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.

PART I

THE FUNDAMENTALS OF SUPERCONDUCTIVITY

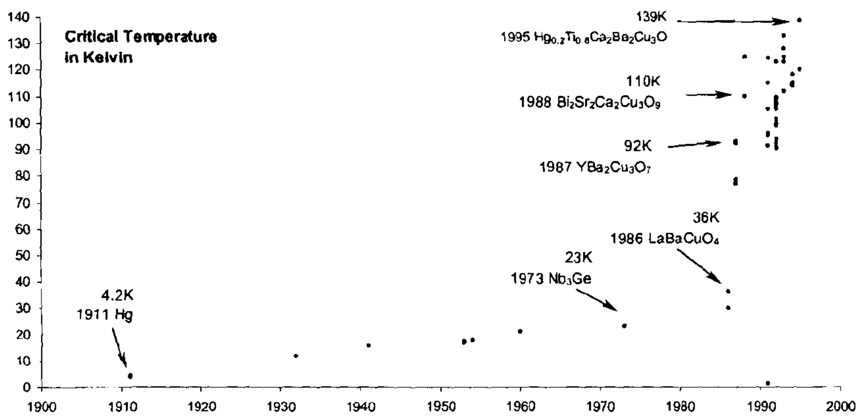
THE HISTORY OF SUPERCONDUCTORS

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 at somewhat higher temperatures. Unfortunately, none of these alloy 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 43.

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 Cooper Pairs, 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 superconductor must operate below a certain temperature called the Critical Temperature, or T_c. 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 $YBa_2Cu_3O_7$, and for $Bi_2Sr_2Ca_{n-1}Cu_nO_9$, 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 Critical Current Density, 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 quenched, and can carry no superconducting current. This maximum magnetic field is called the maximum 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. Perovskites 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 $YBa_2Cu_3O_7$ 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 $YBa_2Cu_3O_9$. Surprisingly, scientists have found that this superconductor has about 2 oxygen atoms less than predicted, and instead has the approximate formula $YBa_2Cu_3O_7$, this is also sometimes written as $YBa_2Cu_3O_{7-\delta}$.

One should note that the proportions of the 3 different metals in the $YBa_2Cu_3O_7$ 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 1-2-3 superconductor. For the bismuth-based superconductor, one chemical formula is $Bi_2Sr_2Ca_{n-1}Cu_nO_9$ and it is often referred to as the 2-2-(n-1)-n 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 $YBa_2Cu_3O_7$ 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 $YBa_2Cu_3O_7$, 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. $Bi_2Sr_2Ca_{n-1}Cu_nO_9$ 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.

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.

Conversion from mV to Kelvin

°K	0	1	2	3	4	5	6	7	8	9	10	°K
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
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
180	2.82	2.79	2.76	2.73	2.70	2.67	2.64	2.61	2.58	2.53	2.52	180
190	2.52	2.49	2.46	2.43	2.40	2.37	2.34	2.31	2.29	2.26	2.23	190
200	2.23	2.20	2.17	2.14	2.11	2.08	2.05	2.02	1.99	1.96	1.93	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

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 III

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, and thus the associated magnetic field would also continue to oppose the applied field. Conversely, if the 'perfect' conductor was already in a magnetic field, and then that applied field was removed, the same physical law 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 quite 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 <u>Meissner Effect</u> 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. ACTION: Using the provided tweezers, carefully place the black superconductor disk carefully in a Pyrex dish, or in a appropriately shaped Styrofoam cup.

2. ACTION: 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.

3. ACTION: 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.

   RESULT: 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 12 to 14), 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 frictionless magnetic bearing 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

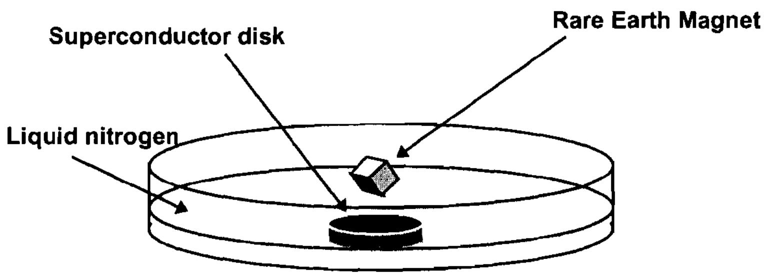
1. Why does the liquid nitrogen boil when you pour it into the dish? Why does it boil around the superconductor disk?

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 Temperature Comparison Kit* are designed for this purpose.

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. ACTION: Carefully straighten the thermocouple leads and attach them to a voltmeter that can measure and display in the 0.01 milliVolt range.

