



## YBCO Magnetic Characterization (Vibrating Sample Magnetometer Option)

<http://education.qdusa.com/experiments.html>

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*In this Educational Module, students will magnetically characterize a sample of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) superconductor, the first material discovered with a superconducting critical temperature ( $T_c$ ) above liquid nitrogen's boiling temperature.*

### **Introduction:**

Superconductivity is an exciting phenomenon whereby certain materials at low temperatures demonstrate zero electrical resistance and the expulsion of magnetic field, also known as the Meissner effect [1]. Although the former can be investigated using four probe electrical resistivity measurements, the latter can be measured from the magnetic susceptibility, as the behavior will appear to be diamagnetic at the transition. This is particularly useful when it is not practical or possible to attach electrical leads for the measurement, such as for a powder sample. With larger pieces of superconductor, it is even possible to levitate magnets (Figure 1).

This activity will make use of the Vibrating Sample Magnetometer (VSM) option of the VersaLab. In essence, a VSM oscillates a sample between detection coils in the presence of an applied magnetic field. The motion of the sample creates a changing flux in the detection coils, which in turn creates a measurable voltage. You should read more about the theory of operation in both the VSM manual and in reference [2].

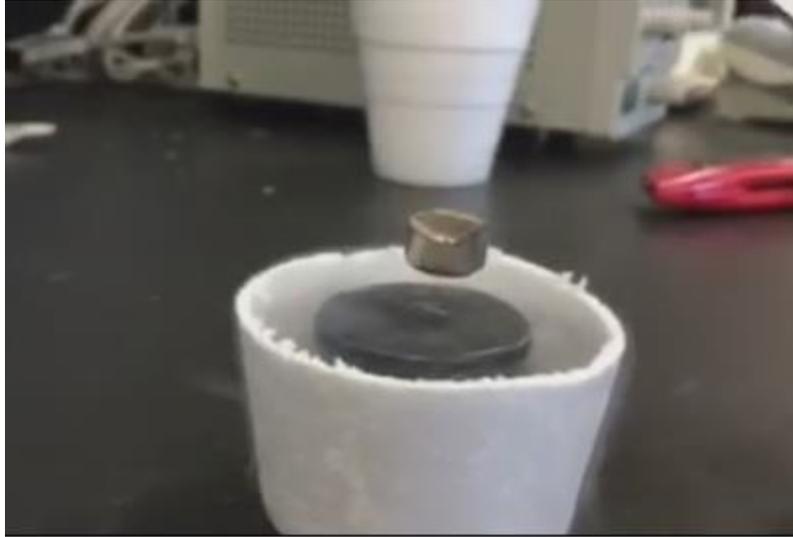


Figure 1. Levitation demonstration of a neodymium iron boride (NIB) magnet using  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) immersed in liquid nitrogen. One can see the potential benefits of having a superconductor that has a transition above 77 K.

You will magnetically characterize a sample of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) superconductor, which possesses a superconducting critical temperature ( $T_c$ ) near 90 K, 13 K above liquid nitrogen's boiling temperature (77 K) [3]. The observation of such a "high  $T_c$ " was exciting because previous superconductors required liquid helium cooling, which was impractical and cost prohibitive. The pursuit of high temperature superconductivity in this era created such a fervor that the 1987 American Physical Society March Meeting will forever be remembered as the "Woodstock of Physics," where on March 18, two thousand physicists and chemists crammed themselves into the New York Hilton ballroom and overflow rooms to listen to talks and hold discussions from 7:30 pm to 3:15 am [4].

Type-I and type-II superconductors have markedly distinct magnetic properties [1]. A sufficiently high magnetic field ( $H$ ) suppresses the diamagnetism abruptly in type-I, making the whole sample transition from superconducting to normal. A type-II superconductor in magnetic field behaves like a type-I, that is diamagnetically, up to the lower critical field  $H_{c1}$ . Between  $H_{c1}$  and the upper critical field  $H_{c2}$ , quantized flux lines start to permeate the material, such that this "mixed state" consists of both superconducting and normal regions. At  $H_{c2}$ , no superconducting regions remain, and the material becomes normal.

