Low Temperature Superconductivity

1 *Background*

The applied magnetic field at which a conductor changes from the superconducting state to normal (non-superconducting) state is known as the critical field, H_c. For many Type I superconductors (mostly pure elements), the critical field as a function of temperature behaves as sketched in Fig. 1.

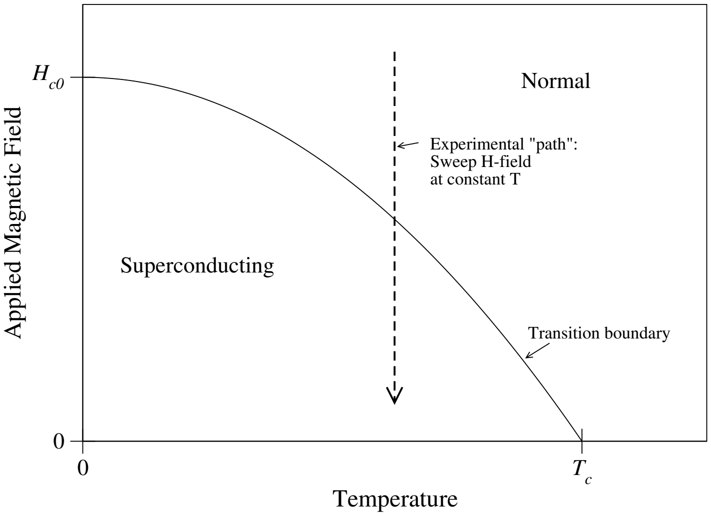

Figure 1: Typical phase diagram for a Type I superconductor. At a given temperature, as the magnetic field is decreased there is an abrupt change from the normal (non-superconducting) to the superconducting state as the field crosses the transition boundary.

This curve can be approximately represented by

$$H_c = H_{c0}\left[1 - (T/T_c)^2\right] \tag{1}$$

where T_c is the superconducting transition temperature at zero applied field, and H_{c0} is the applied field that destroys superconductivity at $T = 0$ K.

The sample in this experiment is mercury (a small sphere ≈ 6 mm in diameter). The object of the experiment is to map out the critical field as a function of temperature and fit the results to the above equation in order to find values for T_c and H_{c0} for Hg.

The experiment is carried out by immersing the Hg sample in a liquid helium (LHe) bath. Figure 2 shows a sketch of the apparatus. The applied magnetic field, generated by an electromagnet, is swept up and down while the temperature (LHe bath pressure) is kept constant, and the critical field at that temperature is recorded. By pumping on the LHe bath and lowering the vapor pressure above it to below atmospheric pressure, the bath temperature can be lowered so that H_c can be recorded at a number of different temperatures. The normal boiling point of ^{4}He (at 1 atmosphere pressure) is approximately 4.2K, which turns out to be very close to T_c for Hg. By starting to