2. ACTION: 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. ACTION: 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. ACTION: Carefully balance the small cubical magnet so that it 'floats' via the Meissner Effect over the center of the disk.

5. ACTION: 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.

   RESULT: 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 $YBa_2Cu_3O_7$, and about 110 Kelvin for $Bi_2Sr_2Ca_2Cu_3O_9$.

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 13) before using liquid nitrogen.

2. Use the provided non-magnetic tweezers when handling the device or magnet.

3. 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?

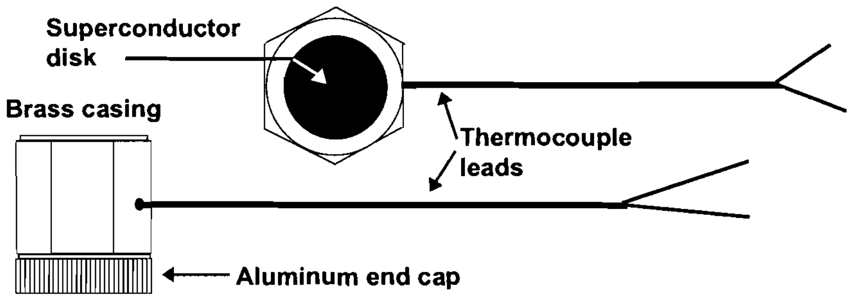


Figure 1: **The Superconducting Thermocouple Device**

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 $Bi_2Sr_2Ca_2Cu_3O_8$ 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 & 3. Thus, since there is no potential drop across the contact resistance associated with probes 2 and 3, the resistance associated with only the superconductor between probes 2 and 3 is measured.

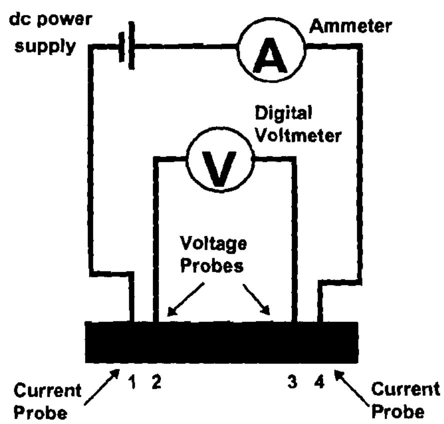


Figure 2: **Schematic of Four Point Probe**

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 40).

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.

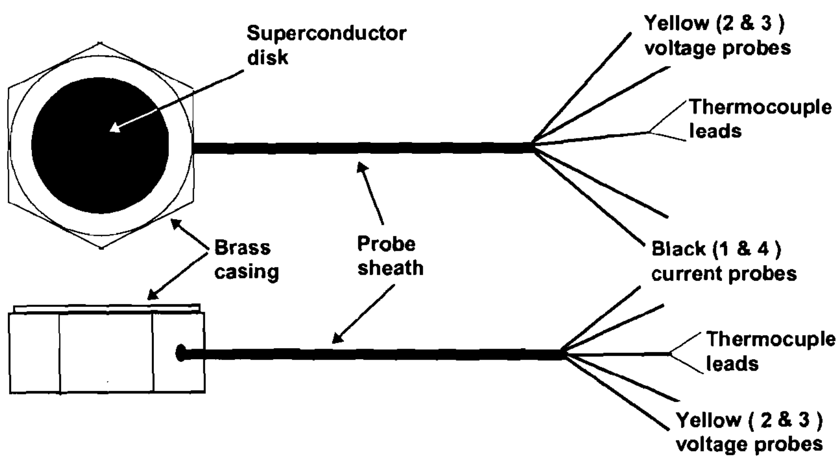


Figure 3: **The Superconducting Four Point Probe**

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 Critical Temperature, Critical Current Density, and the Critical Magnetic Field, can all be obtained through variations of this basic experiment.

This experiment requires the following pieces of equipment:

1. 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 I_{14}.

2. 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 V_{23}.

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. ACTION: Set up the measurement equipment as described above, but do not as yet immerse the device (four point probe) in liquid nitrogen.

2. ACTION: Insert the device into the CSI Sand Cryostat or other certified container and carefully fill it with liquid nitrogen. Ensure that the current (I_{14}) remains constant at less than 0.5 Amp.

   RESULT: The nitrogen boils furiously. Wait until the boiling subsides.

3. ACTION: Record the voltage V_{23}, and across the thermocouple junction.

   RESULT: V_{23} should equal zero. The thermocouple temperature reading should be 77 K.

4. ACTION: 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 V_{23}. Record the thermocouple temperature each time V_{23} is recorded.

