Abigail Lee, Annette Setzer
Alexander Tsirlin

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Introduction

This lab course aims to give practical introduction into several experimental techniques that are commonly used in magnetism research. Superconducting samples will be used as test cases. Upon completing this lab, you will:

- gain experience with commercial He-based cryostats and superconducting magnets, including the PPMS and MPMS systems that are commonly used in solid-state labs all over the world
- learn low-temperature measurements of magnetization and heat capacity
- learn nuclear magnetic resonance (NMR) measurements
- observe manifestations of superconductivity in practical experiments
- determine critical temperatures and critical fields of a superconductor

General introduction into superconductivity can be found in the lecture notes of the Superconductivity I module. Studying this module is strongly recommended before or along with the lab.

The lab comprises three parts:

- magnetization measurements
- heat capacity measurements
- NMR measurements

Four or five time slots should be allocated in total.

Contacts:

- *Annette Setzer*, setzer@rz.uni-leipzig.de – magnetization
- *Alexander Tsirlin*, alexander.tsirlin@uni-leipzig.de – heat capacity
- *Abigail Lee*, abigail.lee@physik.uni-leipzig.de – NMR

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1. Magnetization

1.1. Experimental goals

- Perform and analyze results of magnetization measurements on a superconducting sample
- Observe Meissner effect
- Determine the critical field(s) as a function of temperature
- Map out the temperature-field phase diagram of a superconductor

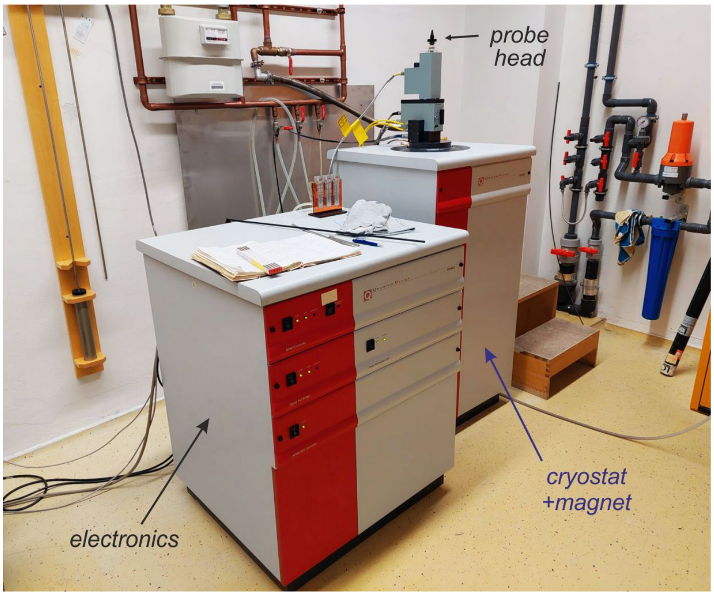
1.2. Pre-experiment tasks

In order to set up the experiment, you need to have a rough idea about the critical temperature and critical field of your sample. Additionally, you should understand how the magnetometer works and what information will be obtained from the measurement. Answer the following questions prior to your experiment:

1. How large is T_c in zero field and B_c at zero temperature for your sample? Check the literature in order to find these values. They will help you to set up the right temperature and field ranges for your measurement.
2. What magnetic response do you expect from a superconductor? Sketch χ vs. T (magnetic susceptibility vs. temperature) and M vs. H (magnetization vs. field)
3. How does magnetometer work? What is the pickup coil?
4. What is SQUID and how is it used in magnetometry? (this question in covered in lecture 8 of Superconductivity I)
5. What are the CGS units of the magnetic moment and magnetic field?
6. How large is the expected diamagnetic response of a superconductor?