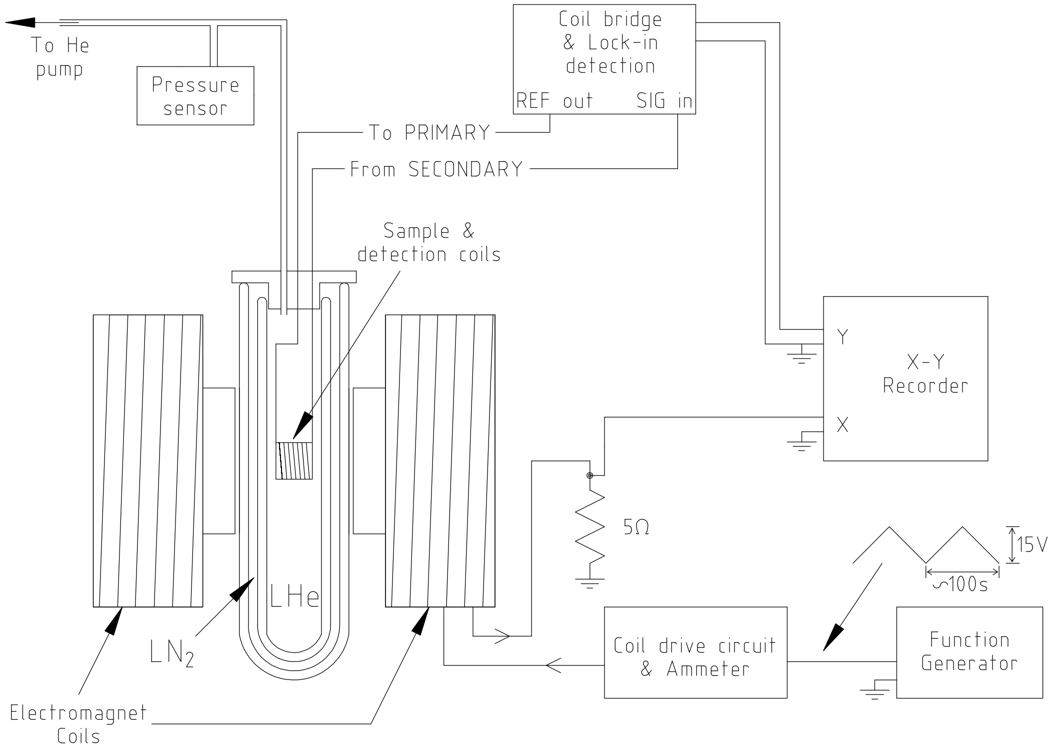

Figure 2: Schematic of the apparatus used to measure the superconducting transition in Hg.

take data at bath pressures near atmospheric pressure, and continuing down to the lowest pressure this system can achieve (≈ 6 torr), one can map out a good portion of the of the transition curve. Graphs showing the relationship between LHe bath temperature and equilibrium vapor pressure are attached to this write-up in Figs. 6–8. A detailed table is available in the lab.

When the transition from the normal to the superconducting state occurs, the Hg sample changes abruptly from a weak diamagnet (magnetic susceptibility $\approx -0.0000x$) to a perfect diamagnet (magnetic susceptibility $= -1$). The sample is located on the common axis of a primary/secondary coil pair, so that when the susceptibility changes, the mutual inductance of the coil pair changes, and the magnitude of the signal coupled from primary to secondary changes. This coil pair is part of a bridge circuit that is driven by a lock-in amplifier, and the transition in the Hg sample shows up as a change in the lock-in output. More details on this detection scheme are presented later in this write-up.

There is much to be learned by seeing this experiment in its entirety. There are vacuum systems to be checked and monitored, cryogenic fluids to be handled and transferred into the apparatus, and an elegant detection scheme that gives a very well defined signal for the transition of interest. And there is the added bonus of actually seeing liquid helium, and of observing how its behavior changes as it undergoes a transition from a normal fluid to a superfluid at a temperature just below 2.2K

2 *Procedure*

The experimental procedure can be divided into two main parts: use of the electronics and operation of the cryogenic system. If you are reading this document for the first time while doing the experiment (bad form indeed!), you will find it more efficient to skip to section 2.2 and perform steps 1 through 4, cooling down the system and zeroing the pressure sensor; then return to the instrumentation and measurement section below (Sec. 2.1) while the cryostat cools (typically 30–40 minutes).

2.1 Instrumentation and Measurements

The superconductivity experiment uses a bridge circuit plus lock-in amplifier combination to detect the transition between normal and superconducting states of the mercury sample. The detection technique is described in detail in the article by Soulen, Schooley and Evans (1973); see the references for details, but a brief description follows.

There are two identical coil pairs, each consisting of a primary and secondary, with one pair inside the "Bridge Balancing Box" at room temperature, and the second pair inside the cryostat. The mercury sample is located along the common axis of the "Cryostat" coils. Both primary coils are driven by a constant amplitude sine wave from the reference output (OSC OUT) of the lock-in amplifier.