   RESULT: Initially, V_{23} remains constant even as the thermocouple temperature increases. Then the voltage between the probes (V_{23}) abruptly increases, the thermocouple reading corresponding to this jump in voltage is the Critical Temperature, or T_c of the superconductor. The ratio of the voltage between probes 2 & 3 (V_{23}) to current flowing between probes 1 & 4 (I_{14}) 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.

4. 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 $Bi_2Sr_2Ca_2Cu_3O_9$ superconductor is about 110 Kelvin versus about 92 Kelvin for the $YBa_2Cu_3O_7$ material. These results are shown below in figure 4.

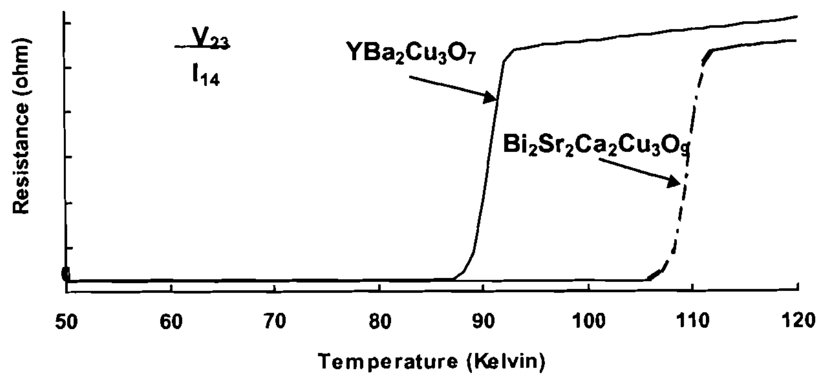


Figure 4: **Resistance versus Temperature**

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. ACTION: Set the current through probes 1 and 4 at 0.1 Amp, and measure the Critical Temperature as described on page 23 of the Instruction Manual. Record the measured T_c versus the value of current used.

2. ACTION: 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.

   RESULT: Five data points will be obtained, each at a Critical Temperature, T_c, versus the set current, I_{14}. 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 $Bi_2Sr_2Ca_2Cu_3O_9$ 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
$Yba_2Cu_3O_7$	24 mm	4 mm	17.5 mm	11 mm	Surface contact
$Bi_2Sr_2Ca_2Cu_3O_9$	30 mm	5 mm	17.5 mm	11 mm	1.75 mm

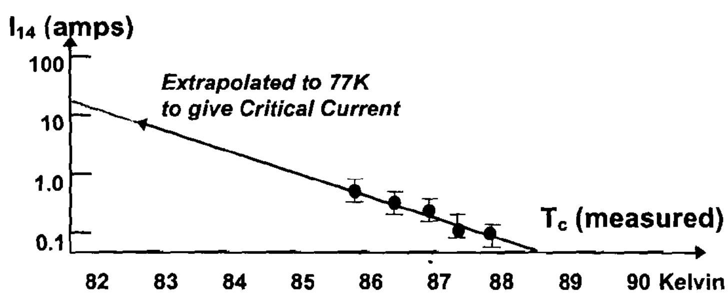


Figure 5: **The evaluation of Critical Current**

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 24 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 V_{23} will show an abrupt increase at some value of applied magnetic field strength. This value of magnetic field is the upper Critical Magnetic Field, Hc_2 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 Hc_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 24), 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.

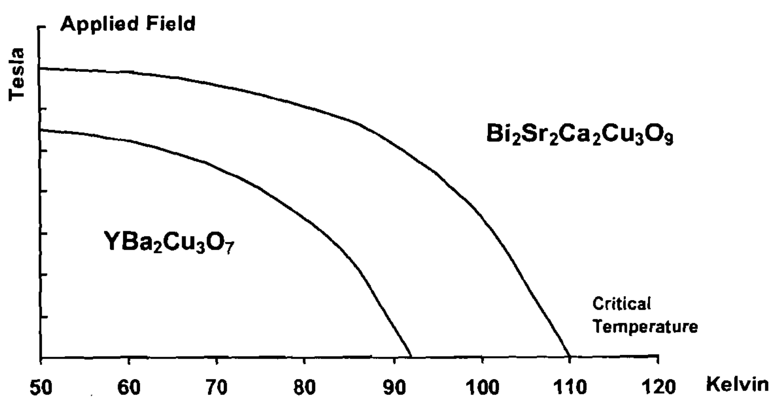


Figure 6: **Effect of Applied Magnetic Field on Critical Temperature**

Conversion from mV to Celsius

°C	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 11 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.
3. The thermocouple leads are placed in an ice water bath, to maintain a constant temperature of 0°C, and not that of room temperature.