \cong L_{\circ}(1 + f\chi)
```

Where *f* is the fraction of the coil volume occupied by the sample.

It follows that χ is given by the equation:

$$\chi = \frac{1}{f} \left(\frac{L}{L_{\circ}} - 1 \right)$$

Calculate χ with the data collected in the previous exercises and plot χ vs. T. You will notice that the plot yields the same type of graph as ω L, as well as the same critical temperature.

You may notice that although the superconducting transition is clearly visible, the susceptibility in the superconducting state is not exactly -1. This is due in part to geometrical corrections, which were neglected in our equation for χ , which can result in an error in susceptibility. In addition, polycrystalline samples of YBa₂Cu₃O₇ contain grains of material with large lower critical fields that undergo a transition at 90 Kelvin, but material between the grains has smaller lower critical fields and not all of this material will become superconducting in the apparatus. This can be verified by noticing that the fraction of the sample which screens the B-field is smaller in an applied field of 30 Gauss than in an applied field of 3 Gauss, and the transition temperature is depressed in the stronger field.



Figure 12: Probe coil induction versus temperature

We thank Professor Howard Lukefahr and his team at Whittier College, California for the basis and guidance for this experiment.

WHERE DO WE GO FROM HERE?

We hope that your Kit has been a good introduction to the new, high temperature superconductors. The Kit represents a very unique opportunity for you to have become involved in the very cutting edge of science. To many physical scientists these superconductors are still as novel as they are to you.

High temperature superconductors are expected to have a major impact upon several industries. For example, in the United States today, it is estimated that fully one-half of the generated electricity is lost in transmission. Electric power transmission lines made from superconductors will lose little energy. resulting in major economic, environmental, and political impact. It is also possible to make extremely powerful magnets with superconductors that could revolutionize basic research, medical diagnostics, resource recovery, and a host of other industrial fields. Magnetically levitated trains, frictionless magnetic bearings, vibration-free mounts will be among the first applications. Powerful and extremely fast computers can be made utilizing the Josephson Effect and other properties of superconductors. A host of sensors with esoteric names like SQUIDs (Superconducting Quantum Interference Device) and IR bolometers are already being used as probes for sensitive, non-invasive investigations in medicine. science, and defense technologies. As demonstrated by our Superconducting Energy Storage (Battery) Kit, another application harnesses the persistent currents in superconductors to store electrical energy far more efficiently than in any extant battery, with important application in lasers, space propulsion, and electricity generation and storage. Yet, it is in the completely new and unique applications of superconductivity that one should expect to see the greatest and most far-reaching implications.

While using this kit, you will have become familiar with the use of liquid nitrogen. You will note that with due precautions, it is quite easy to handle. Therefore, even though the pursuit of ever higher Critical Temperatures continues, the superconductors on hand are already viable for commercial exploitation.

Rapid progress is occurring in this field. Scientists are discovering new features of these materials at a breathtaking rate. There are expectations that one day perovskite superconductors will be found that may function at room temperature. This will be an even more incredible breakthrough. As it is, one can quite easily use the current superconductors with simple refrigeration systems. With room temperature superconductors, we will perhaps witness an industrial revolution rivaling the magnitude of the last one.

Given the rate of progress, and the potential impact of this new discovery, it behooves us to keep up to date in this emerging field. The newspapers and even popular magazines carry articles about the new superconductors almost daily. These superconductors could very well be the basis of your job in the near future.

PART IV

APPENDIXES

Sand Cryostat[™] Instruction Manual

Introduction:

The Sand Cryostat was developed as a simple and inexpensive system of temperature regulation for use specifically with the Four-Point Probe systems and Susceptibility probes created by Colorado Superconductor, Inc. for controlled measurement of superconductivity versus temperature. Because of its simplicity, however, the Sand Cryostat is easily adaptable to most any application requiring a controlled temperature environment.

General Theory:

By having a small space between the insulated mug, and the area where the probe will be (inside the aluminum can), one can have better control of the rate that the probe will warm up, along with reducing the temperature gradient the probe experiences.

Instructions:

As with any experiment, read all of the instructions and safety precautions before beginning.