### **Student Learning Outcomes:**

- Students will learn how to synthesize ceramic superconductors by solid state reaction.

- Students will develop proficiency in sample mounting techniques for magnetic measurements.
- Students will operate the Vibrating Sample Magnetometer (VSM) option of the VersaLab cryostat and gain experience in low temperature experiments.
- Students will apply relevant and fundamental solid state physics knowledge to the magnetic susceptibility vs. temperature characterization of a superconductor.

### **Safety Information:**

Before attempting to perform any parts of this student experiment, please read the entire contents of: this Educational Module, the VersaLab User's Manual (1300-001), and the Vibrating Sample Magnetometer Manual (1096-100), and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the equipment, perform the experiments and reach the best student learning outcomes.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse, improper operation of the system and the information contained in this Educational Module.

The following Safety warnings apply to this Educational Module. We recommend that you study them carefully and discuss the details with your instructor before starting the work:

**WARNING!**



Always use **Personal Protective Equipment (PPE)** during every step of sample preparation. Failure to do so might cause bodily harm.





**TOXIC HAZARD!**



Acetone is toxic if swallowed. For more information consult the Material Safety Data Sheet available on this website:

<http://www.guidechem.com/msds/110-20-3.html>

### **CRUSHING HAZARD!**



Before using the die and hydraulic press, please read the entire contents of the User's Manual specific to that equipment, and observe all instructions, warnings and cautions. Failure to do so might cause bodily harm.

### **HOT SURFACE!**



Before using the furnace and combustion boats to sinter the samples, please read the entire contents of the User's Manual specific to that equipment, and observe all instructions, warnings and cautions. Failure to properly handle hot surfaces might cause bodily harm.

### **FIRE HAZARD!**



Before using the furnace to sinter the samples, please read the entire contents of the User's Manual specific to that equipment, and observe all instructions, warnings and cautions. Failure to do so might cause a fire hazard, bodily harm.

## **Materials List**

<b>Synthesis</b>
Y <sub>2</sub> O <sub>3</sub> Powder
BaCO <sub>3</sub> Powder
CuO Powder
Mortar and Pestle
Balance
Weighing Paper or Weighing Boats
Spatula or Scoopula
Hydraulic Press
13 mm Press Die
Tube Furnace
Alumina Combustion Boat

## YBCO Synthesis by Solid State Reaction

- 1) The precursor compounds are  $\text{Y}_2\text{O}_3$ ,  $\text{BaCO}_3$ , and  $\text{CuO}$ . Determine the stoichiometric ratios necessary to yield 2 grams of  $\text{YBa}_2\text{Cu}_3\text{O}_7$ . Focus on the Y, Ba, and Cu ratios, since the heat treatment will remove the C and supply the oxygen content.
- 2) You will decide on how to process your materials in the tube furnace. Search the physics and chemistry literature in order to propose a heat treatment recipe. Note that typical synthesis procedures require many hours in the furnace, such that it may take days to synthesize your sample.
- 3) Carefully weigh out the quantities required and place the compounds into a mortar bowl. Grind the combined powder using the mortar and pestle, applying a circular motion as you grind.
- 4) Use a chemical spatula or scoopula to remove the compound from the mortar and into a crucible. Apply your heat treatment procedure using the tube furnace. When heating the samples, it is suggested to provide oxygen flow via an air supply line. Note that many recipes call for multiple heating ramps with re-grinding in between treatments.
- 5) In order to create a ceramic pellet, extract the powder from the crucible and place it in a die for pressing. An application of 10,000 psi for one minute should be sufficient.

The material should be a deep black by the end of the process.



Figure 2. Completed YBCO pellet.

You will not actually require this much sample to perform the experiment. Pieces of the pellet can be broken off for different purposes, as will be discussed later.