Recommended Literature

- Your favorite solid-state or superconductivity textbook, for example Chapter 34 from Ashcroft and Mermin or Chapter 12 from Kittel
- W.F. Brown's Tutorial paper on Dimensions and Units and N. Carron's Babel of Units are your tools to understand the relation between the SI and CGS units
- MPMS User Manual



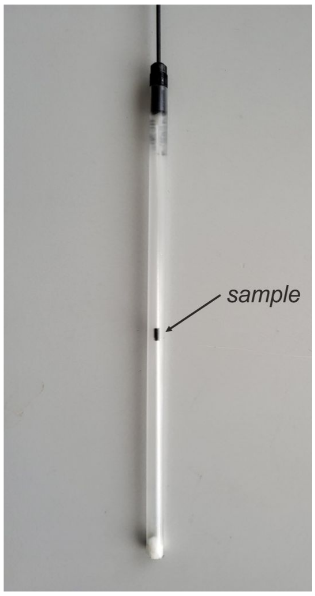


Figure 1: Left: MPMS SQUID system in room 521. Right: sample mounted inside the plastic straw and attached to the sample rod from above.

1.3. Experimental tasks

The measurements will take place in room 521 (Linnestr. 5). We will use the MPMS SQUID magnetometer from Quantum Design (MPMS stands for Magnetic Properties Measurement System) and study a sample of an elemental superconductor, Nb or Pb.

Step 1. Measure the sample weight. You should measure the weight of your sample prior to the measurement. You will need this value later for scaling magnetic moment and calculating the volume susceptibility. Superconducting magnets have small bores, so we work with small samples that typically have the mass below 100 mg. Measure the mass several times and take the average. Use tweezers to handle the sample.

Step 2. Mount the sample. Put your sample inside the drinking straw. Different mounting techniques can be used depending on the expected signal. For low-background signals quartz holders are preferable. They are very uniform, so moving this quartz holder through the coils does not generate any background. Since superconductors show a strong diamagnetic signal below T_c, it is sufficient to mount the sample in the middle of the straw with an additional piece of straw or tape. Those will create some background, but it is negligible compared to the diamagnetic response of the superconductor.

When you are done, attach your assembly to the sample rod of the magnetometer.

Step 3. Insert the sample. Make sure that MPMS is at room temperature. Vent sample space until it reaches atmospheric pressure. Carefully insert the sample and purge sample space several times. We do not want any water or air inside. They will freeze at low temperatures and may damage the instrument.

Step 4. Define sample data. Put information about the sample and sample mass into the MultiVu program. This information will be written into the data files, so that they are self-contained and can be analyzed later without any additional lab notes.

Step 5. Center the sample. Set the temperature to 5 K and wait until it's reached. Then set the magnetic field of 100 Oe and use the centering command to find the sample position and align the sample with the pickup coil. This step is crucial, as otherwise you won't measure the full magnetic moment of the sample.

Step 6. Set up the measurement program. Use the commands in the right panel of the MultiVu software to create your own measurement program ("sequence"). Remember to open a data file. You can choose to put all data into a single file and split it later, or set up individual data files for each part of your measurement.

Your measurement should include:

- M vs. T scan in a low magnetic field (25 Oe is a good choice) to observe Meissner effect. Cool the sample down in zero field and measure on heating. Use the temperature range between the base temperature of the cryostat (1.9 K) and $2T_c$. This will be the zero-field-cooled data (ZFC). Then perform a similar measurement on cooling to obtain field-cooled (FC) data.
- M vs. H scans at different temperatures in order to determine the critical field(s). Make sure to do zero-field cooling prior to each scan. It means that for each scan you should go above T_c, set field to zero, cool down to the target temperature, and start the measurement.

Important: even tiny magnetic field trapped inside the superconductor may affect its magnetic response. Superconducting magnets may have residual fields on the order of 10 Oe, which seem small but may not be benign for a superconductor. Use oscillating mode for setting field to zero. This will make sure that your magnet is as close as possible to the true zero field.

Step 7. Run the measurement. The measurement itself will take quite some time, but it will be done automatically assuming that you set up the correct program.