Figure 1: Cross section of Sand Cryostat

4 point probe procedure:

- 1. Place the probe inside the aluminum can.
- 2. Pour the sand into the cryostat chamber until it completely covers the probe.
- SLOWLY pour liquid nitrogen into the Sand Cryostat until it is at a visibly higher level than the sand.

Note: As the sand and probe are cooling, the liquid nitrogen will boil furiously. Be sure that the sand is still covered by the liquid nitrogen when the boiling has subsided.

4. Carefully put the fiberglass insulation and foam cap on the can (there is a small length of tubing, that will fit though the hole in both, so that the wires can easily be passed though.

Warning: At first, when adding the nitrogen to the space between the can and mug, it may "spit" (as one would expect bacon grease to on a hot grill) as it boils.

- Slowly add liquid nitrogen to the space between the can and the mug. The material under the can will act as a secondary heat sink.
- As soon as the measured temperature starts to rise (@ 79K), add some liquid nitrogen to the space between the can and the mug. Continue doing this for each increase of 1 Kelvin in temperature, as measured by the thermocouple.

Tip: if you gently, and slowly pour it against the lip of the can, it will fall down the sides, with little splash over.

If you see a sudden "jump" in the measured temperature, add a little liquid nitrogen (just a tiny bit to cause the temperature to drop to 77K, but not so much, that there will be a large pool of liquid nitrogen present that one will have to wait for to evaporate) to the inside of the can, where the probe is. After a minute or so, it should begin to warm up again.

AC Susceptibility probe procedure:

The aluminum can, for these measurements, must be removed. Gently pulling on the can, while holding the mug in place will allow it to easily slide free. There is a layer of fiberglass insulation that is wrapped around the can that should be removed as well. Rubber gloves should be used if one's skin is easily irritated by fiberglass. The insulation used is simple fiberglass insulation, so it is readily obtainable from any building supply store, in the event that more is needed.

- 1. Add enough sand until the narrowest section of the sand cryostat is filled.
- 2. Place the probe inside the sand cryostat, and then add the rest of the sand.
- SLOWLY pour liquid nitrogen into the Sand Cryostat until it is at a visibly higher level than the sand
- 4. Place the fiberglass insulation over the top of the sand, then put the lid on the cryostat, with the wires emerging from the hole.

Precautions:

- 1. When pouring liquid nitrogen, be careful to prevent any splashing. Read the section on safety and handling starting on page 13 of the Experiment Guide before using the Sand Cryostat.
- Be careful not to touch the probe, its wires, the sand, or the can when they are cold. The fiberglass insulation that is present, may contain trapped pockets of liquid nitrogen, one needs to be careful, (use tweezers, and not fingers) when it is being removed so that the probe can be taken out of the cryostat. Follow the safety directions.
- 3. Use a hair dryer to carefully dry any superconductor devices after use. Store them with a desiccant.
- The Sand Cryostat is designed for sturdiness and durability, however devices used in the Sand Cryostat may become very brittle and fragile when cold.
 Handle with care.

When liquid nitrogen is poured into the Sand Cryostat, it boils furiously and may cause sand to be ejected from the container if not careful. Take caution to pour slowly, thus minimizing any splashing outside of the cryostat. This may take some practice. Place an open newspaper page underneath the Sand Cryostat to catch any sand that may boil out.

A few experiments with condensing air:

A rather nifty experiment you can demonstrate with this cryostat, is to leave the aluminum can empty, remove the fiberglass insulation between the can and the mug, then continually pour liquid nitrogen in the space between the can, and the mug. After a short time, you will notice that air will begin to condensate along the surface of the can, and will pool in the bottom. Once you have a pool of air, you can take a magnet and dip it in the condensed liquid (which is a mixture of nitrogen, oxygen for the most part). When the magnet is removed, it will have attracted the oxygen to it, and you will have liquid oxygen sticking to your magnet. Ideally, it would be nice to have two magnets like that, separated by a small distance, so the oxygen will fill the gap.