## Sample Mounting with the VSM

Please refer to the VSM manual to properly load and measure samples. Samples may be mounted on either the brass holder or quartz paddle that comes with your VSM. GE 7031 varnish is a convenient medium to adhere samples to the holders, but time and care must be used to clean the varnish off with alcohol or toluene. You may also use Kapton tape to mount samples. Whatever you choose, be aware that mounting media may contribute to the measured signal, so it is best to establish what your experimental background is. For example, even the brass holder itself can provide a contribution on the order of  $10^{-6}$  emu at very high applied magnetic fields (Figure 3). This small signal will not interfere with your results, but a contribution of  $10^{-3}$  emu would be troubling.

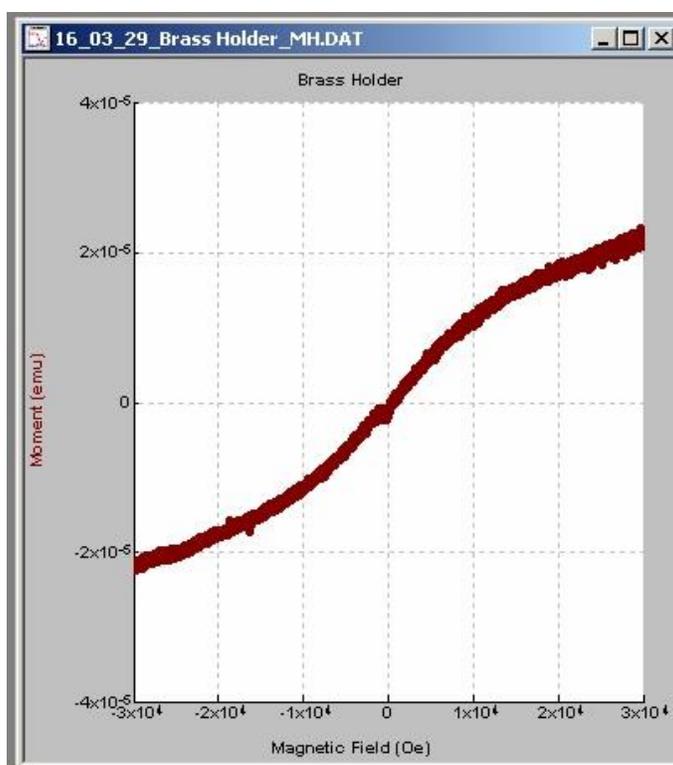


Figure 3. Moment vs. field for the brass holder without sample. Always determine the sample holder background before performing measurements.

Please read Application Note [1096-306](#) [5] on VSM sample mounting on the QD website for more guidance on this topic.

For now, choose your mounting medium (Kapton tape, GE varnish, etc.) and place that 35 mm from the end of your holder. This is facilitated by the brass sample holder mounting station (Figure 4). For future reference, you should

obtain the mass of the mounting medium, as the magnetization is often reported in units of emu/g.

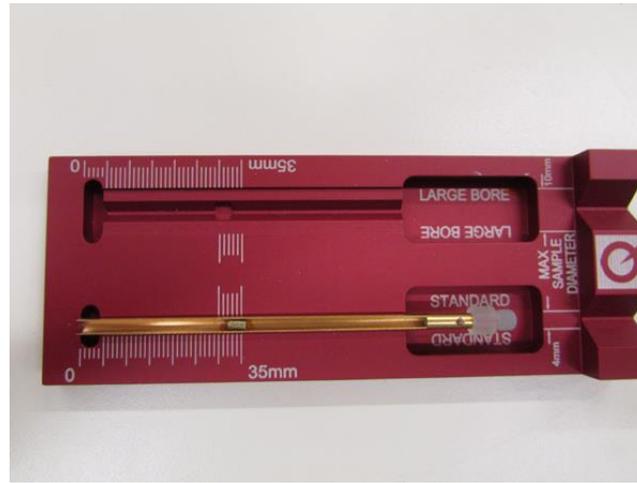


Figure 4. Brass sample holder with sample in mounting station.

### **Sample Holder Measurement Using the VSM**

Refer to your VersaLab manual to prepare the VSM for operation.

1) In order to load your sample in the VSM, first bring the temperature up to 300 K and allow it to stabilize for a few minutes. In MultiVu, click "Open Chamber" on the VSM Install/Remove Sample Wizard dialog box. You will notice the VSM motor head move to the top "load" position (Figure 5). Remove the black cap.