1.4. Report and analysis

You will get the data via e-mail after the measurement. These data should be used to create a report with the pre-experiment tasks and the following information from your experiment:

1. Demonstration of the Meissner effect: $\chi(T)$ data measured in the FC and ZFC modes. The χ values should be volume susceptibilities expressed in SI or CGS units (your choice, but please make sure to indicate which units you are using). Explain in the text how you converted experimental values of the magnetic moment into the volume susceptibility
2. How does your volume susceptibility compare to the theoretical value of χ for the Meissner effect? What could cause the deviation from the theoretical value?
3. What could be the reason for the difference between the FC and ZFC curves?
4. $M(H)$ curves measured at different temperatures
5. Critical field(s) determined at different temperatures and summarized in a $B-T$ phase diagram

2. Heat capacity

2.1. Experimental goals

- Perform and analyze results of heat capacity measurements on a superconducting sample
- Observe thermodynamic signatures of the superconducting transition
- Determine the critical field(s) as a function of temperature
- Determine the Sommerfeld coefficient
- Analyze the specific heat jump at T_c

2.2. Pre-experiment tasks

In order to set up the experiment, you need to have a rough idea about the critical temperature and critical field of your sample. Additionally, you should understand the physical principle of heat capacity measurement using the relaxation method. Answer the following questions prior to your experiment:

1. What is the flow of the heat capacity measurement? How does the relaxation method work?
2. How does the superconducting transition manifest itself in heat capacity?
3. What temperature dependence of the heat capacity do you expect in the normal state?
4. What is Sommerfeld coefficient and how is it related to the electronic structure of the metal?
5. How large is the jump in the heat capacity at the superconducting transition?

Recommended literature

- Your favorite solid-state physics or superconductivity textbook from Part 1.
- PPMS Heat Capacity Option User Manual

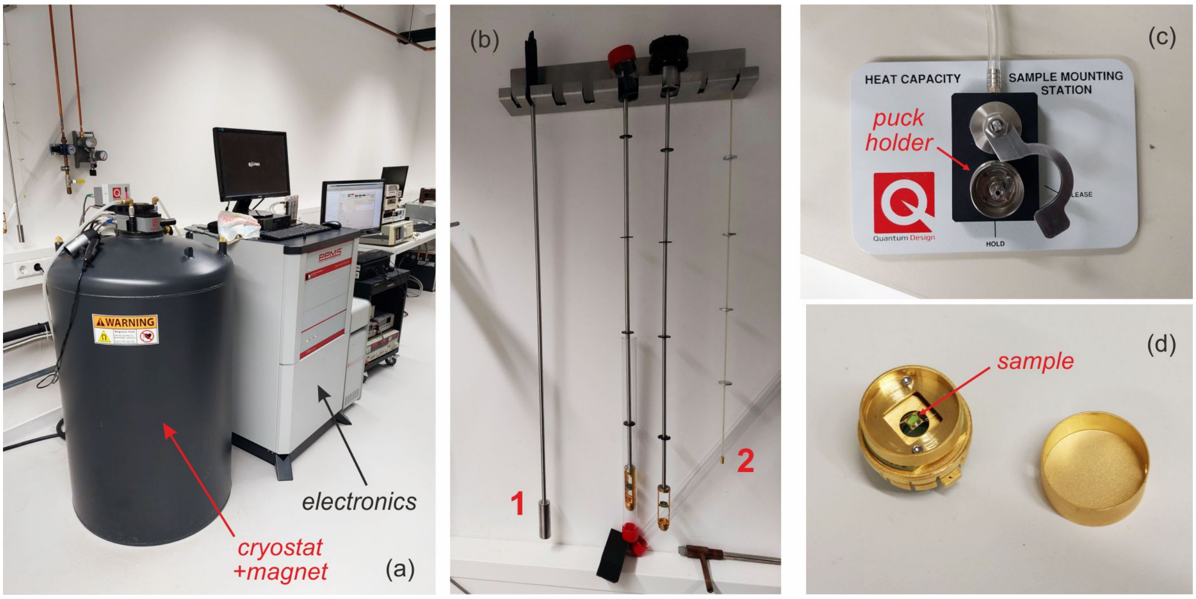


Figure 2: Left: PPMS system in room TA317. (a) General view. (b) Additional tools: puck extraction tool (1) and sample rod (2) placed on top of the heat capacity puck. (c) Sample mounting station connected to the vacuum pump. *Never touch the sample platform without placing the puck onto the mounting station and activating the pump. Otherwise, you risk breaking the wires.* (d) Inside of the heat capacity puck with the sample mounted on the platform.

