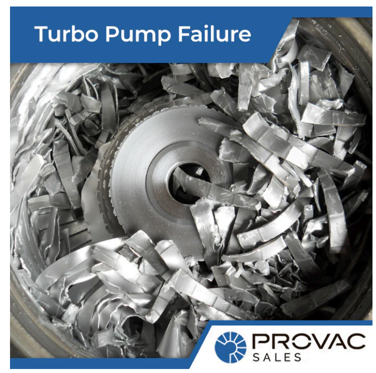
Superconductivity Short Lab Guide

This document serves as a succinct and pictorial lab guide of the superconductivity experiment and emphasizes the critical steps of the experiment. **Please also fully read the TeachSpin manuals before you start the experiment. Note**: This document may be further updated, as this experiment is new and we are also learning how to best do it. [Updated on April 24, 2024 by Dong Yu]

1. Safety tips



- **Use caution when handling liquid nitrogen.** Always wear protection gloves and face shields when handling liquid nitrogen.
- The HiCube pump can be damaged if you expose the pump to air before the Turbo pump is fully stopped. **The spinning frequency must read 0 Hz before you vent the pump.** Do NOT open the valve to air when the pump is running. Do NOT open the vent screw when the pump is running. See the result in the right figure when high pressure air is introduced suddenly into a pump with fast-spinning blades.
- **When the sample chamber is cold, do NOT vent it by air.** Always fully warm the chamber to room temperature before you vent it by air. Otherwise, the water vapor in air will condense on the wall of the vacuum chamber, which makes it hard to obtain a good vacuum after. The BSCCO sample can also be damaged if water accumulates on the sample. A high electric current may also damage the sample. **Do NOT pass a current larger than 100 mA through the sample.**
- Touching the interior of the vacuum chamber with your fingers can leave oil on the surface which can slowly evaporate and create a challenge for obtaining a high vacuum. As a good practice, **always wear gloves when handling components inside the vacuum chamber.**

2. Tips for conducting the superconductivity experiment

- Before carrying out the experiment, **first carefully read the Teachspin lab guides**. There are five files in the superconductivity folder in Canvas. The most important ones are “1. CMP Variable Temp and Support Electronics Manual” and “5 Superconductivity Chapters”.
- You may **skip magnetic susceptibility measurements** when reading, since we do not have the instruments to do that part.
- Spend some time to locate and study each component of the setup before you start the real measurements.
- It is a good idea to double check all electrical connections and do some tests before you seal the inner space containing the sample, as the assembly, pumping, cooling, and warming processes take a lot of time.
- **Develop a plan and manage time properly.** It takes about 1 hour to cool the sample to 77 K.
- Don’t hesitate to ask questions. Instructors, TAs, and lab managers are happy to help. **If you are uncertain, ask!**

3. Tips on sample change and electrical connection

Use caution when replacing samples. First invert the cryostat and lock it with the restraining screw. After removing the outer chamber wall, the inner sample chamber can then be opened by carefully unscrewing the 18 screws (Fig. 1 left). Each screw has two conical spring washers. Keep all screws and washers in a box. Use the small screwdriver to loosen and tighten the electrical connectors (Fig. 2 right). The wires are thin, so be careful not to break them. Write down how these wires are connected.