Figure 5. The VSM motor in “load” position.

2) Screw the sample holder onto the sample rod and gently insert that into the VSM. The end of the rod is held in place by a set of magnets, so you will feel a tug as the rod seats itself into place (Figure 6).

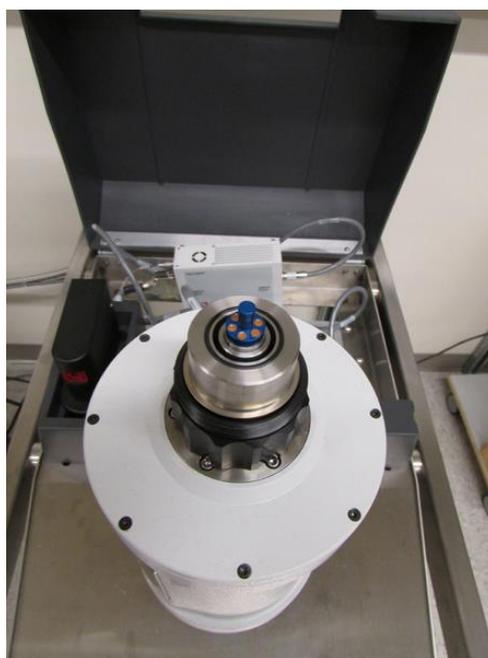


Figure 6. Inserted sample rod.

3) In MultiVu, click "Next" and then click on "Browse" to begin inputting your data file properties.

4) Clicking "Next" again will bring you to the sample centering dialog (Figure 7). Since this is only a measurement of your mounting media, we will choose a high field in order to determine what the maximum contribution might be. Feel free to set the field between 1 and 3 T (or 10,000 to 30,000 Oe).

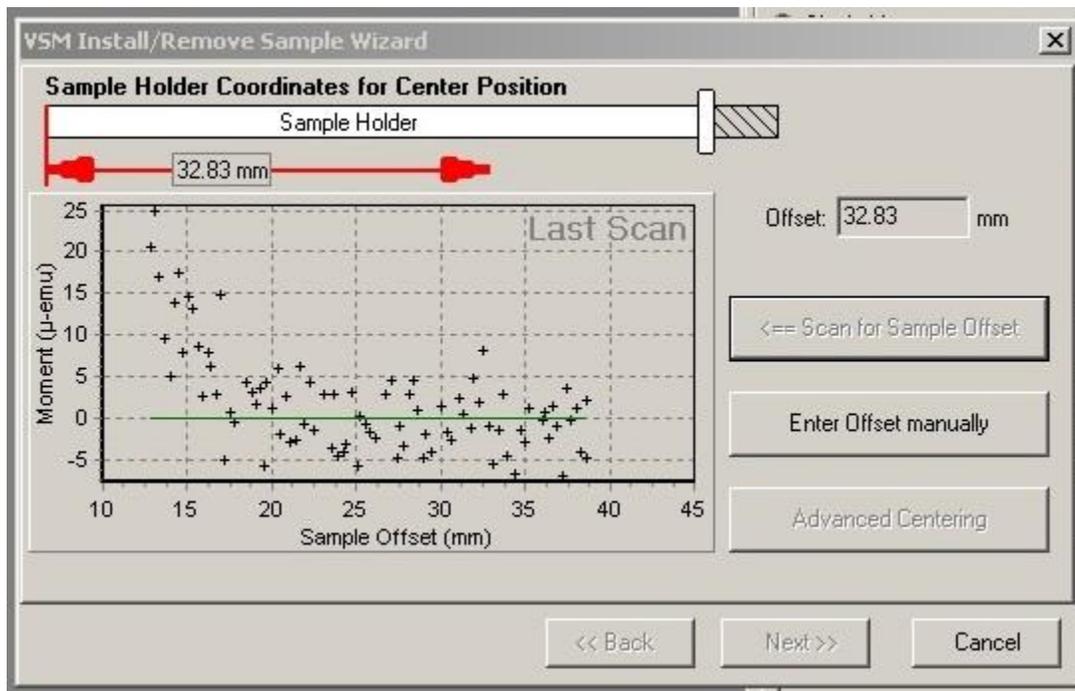


Figure 7. Sample centering scan dialog.