A similar experiment is to remove the can completely, from the mug, and pour liquid nitrogen into the can. Then with a pen, gently tip the can, so you can slide a small shim (two quarters should work well enough) under it, such that the can is gently leaning to one side (don't tip it so far that it falls over and spills mind you, that stuff is what an old professor of mine would have technically called "damn cold"). With a large magnet, place it near the side that the can leans towards. Air (nitrogen, oxygen, mostly) will condensate on the surface the can, in the same way that water condensates on a cold can of soda. However, the oxygen in the air will be attracted to the magnet, an instead of dripping down to the table, will drip horizontally towards the magnet.

WARNING: Anything that burns in air will burn violently in the presence of liquid oxygen, and that oxygen is a strong oxidizer. Liquid oxygen, while non-flammable, supports and vigorously accelerates combustion of flammables. Prevent liquid oxygen from contacting grease, oil, asphalt or combustibles. These experiments are not meant to be performed by those not familiar with the safe handling of oxygen.

Other Applications for the Sand Cryostat

The sand included with this kit has been washed to remove all fine dust that could damage superconductor devices. It is a thermally stable material, and is readily accessible, making it an excellent medium for use in temperature control experiments like the Critical Temperature experiment described in this manual. The sand's physical properties also make it ideal for many other applications. Its potential as a medium of temperature control is limited only by your imagination.

Here are a few ideas and points to consider:

- The rate at which the sand changes temperature is approximately proportional to the surface area
 of the sand divided by the volume; in other words, by minimizing the surface area of the sand, you
 also minimize the rate of temperature change.
- The sand can be heated to temperatures higher than room temperature by means of a microwave oven (don't microwave the probe), but a conventional or lab oven would be more effective for heating to a specific temperature. The plastic design of the container, however, will not withstand temperatures above 100 Celsius. Caution should always be used when handling hot items.
- To avoid errors in recording data, the device outputs could be input directly into a computer or chart recorder. This would also facilitate the interpretation of the acquired data.

What other ideas can you think of?

Thermocouple Reference Manual

Introduction:

A temperature difference between any two points in a non-superconducting metal, when no current is allowed to flow, will result in a electrostatic potential difference. This potential difference is proportional to the temperature difference between the two points.





However, with the voltmeter leads directly connected to a point at temperature T1, and to a point at temperature T2 the measured potential will be the difference between the electrostatic potential due to the metal sample, and also the electrostatic potential that will arise within the wires directly connecting the potentiometer to the metal sample.

To avoid any additional potential developing at the points of contact, these points must be at the same temperature. This can be accomplished by using another strip of metal that is dissimilar to the first. Metal 1, will have a potential drop of V1 between the temperatures of T2 and T1. Metal 2 will have a potential drop of V2 between the temperatures of T1 and T2. The voltmeter will show the sum of the drops, V1+ V2.



Figure 2

The measured potential is dependent upon the temperature difference between T1 and T2, and not on the specific temperatures. It is standard practice therefore to hold the temperature T2 constant. An easy method is to set T2 to 0°C by placing it in an ice water bath. As long as ice is present, the temperature of the bath will remain 0°C.

Using a thermocouple to make accurate temperature measurements:

Figure 3 shows how one can set up a thermocouple using a reference junction. The **thermocouple measuring junction** is at the unknown temperature, T1, and the **reference junction** is at the known temperature, T2, which in this case is a 0° C ice bath.