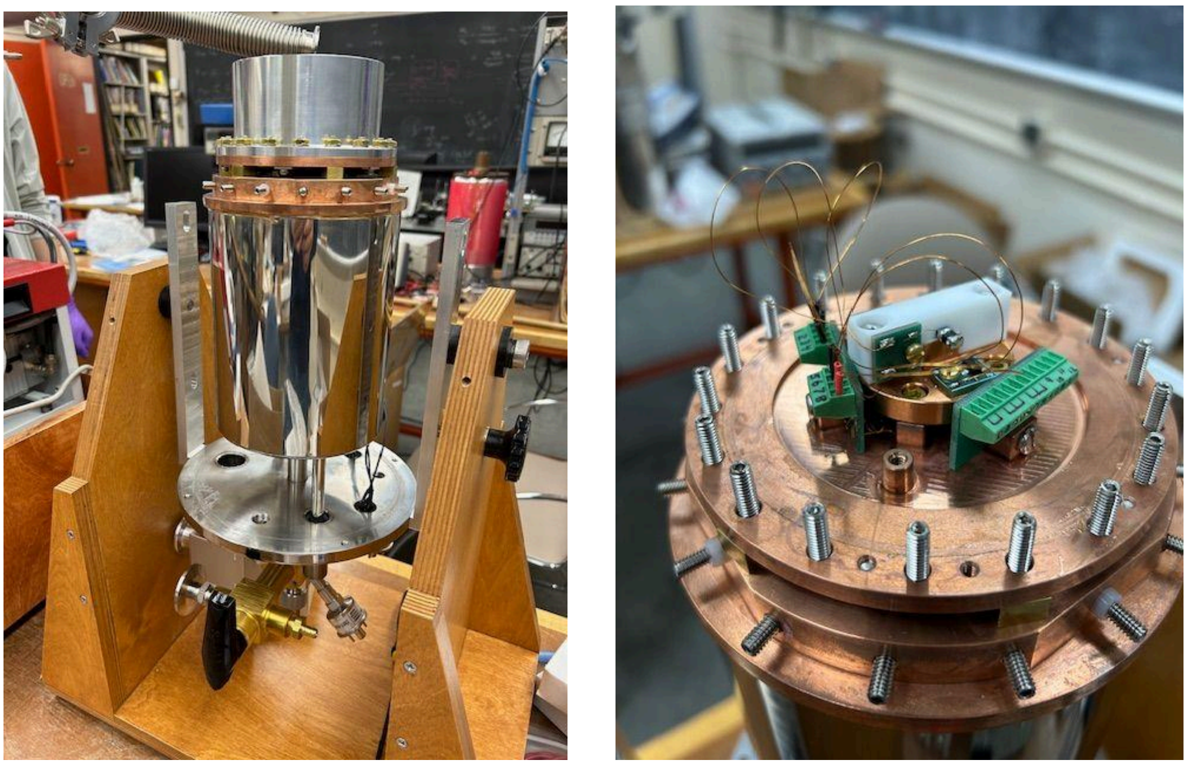
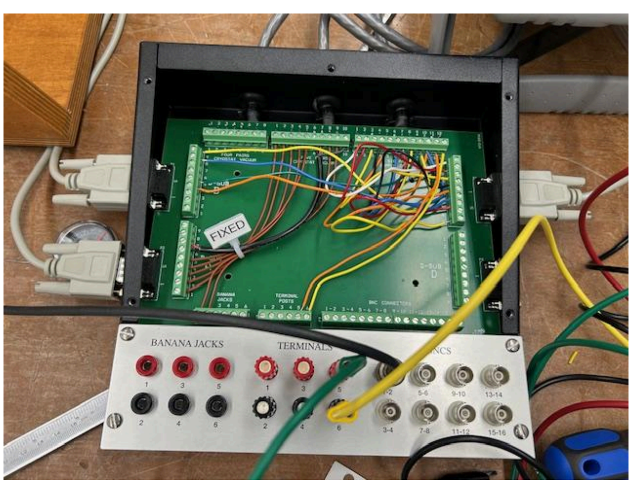


Fig. 1. Left: inverted cryostat with the outer chamber wall removed. **Right:** the inner sample chamber with the cap removed.



The Cryostat Interface Box provides convenient electrical access to the samples as well as temperature sensor and heater. You may open it and connect terminals by provided wires. You can choose any configurations you like for the 12-wire channels. But **do NOT change the bundled wires labeled FIXED** shown in Fig. 2, as they are reserved for temperature control.

Fig. 2. Cryostat Interface Box with cover removed.

4. Tips on pumping the system

- Browse over the Pfeiffer HiCube ECO Manual. Understand that the system is made of two pumps: a diaphragm pump (under the red cover) and a turbo pump (above the red cover). The pump is fully automatic and most operations can be achieved from the DCU panel.