5) After the field settles, click "Scan for Sample Offset" and wait for the VSM. You will notice that the VSM motor drops down to the bottom "touchdown" position and then scans the holder. This will take a few seconds.

6) When the scan is complete, you may see something similar to that of Figure 8. The first dialog box will identify the center position. If you were attempting to measure something with a small magnetic moment that did not come up in the centering scan, you could enter the sample position manually. In this case, we see that the maximum background contribution of the holder at 3 T is  $2 \times 10^{-5}$  emu, which is fine.

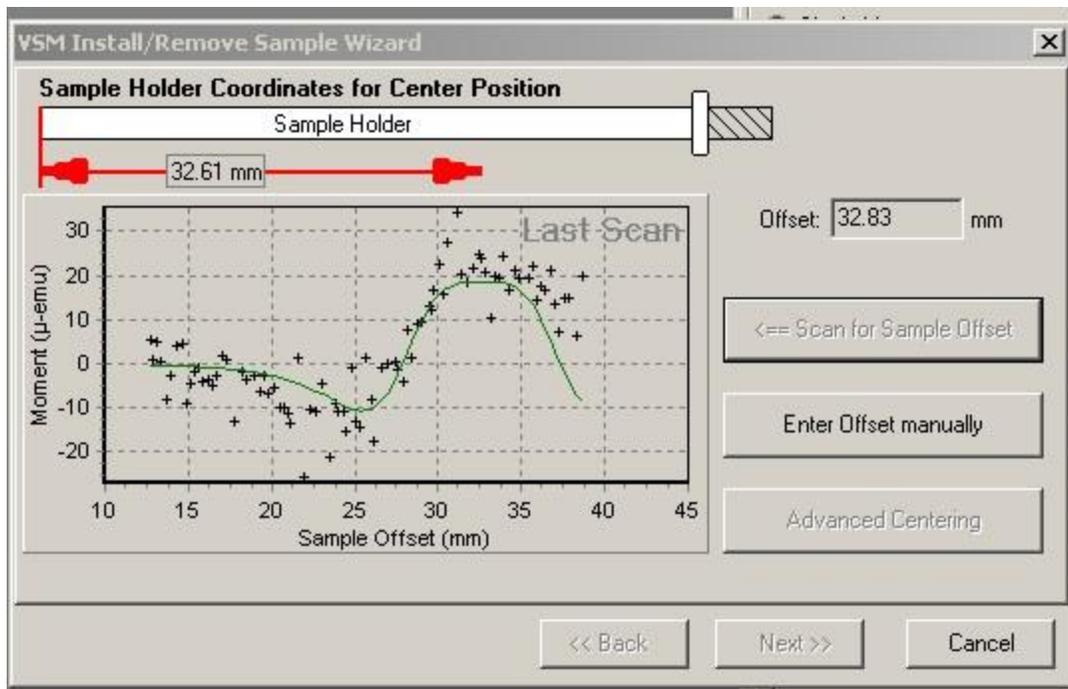


Figure 8. Sample centering result for the brass holder.

7) Ask your instructor if you should proceed to measure the Moment vs. Magnetic Field or Moment vs. Temperature properties of the sample holder. If your background contribution is large ( $10^{-3}$  or perhaps  $10^{-4}$  emu), click “Back” on the dialog box until you get back to the first window that allows you to click on “Open Chamber.” You need to either choose a new mounting medium or thoroughly clean your sample holder and mounting choice of any magnetic contamination.

If your instructor tells you to perform a scan, then click “Next” and “Close Chamber” on the next dialog. Wait for the chamber to pump down and for the “Finish” button to be active.

Write a short sequence using either the “Moment vs. Field” or “Moment vs. Temp” command, and hit “Run” when you are ready to go.