2.3. Experimental tasks

The measurements will take plane in room TA317 (Technikum Analytikum building, you may know this building from the semiconductor labs). We will use the multi-purpose PPMS instrument from Quantum Design (PPMS stands for Physical Properties Measurement System) and study a sample of an elemental superconductor, Pb or Nb, same as in Part 1.

Since heat capacity experiment includes an addenda measurement, it is best to start in the morning, set up the addenda measurement, and proceed with the sample measurement after lunch.

Step 1. Prepare the addenda measurement. Heat capacity measurements requires a good thermal contact between the sample, thermometers, and heater. Special grease is used to ensure the thermal contact. However, this grease has its own heat capacity that will add up to the heat capacity of our sample. Therefore, the measurement is done in two steps. First the platform is measured with grease and without the sample. This is the so-called *addenda* that will be later subtracted from the signal measured with the sample.

Open the puck. Ensure that the platform is clean and place a small amount of grease onto the platform. Close the puck. Note down the puck number, as each puck is individually calibrated to produce the correct temperature readings.

Step 2. Set up the addenda measurement. Place the puck into the PPMS sample chamber. Insert the rod and close the chamber with the flange. Start pumping the sample chamber and activate high vacuum. While the system reaches this high vacuum (10^{-5} Torr), initialize the addenda measurement by specifying the puck number and the temperature range of your measurement. The interval between 1.9 K (base temperature) and $2T_c$ should be sufficient. This interval can be sampled by $10-15$ data points, as grease shows a smooth temperature dependence of the heat capacity.

Step 3. Run the addenda measurement. It may take $3-4$ hours, including cooling down to 10 K and warming back up to room temperature, which take $20-30$ min each.

Step 4. Prepare the sample. The sample should be in good thermal contact with the puck, so it needs at least one flat surface that can be attached to the platform. Remember to measure the sample mass beforehand, as you need it for scaling the heat capacity and calculating specific heat. The program will actually do this calculation for you, but you have to provide the molar mass, so note it down as well.

Step 5. Install the sample. Bring PPMS to room temperature and ambient pressure. Open the sample chamber and extract the puck. Repeat the operations from step 2, but now place the sample onto the grease and press it gently. Close the puck and place it into the PPMS sample chamber. Close the chamber, start pumping.

Step 6. Prepare the measurement. Initialize the sample measurement using the respective wizard. You will be asked to provide sample mass and molar mass. Make sure to choose J/mol K as units (unless you prefer different units, of course). Then you get molar specific heat directly in your measurement file.

Step 7. Set up the measurement program. Use commands in the right panel to create your measurement program ("sequence"). It should include:

- C_p vs. T measurement in zero field
- C_p vs. T measurement in the normal state, i.e., in the applied field above $B_c(0)$. You will use these data to determine the Sommerfeld coefficient
- C_p vs. T measurement at several intermediate fields in order to determine the critical field as a function of temperature

When setting up the program, consider that you need a fine temperature step, often less than 0.1 K, in order to see the transition anomaly. On the other hand, you will need less points away from the transition where heat capacity evolves in a smooth manner.

Step 8. Run the measurement. It will take a while. Heat capacity measurements are generally slow, so it is important to think about the measurement program and make sure that you measure a lot of points only in those temperature ranges where it is really necessary.

Important: the heat capacity puck contains thin wires, which are very fragile. Never touch the sample platform without placing the puck onto the mounting station and starting the pump that will hold the platform to avoid breakage of the wires. Even with the platform secured, make sure to do very gentle moves with the tweezers.