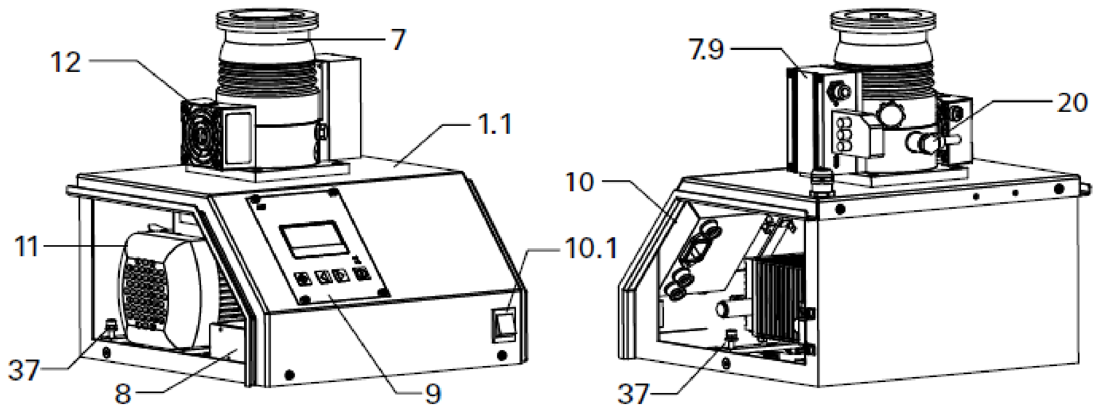
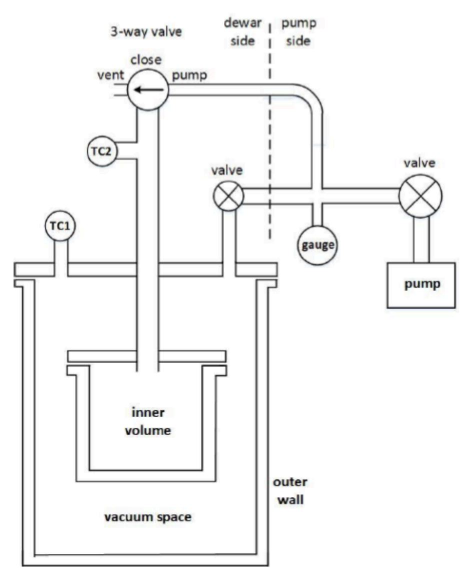


Fig. 2: Views of HiCube 80 Eco

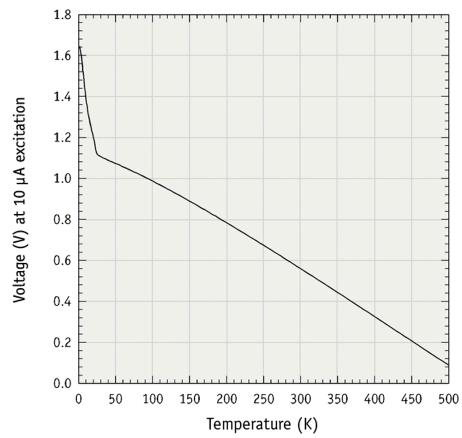
1.1	Casing	9	Display and control unit DCU 002 (option)	12	Air cooling unit
7	Turbopump HiPace 80	10	Relay box	20	Fore-vacuum connection
7.9	Electronic drive unit TC 110	10.1	Main switch	37	Hexagon nut, transporta-tion lock
8	Power supply pack TPS 110	11	Backing pump MVP 015-2		

- The turbo pump should generate little noise. The main sound you hear should be from the diaphragm pump. If you hear a high pitch noise, it is a warning sign and the pump should be turned off immediately. The DCU may also give a warning sign. Another warming sign is that the turbopump gets hot (it is normal if the pump gets a bit warm). Inform instructor and TA immediately if these warning signs occur.
- After the turbopump is switched off, it must be vented to avoid contamination due to particles streaming back from the fore-vacuum area. The pump rotation should be completely stopped if you want to manually vent the pump. **Never vent the pump when it is rotating at full speed! The sudden inrush of air can destroy the blades rotating at high speed.**
- Check o-rings carefully to remove any visible dirt, hair, and apply a small amount of grease when it looks dry. The KF clamp needs to be tightened firmly, though not with all your strength.
- The cryostat has two separate chambers sealed from each other as shown on the right. The "inner volume" contains the sample and its electrical connection. The "vacuum space" is outside the sample chamber. The two chambers are connected to the pump through separate paths and controlled by separate valves. Both should be pumped down to a pressure lower than 10^{-4} hPa before you add liquid nitrogen. After you add LN2, you should see that the pressure drops quickly (why?).