If, however, your instructor tells you to move on, then click “Back” repeatedly until you can return to the first dialog and hit “Open Chamber” to remove the sample rod.

## **Sample Mounting**

- 1) Break a small piece of YBCO sample and weigh it. A small piece with a mass of a few mg should yield a good measurement. You should also obtain a rough measurement of the volume of the sample.
- 2) Mount the YBCO 35 mm from the end of your sample holder using whatever mounting medium you previously measured. Keep in mind that the sample will be oscillated by the motor, so be sure that it is secure!
- 3) In MultiVu, click "Open Chamber" in the VSM Install/Remove Sample Wizard dialog box and load your sample.
- 4) In MultiVu, click "Next" and then click on "Browse" to begin inputting your data file properties.
- 5) When an actual sample is mounted on the holder, a good initial scan field is 1000 Oe. Set the field to this value.
- 6) When the field is stable, click "Next" to open the centering dialog window and perform a scan. There should be no problem in locating and centering the YBCO sample.
- 7) Click "Next" and check the "Extended Purge" box before you click on "Close Chamber." Extended Purge is a good idea since we will be cooling the sample and want to make sure that no water is present. When the "Finish" button becomes available, click "Finish."

## **Identifying Superconductivity Using the VSM: Moment (T)**

The first thing to do is to make sure that your YBCO is superconducting! We will perform what is called a Zero Field Cooled (ZFC) warming measurement followed by a Field Cooled (FC) cooling measurement.

When doing a ZFC measurement, you cool the sample to the lowest temperature in the absence of any magnetic field. Once that temperature has been reached, the field is turned on, and the sample's magnetization is measured as it warms up. This will illustrate what happens to a superconductor that has been placed in a magnetic field.

When doing a FC measurement, you measure the sample's magnetization as it cools in the presence of an applied magnetic field. This is important because it will show what happens as the sample transitions from the normal state above  $T_c$

to the superconducting state below  $T_C$ . The text *Superconductivity [6]* is a great reference to learn more about ZFC and FC measurements on a superconductor.

A sample sequence might look like this:

- Set Temperature 300 K
- Set Field 0 Oe
- Wait for Temperature, Field
- Set Temperature 50 K
- Wait for Temperature
- Set Field 1000 Oe
- Moment vs. Temperature, 50 K to 150 K, measure continuously, 2 K/s sweep
- Moment vs. Temperature, 150 K to 50 K, measure continuously, 2 K/s sweep
- Set Standby Mode

In the sequence above, we make sure to begin at room temperature with no applied magnetic field. Then we zero field cool down to 50 K. The field is applied once the sample is cold, and the measurement occurs as the sample warms. Measuring continuously with a 2 K/s warming rate should yield high quality data. You may adjust the sweep and averaging parameters as you see fit. The second Moment vs. Temperature scan is the FC measurement. After that, Standby will return the system to 300 K and 0 magnetic field.

It should be clear from your data what  $T_C$  is, and you will see the difference between the ZFC and FC curves.

### **Magnetic Characterization Using the VSM: Moment(H)**

Now that you have identified  $T_C$ , you can further characterize the sample's magnetic properties using the Moment vs. Field measurement. Either create a new data file from the File tab of the VSM command dialog window, or use the New Data File command in writing a sequence.

It will be important to compare a Moment vs. Field scan above and below  $T_C$ , to identify the magnetic behavior of the YBCO in both the normal and superconducting state, respectively.

A sample sequence might look like this:

- New Data File
- Set Temperature 300 K
- Set Field 0 Oe

- Wait for Temperature, Field
- Moment vs. Field, 0 Oe to 30,000 Oe, five quadrants, measure continuously, 50 Oe/s sweep
- Set Field 0 Oe, 200 Oe/s, Oscillate
- Wait for Field
- Set Temperature 50 K
- Wait for Temperature
- New Data File
- Moment vs. Field, 0 Oe to 30,000 Oe, five quadrants, measure continuously, 50 Oe/s sweep
- Set Field 0 Oe, 200 Oe/s, Oscillate
- Set Temperature 300 K

In the above sequence, we open a new data file and then perform the Moment v. Field measurement at room temperature. Again, you may choose the measurement sweep parameters that fit your time constraints. Five quadrants can be selected in the Moment vs. Field dialog box by clicking and dragging so that a white field appears over the amplitude points (Figure 9). The measurement will start at 0 Oe, ramp up to the highest field of 30,000 Oe (3 T), then down to -3 T, and then back up to 3 T (five quadrants on the graph). This will yield a complete hysteresis loop. Note that this sequence may take a while to complete depending on your sweep speed.