Figure 3

Conversion from mV to Celsius

٥C	0	1	2	3	4	5	6	7	8	9	10	°C
-190	-5.439	-5.456	-5.473	-5.489	-5.506	-5.523	-5.539	-5.555	-5.571	-5.587	-5.603	-190
-180	-5.261	-5.279	-5.297	-5.316	-5.334	-5.351	-5.369	-5.387	-5.404	-5.421	-5.439	-180
-170	-5.070	-5.089	-5.109	-5.128	-5.148	-5.167	-5.186	-5.205	-5.224	-5.242	-5.261	-170
-160	-4.865	-4.886	-4.907	-4.928	-4.949	-4.969	-4.989	-5.010	-5.030	-5.050	-5.070	-160
-150	-4.648	-4.671	-4.693	-4.715	-4.737	-4.759	-4.780	-4.802	-4.823	-4.844	-4.865	-150
-140	-4.419	-4.443	-4.466	-4.489	-4.512	-4.535	-4.558	-4.581	-4.604	-4.626	-4.648	-140
-130	-4.177	-4.202	-4.226	-4.251	-4.275	-4.300	-4.324	-4.348	-4.372	-4.395	-4.419	-130
-120	-3.923	-3.949	-3.975	-4.000	-4.026	-4.052	-4.077	-4.102	-4.127	-4.152	-4.177	-120
-110	-3.657	-3.684	-3.711	-3.738	-3.765	-3.791	-3.818	-3.844	-3.871	-3.897	-3.923	-110
-100	-3.379	-3.407	-3.435	-3.463	-3.491	-3.519	-3.547	-3.574	-3.602	-3.629	-3.657	-100
-90	-3.089	-3.118	-3.148	-3.177	-3.206	-3.235	-3.264	-3.293	-3.322	-3.350	-3.379	-90
-80	-2.788	-2.818	-2.849	-2.879	-2.910	-2.940	-2.970	-3.000	-3.030	-3.059	-3.089	-80
-70	-2.476	-2.507	-2.539	-2.571	-2.602	-2.633	-2.664	-2.695	-2.726	-2.757	-2.788	-70
-60	-2.135	-2.186	-2.218	-2.251	-2.283	-2.316	-2.348	-2.380	-2.412	-2.444	-2.476	-60
-50	-1.819	-1.853	-1.887	-1.920	-1.954	-1.987	-2.021	-2.054	-2.087	-2.120	-2.135	-50
-40	-1.475	-1.510	-1.545	-1.579	-1.614	-1.648	-1.683	-1.717	-1.751	-1.785	-1.819	-40
-30	-1.121	-1.157	-1.192	-1.228	-1.264	-1.299	-1.335	-1.370	-1.405	-1.440	-1.475	-30
-20	-0.757	-0.794	-0.830	-0.867	-0.904	-0.940	-0.976	-1.013	-1.049	-1.085	-1.121	-20
-10	-0.383	-0.421	-0.459	-0.496	-0.534	-0.571	-0.608	-0.646	-0.683	-0.720	-0.757	-10
0	0.000	-0.039	-0.077	-0.116	-0.154	-0.193	-0.231	-0.269	-0.307	-0.345	-0.383	0
0	0.000	0.039	0.078	0.117	0.156	0.195	0.234	0.273	0.312	0.352	0.391	0
10	0.391	0.431	0.470	0.510	0.549	0.589	0.629	0.669	0.709	0.749	0.790	10
20	0.790	0.830	0.870	0.911	0.951	0.992	1.033	1.074	1.114	1.155	1.196	20

Note that this conversion table differs from that on page 12 on three important points.

1. The measurements are more exact, 0.001mV resolution as compared to the 0.01mV.

2. These values are converting from millivolts to degrees centigrade.

The thermocouple leads are placed in an ice water bath, to maintain a constant temperature of 0°C, and not that of room temperature.

PART V

REFERENCES

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Science News High Technology Business

Some more in-depth journals with longer articles:

Nature Science

For business news on the New Superconductors:

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Introductory books on superconductors, and electricity & magnetism:

Superconductivity - The Threshold of a New Technology. Jonathan I. Mayo.

This book explains the properties of superconductors. What superconductors can and cannot do. The book explores the impact of the applications of superconductivity, with particular emphasis on power systems, electronics, science, medicine, and transportation. 144 pages.

Superconductivity - Experimenting in a New Technology. Dave Prochnow.

The exploration of the basics of superconductor chemistry, quantum mechanics, and thermodynamics along with the relevant underlying equations makes this a suitable companion volume to our instruction manual. The step-by-step experimental procedures find a parallel in our suggested approach. 138 pages.

Understanding Magnetism, Magnets, Electromagnets, Superconducting Magnets. Robert Wood. This very unassuming book describes the mysteries of magnetism and electromagnetism for the young reader with thirteen hands-on experiments to provide a better understanding of magnetic fields that are so important to the understanding of superconductivity. 176 pages.

Additional copies of the instruction manual.

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Also . . .

We would appreciate hearing from you if you have any comments about our Kit(s) or instruction manual. Please write or call us at:

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