2.4. Report and analysis

You will get the data via e-mail after the measurement. These data should be used to create a report with the pre-experiment tasks and the following information from your experiment:

1. Zero-field heat capacity demonstrating the superconducting transition
2. Heat capacity measured in intermediate fields to determine $B_c(T)$. Add these points to the $B-T$ phase diagram from Part 1.

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3. Heat capacity measured in the normal state, the estimation of the Sommerfeld coefficient (γ_n)
4. Compare your measured Sommerfeld coefficient to the free-electron model. What could be the reason for the difference? Determine density of states at the Fermi level
5. Analyze the jump in the zero-field heat capacity at the superconducting transition, ΔC_p. Calculate $\Delta C_p/(\gamma_n T_c)$ and compare it to the BCS prediction.

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3. NMR

3.1. Experimental Goals

- Perform and analyze results of some basic NMR experiments on a superconducting sample ($YBa_2Cu_4O_8$).
- Identify the superconducting transition temperature, T_c, of the sample in the external magnetic field.
- Observe differences in the temperature-dependent susceptibility, χ, compared to that expected from a conventional superconductor.
- Provide an estimate for the zero-temperature superconducting gap, Δ_0.

Figure 3: View inside the lab: A 500 MHz magnet. The inserted probehead can be seen at the bottom.

3.2. Pre-experiment Tasks

In order to perform the experiments, some preliminary knowledge is needed. It is recommended that you study or review the basics of NMR in order to familiarize yourself with the terminology. You should be able to explain the most basic experiment types in NMR (FID, Hahn echo, inversion recovery). In order to easily analyze data, you will need to have Origin 7.5 installed in order to use ONMR. Answer the following questions before the experiment days.

1. Give the Zeeman Hamiltonian and explain each parameter. Give the criteria for a $\frac{\pi}{2}$ pulse.
2. What is the Knight shift (give a formula)? How does its behavior differ qualitatively between metals and superconductors? What is the Yosida function?
3. What is the gyromagnetic ratio and nuclear spin of ^{63}Cu? Does it have a quadrupole moment?
4. What are T_1 and T_2 in NMR?
5. Derive the Curie law in the high-temperature limit (Hint: start with the density matrix and assume thermal equillibrium).
6. Consider a coil containing approximately 30mg of Y-1248 in an external field of 11.7 T, at $T = 290K$, and with a coil volume of 0.5 cm^3. Estimate the magnetic flux induced in the coil by the precessing ^{63}Cu nuclei after a $\frac{\pi}{2}$ pulse. Assume the volume of the sample to be equal to that of the coil. Hint: Start with the formula derived in Question 5. The natural abundance of ^{63}Cu is 69.2%.

7. Given the above result, what do you expect for a maximum of the voltage induced in the coil if said coil is made of 10 turns? Assume a cross-sectional area of the coil of 0.5 cm^2.
8. The thermal noise voltage is given by $\sqrt{4k_BTRB_w}$, wherein T is the temperature, R is resistance, and B_w is the bandwidth. At room temperature, for a bandwidth of 1 MHz and a resistance of 50 Ω, what is the expected thermal noise voltage?
9. What is skin depth? Calculate the skin depth for metallic copper assuming an RF pulse of 132.6 MHz.

Recommended literature

1. *Principles of Magnetic Resonance* by C.P. Slichter. Chapters 1, 2, 4, and 5 may be particularly useful.
2. *Solid State NMR: Basic Principles and Practice* by D.C. Apperley, R.K. Harris, and P. Hodgkinson. Chapter 3 offers a simple view into the parameters of an NMR experiment, and Chapter 6 offers an introduction to NMR of quadrupolar nuclei.
3. *Introduction to Superconductivity* by M. Tinkham.

3.3. Experimental Tasks

IMPORTANT: Do not bring any ferromagnetic materials, food, or drink into the rooms containing magnets.