5. Tips for temperature control

- Always wear a face shield and insulation gloves when handling LN2. Gradually pour LN2 into the funnel and wait for the funnel to empty itself. Watch the voltage from the diode sensor increasing as temperature drops. Then repeat the process until the desired temperature is reached. It takes about 2 liters of LN2 to cool down the cryostat to 77K, then another 3 liters to fully fill the reservoir. Do not overfill.
- It takes about 1 hour to cool down the cryostat to 77K. The reservoir can store about 3 liter LN2. After the reservoir is filled, the base temperature can be maintained at 77 K for several hours.



- The diode sensor should read a voltage about 450 mV at 300 K and 990 mV at 77 K, when a 10 µA current flows through. The current is automatically applied when using the Teachspin PI Temperature Controller or SRS SIM922 Diode Temperature Monitor as shown on the right. The set point on the PI Temperature Controller is 10x the voltage of the diode sensor (for example, to reach 450 mV, you need to set it to 4.5 V). You may read the voltage output from the Teachspin PI Temperature Controller using a voltmeter or an oscilloscope. See a plot of voltage vs temperature above as a reference but keep in mind our sensor is slightly off from the above voltage values.
- If the temperature reading is off from the real temperature, you need to **calibrate the transdiode** temperature sensor properly to obtain the more accurate temperature value. The best way is to use 3-point calibration, with thermal baths of dry ice, LN2, and the ambient.
- There are **two transdiode sensors**, one for the sample, one for the base. Always use the transdiode close to the sample to report the sample temperature.
- Read and understand how to control temperature by balancing the cooling power from the LN2 reservoir and the heating power from the electrical heater. There are also **two heaters.** Each heater has a total resistance of 66 Ω and can output a max power of 60 W, as shown below. The two heaters can be used together when needed to reach a higher temperature.

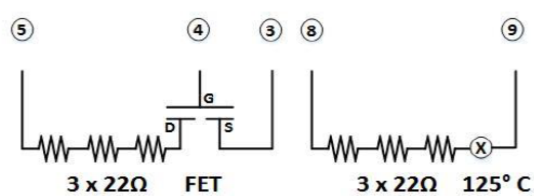


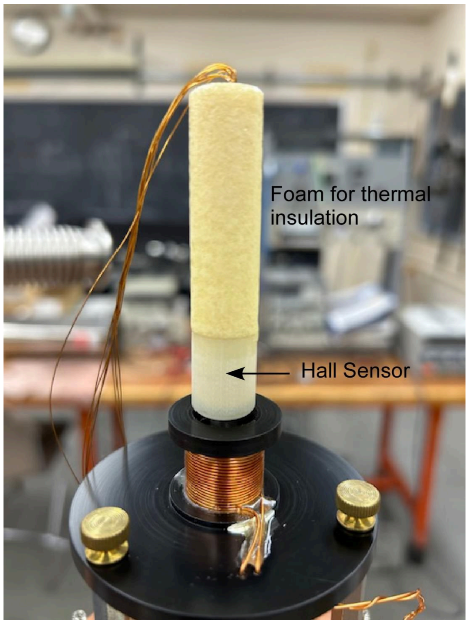
Fig. 1.3: The electrical connections from the 'bundle of 10' to the two separate heater circuits that are both mounted on the 'back' side of the experimental baseplate. The first heater shown delivers to the baseplate the dissipation in three resistors *and* in the current-controlling FET. The second 66-Ω heater shown has a reversible 'thermal fuse' which cuts off current above about 125 °C or 400 K.