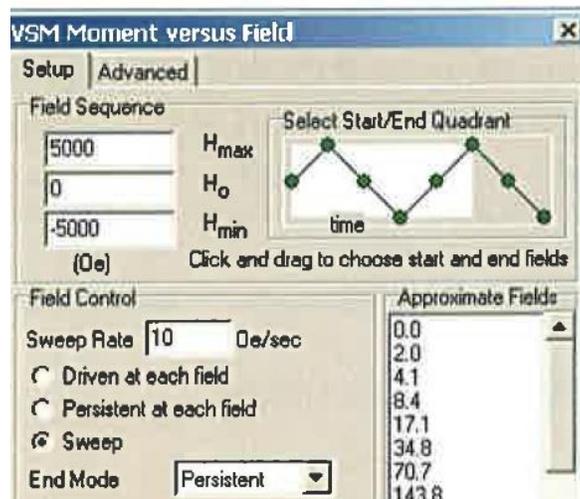


Figure 9. Moment vs. Field dialog showing the five quadrant measurement sequence. Clicking and dragging on the white box removes or adds quadrants.

Afterwards, the field is set to 0 Oe in Oscillation mode to minimize the remnant field in the VersaLab magnet.

The sequence then cools the sample in zero field and opens a new data file to obtain the Moment vs. Field measurement while it is superconducting. Can you predict what the data will look like? You may be surprised. For reference, you may take a look at [5] or [6].

Finally, the system is returned to a standby state, i.e. 300 K, 0 Oe.

### **Data and Discussion**

- 1) If you achieved superconductivity, what is your value of  $T_c$ ?
- 2) Explain the fundamental physics principles of a VSM measurement. The VSM manual is a good place to start, and you should also explore other references.
- 3) Explain the difference between your ZFC and FC result of the Moment vs. Temperature sweeps. What is physically happening to the sample during the measurement?
- 4) While in the normal state, what type of magnetic behavior does your sample exhibit? Ferromagnetic, paramagnetic, diamagnetic? How do you know? Consider examining [6] for reference.
- 5) In the superconducting state, you may have seen some irreversibility in the Moment vs. Field behavior at high magnetic fields. Search the literature and explain what is happening to the sample during each quadrant.

### **References**

- [1] For example, C. Kittel, *Introduction to Solid State Physics*, Wiley.
- [2] C. D. Graham, "High-sensitivity magnetization measurements," *J. Mater. Sci. Tech.* **16**, 97 (2000).
- [3] M.K. Wu, J.R. Ashburn, C.J. Torng, P.H. Hor, R.L. Meng, L. Gao, Z.J. Huang, Y.Q. Wang, and C.W. Chu, "Superconductivity at 93 K in a new mixed-phase Y-Ba-Cu-O compound system at ambient pressure," *Physical Review Letters* **58**, 908-910 (1987).
- [4] K. Chang, "Physicists remember when superconductors were hot," *The New York Times*, March 6, 2007.  
[http://www.nytimes.com/2007/03/06/science/06supe.html?\\_r=0](http://www.nytimes.com/2007/03/06/science/06supe.html?_r=0)
- [5] "VSM Sample Mounting Techniques," QD Application Note 1096-306,  
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- [6] C.P. Poole, H.A. Farach, and R.J. Creswick. *Superconductivity*, Chapter 2, Academic Press: San Diego, 1995.
- [7] M. McElfresh, "Fundamentals of Magnetism and Magnetic Measurements" (1994). <https://www.qdusa.com/sitedocs/appNotes/mpms/FundPrimer.pdf>