Figure 4: Inside the probe: a coil sits at the end, enclosing the sample. The two legs are connected to the hotwire and ground, respectively, while the white cylinders are adjustable capacitors.

3.3.1 Sample and Probe Preparation

The first step to any experiment is sample preparation. We will be using a single-crystal sample of Y-1248, so we first need to identify the orientation of our sample. Once the sample is ready, it is time to make a coil, and fit the coil and sample to the probehead. The sample will be placed inside the coil so that the crystal c-axis will be aligned perpendicularly to the magnetic field. Finally, since we are using a resonance circuit, we need to tune and match it to the Larmor frequency of ^{63}Cu in our 11.7 T external field. This may or may not require soldering additional circuit components.
Important are the following:

1. What is the quality factor Q of your circuit?
2. To what frequency is the circuit tuned?
3. What is the reference frequency for ^{63}Cu? What is the Knight shift for metallic copper?
4. Draw a diagram of your circuit.

Remember to record all data about your setup, including coil parameters, amount of Y-1248, & bandwidth of your circuit, etc.

3.3.2 Pulse calibration

Using a calculated value for our $\frac{\pi}{2}$ pulse power as a starting point, your next task is to perform a nutation experiment to optimize the $\frac{\pi}{2}$ condition, using the copper wire of the coil as a test. This can be done in two ways, either by changing the pulse length, but keeping the pulse power constant, or vice versa. The field inside the coil generated by the pulse is given by

$$B_1 = \sqrt{\frac{\mu_0 QP}{2\omega_0 V}}. \tag{1}$$

With the flip angle given by

$$\theta = \gamma B_1 t = \omega_1 t \tag{2}$$

it is evident that either of these two parameters can be varied in order to obtain $\theta = \frac{\pi}{2}$. The $\frac{\pi}{2}$ conditions will be obtained by fitting the nutation data.

3.3.3 Sample orientation adjustment

While we tried to align the sample with the c-axis perpendicular to the field, it is unlikely that this alignment is perfect. Therefore, we will check the orientation of the sample using the variation of the central frequency as a function of angle. The ^{63}Cu frequency will be highest in the perpendicular orientation to due quadrupole effects. After confirming the alignment of the sample, we can finally begin the temperature-dependent measurements.

3.3.4 T-dependent measurements

With the $\frac{\pi}{2}$ conditions and crystal orientation determined, it is now time to begin the temperature-dependent measurements. You will be measuring the Knight shift and T_1 of the central transition of ^{63}Cu as a function of temperature. For each temperature point, you will complete two measurements:

1. Hahn Echo: A $\frac{\pi}{2}$-τ-π-acquisition. In this experiment, the nuclear spins are flipped into the xy-plane and allowed to precess and decohere for time τ. After this, a π pulse refocuses the spins, resulting in an echo at time 2τ. Determine f_0 and linewidth from Fourier transform.
2. Inversion recovery: π-τ-$\frac{\pi}{2}$-acquisition. In this experiment, first the net magnetization is flipped to along $-z$, then given some time τ to relax, before a $\frac{\pi}{2}$ pulse is used to flip the relaxed spins back down into the xy-plane. The delay time τ between pulses is varied in order to determine T_1.
   Hint: you will extract T_1 by fitting the signal intensity as a function of τ in Origin.

3.4. Report and Analysis

After completion of the experiment, you will detail your results in a small report. This should include the pre-experiment tasks as well as the following:

1. Fitted nutation curve with the $\frac{\pi}{2}$ condition extracted from the fit.
2. Frequency $f_0(T)$ and Knight shift $K(T)$ at each temperature point, as well as linewidth.
3. Relaxation time $T_1(T)$ as extracted from the inversion recovery data.
4. Experimental SNR (How does it compare to your idealized calculations?).
5. Conclusions on the symmetry of the superconducting gap Δ_0, and an estimate of its magnitude, from the low-temperature behavior of $K(T)$.
6. *Bonus*: Did you notice a change in the resonance parameters of your circuit at T_c? What property of superconductors explains this?