6. Finish a run

- **Do not vent the cryostat when it's cold to avoid water/ice accumulation in the interior.**
- Instead, follow the procedures below:
  a. First close the main valve of the pump and turn off the pump. Wait until the blades fully stop rotating. Then vent the pump using the vent screw. Retighten the screw after it's fully vented.
  b. Equalize the pressure of the inner can and outer space Close the valves to both (do not vent the vacuum chamber). You may now remove the O-ring clamps connecting the pump to the cryostat.
  c. You may remove the remaining LN2 in the reservoir by carefully inverting the cryostat. Be careful not to spill LN2 on yourself or electronics. You may also boil off the remaining LN2 by turning on both heaters.
  d. After the reservoir is empty, turn off the heaters. Though the inner of the cryostat is still cold, the surrounding vacuum is sufficient to keep water from condensing. You may leave the lab at this time.
  e. The cryostat will slowly warm up to room temperature and the pressure will also slowly increase to about 1 Torr after overnight. Before you vent the chamber the next day (or the day after), double check the temperature sensor indeed reads about 300 K or 450 mV in voltage (the diode can have an offset).

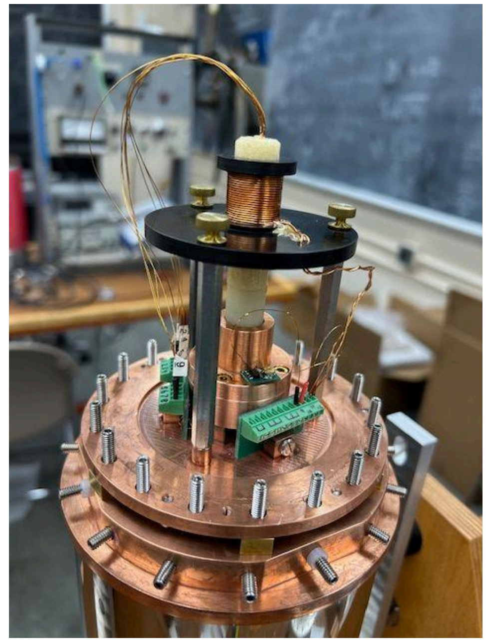
7. Persistent current experiment

- **Concept**: A bar magnet is inserted into the center of a hollow cylindrical superconductor at its normal state. After the sample is cooled to below T_c, the bar magnet is removed, leaving a persistent current in the superconductor. Then a Hall sensor can be used to measure the persistent current induced magnetic field. This can be done as a function of time to provide an estimate of the upper limit of the resistance in the superconductor.
- Vent the cryostat and open up the inner sample chamber. Remove the sample and the electrical connection for the previous measurements. Mount the sample for persistent current experiment. The hollow cylindrical BSCCO sample is sandwiched between and protected by copper sheath. Secure the screws firmly and check to make sure its bottom makes no gap with the baseplate for good thermal contact.

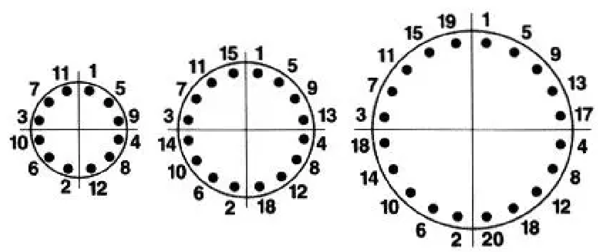


- Install the three standoffs and mount the calibration coil.
- Insert ¼" stainless steel tube of the Hall sensor through the calibration coil. The Hall sensor near the bottom of foam/garolite probe as shown on the right. The probe should be able to move smoothly through the calibration coil. Record the positions of the tube end outside the dewar using the machinist's scale, when the Hall sensor is moved to the center of the coil and the center of the superconductor sample, respectively. Also insert the bar magnet and record the position of the tube end when the bar magnet is moved to the center of the sample. You will need these positions later.

- The extract center position of the calibration coil can be found out by maximizing the magnetic field read from the Hall probe. The center position of the superconductor should be reached when the top (not counting the black handle) of the thinner magnet pushrod is at 11.4 cm reading from the scale.
- Make the electrical connections carefully including the Hall probe, calibration coil, and the temperature sensor, as shown in the right photo. **Do some tests before sealing off**, including applying a current through the calibration coil, measuring Hall voltage, and reading the diode voltage from the temperature sensor.



- Seal off the sample chamber when using the longer inner can. Tighten the 18 nuts following the sequence suggested by the diagram below. Do not fully tighten in one cycle. Instead, tighten evenly around in 2 or 3 cycles for best results. Wait for 5-10 min between each tightening cycle to let the O-ring fully relax. It should be tightened quite firmly but do not use your full strength. **It is important to seal the chamber tightly,** as you will introduce dry N2 to the sample chamber while maintaining a high vacuum in the outer space.



- **Use aluminum foil** to cover the sample chamber to reduce the radiation heat transfer from the outer wall at room temperature. This helps reach a lower temperature.
- Reinstall the outer can, tighten the screws, and reinvert the Dewar.
- The Hall sensor's working temperature is in the 155-180 K range. **Adjust the set point before cooling to roughly 800 mV** (check your calibrated voltage vs. temperature curve for more accurate values).
- Recheck the electrical connections. If all is good, pump down the chamber. Pour in LN2.
- When the baseplate has gotten cold, you may notice a large temperature difference, read from the base, sample, and Hall probe temperature sensors. To more efficiently cool down the sample chamber, you need to fill it with dry N2, which provides better heat transfer through convection.
- To do this, first use the three-way valve to isolate the inner space from the rest of the vacuum system. Then fill the inner space with dry nitrogen gas from the boil-off of the LN2 reservoir. Connect the yellow hose from one of the LN2 reservoir openings to the vent of the sample chamber and then slowly turn the three-way valve to suck in the dry N2 from the boil-off of the LN2 reservoir. **Watch the pressure level of the outer channel when you fill in the dry N2**, as the pressure may go up if there is a leak between the inner sample chamber and the outer space. If it occurs, immediately turn off the Turbo pump. You may then have to warm up the chamber to fix the seal of the inner chamber.
- Once filled to ambient pressure, seal the inner chamber with the top O-ring.
- When the Hall sensor reaches the working temperature range, calibrate it by using the calibration coil. Record the Hall voltage as you change the coil current from -1 A to + 1 A, with about 10 data points

evenly distributed in this range. Plot Hall voltage against the current and fit linearly. Does it go through the origin within the uncertainty of the measurement? If not, why?

- The details of the coil's geometry are found in Appendix B; this information will allow you to connect coil current and magnetic-field value.
- Insert the magnet to the center of the BSCCO above T_c and then lower the temperature. After the sample is well below T_c, remove the magnet. A persistent supercurrent should now flow in your BSCCO sample, creating a magnetic field.
- Now pull up the Hall sensor to the sample. You should see a large Hall voltage (larger than the Hall voltage created by 1 A in the calibration coil). Adjust its height to maximize the magnetic field measured by the Hall sensor.
- Calculate the persistent current using your measured magnetic field. Does the value make sense? Read the Appendix.

Now the fun begins. You can carry out several investigations as suggested in the TeachSpin lab guide and listed below:

- **Time dependence:** Investigate how the persistent current changes over time, as long as your patience allows. If desired, you can fill up the reservoir and check the persistent current next morning. The sample can be maintained at 80 K for >12 hours according to TeachSpin. From this you can estimate the resistance upper limit of the BSSCO sample.
- **Back to normal:** Warm above T_c to remove the persistent current. Does the Hall-voltage go away? If not completely, why?
- **Changing magnets**: Induce supercurrent with various magnets (there are four with different diameters and opposite polarities) and then measure the induced supercurrent with the Hall sensor. Do your results make sense?
- **Critical current**: Slowly increase the temperature when below but close to T_c. Does the persistent current decrease? If so, why? If you lower the temperature again, does the persistent current increase? Why or why not?
- **Field profile:** Move the Hall sensor up and down to determine the magnetic field distribution. Compare this to the theoretical calculated profile.
- **Be creative**: what else can you test to learn more about superconductivity?

[Need more pictures.]