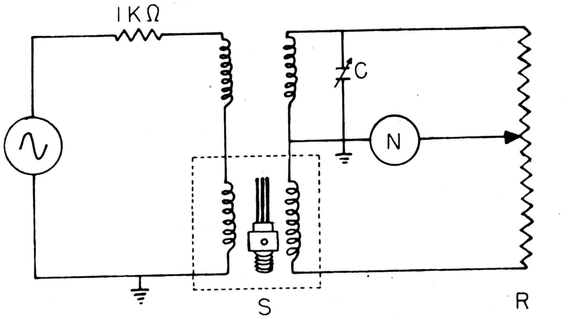

Figure 3: Bridge circuit used to detect superconducting transition. The sine wave generator is the reference output (OSC OUT) from the lock-in. The primary coils are on the left of the figure, and the secondary coils are on the right. The dotted box indicates the coil pair inside the cryostat surrounding the sample of Hg. The upper coil pair and other electronics are inside the Bridge Balancing Box. Here, N: lock-in detector, R: 10-turn pot, S: coils and sample in cryostat. (C represents an adjustable capacitor which is not used in our setup.)

Each coil pair acts like a transformer, with the magnitude of the signal coupled from primary to secondary depending on the magnetic susceptibility of the material between the two coils. There is no material, except air, between the primary and secondary of the Bridge Balancing Box coils, so the coupling between these coils and therefore the magnitude of the signal in this secondary remains constant. But when the mercury sample transitions from normal to superconducting (or vice versa), it changes from slightly diamagnetic (susceptibility ≤ 0) to a perfect diamagnet (susceptibility $= -1$), with the result that the coupling between the primary and secondary of the Cryostat coil pair changes. The magnitude of the signal in this secondary changes, the balance of the bridge circuit changes, and consequently the output of the lock-in amplifier changes.

Data are recorded with an x-y plotter, with x axis proportional to magnet current, and lock-in output on the y axis. The magnet current is swept up and down with a function generator driving a power transistor connected to the magnet coils; see Fig. 4. As the magnetic field passes through the critical value, the mercury sample transitions from one state to the other and the lock-in output changes. For this system it turns out that the normal to superconducting transition (decreasing magnetic field) gives a more well defined signal than the superconducting to normal transition (see example data), and so the former transition is the one used for data acquisition purposes. An understanding of the difference between the observed signals for the two transitions is left as an exercise for the interested student!

The following procedures may be done while the system is at room temperature, but they are most efficiently performed while the cryostat is cooling down before filling the inner dewar with liquid helium.

2.1.1 Check out of the detection electronics.

To start, set the 10-turn potentiometer in the "Bridge Balancing Box" to about 4.00; this will cause the bridge circuit to be far enough out of balance so that there is a readily observed, non-zero output from the lock-in.

Turn on the Stanford Research Systems SR830 lock-in and check that the settings are as follows:

SIGNAL INPUT:	"A/I" BNC with"A" highlighted
Couple:	DC
Ground:	GROUND
FILTERS (Notch):	Line (rejects 60 Hz noise)
TIME CONSTANT:	30 ms
Slope/Oct:	12 dB per octave
SENSITIVITY:	500 microvolts
RESERVE:	NORMAL
REFERENCE (Source):	INTERNAL
Phase:	0 degrees
Ampl:	0.150 volts
Freq:	750 Hz
Harm #:	1
Display (Ch 1):	X

"Display" on both channels should be set to "X" or "Y". Then Channel 1 display shows "X", the part of the signal that is in-phase with the reference signal, and channel 2 shows "Y", the component of the input signal 90° out of phase with respect to the reference signal.

Adjust the potentiometer to *minimize* the signal that is in-phase (X). It should read close to zero. Notice the LED bars underneath the readout. The potentiometer should be somewhere between 4.00 and 7.00 on its multi-turn scale. If you are not able to achieve an output near this, or have trouble with the lock-in settings, ask for help. However, when the sample is at 4 K, the potentiometer wil need to be readjusted to zero the output.

2.1.2 Setup and use of the X-Y recorder.

MODE:	mV/IN
X-axis:	2 mV/IN X100 (with the vernier adjust at full scale)
Y-axis:	20 mV/IN X100, but to get more sweeps/page, use 50 mV/IN
Y-axis zero:	This knob allows you to shift the pen vertically about 0.5 inches for the next trace, when using the 50 mV/IN X100 scale.

Turn the potentiometer knob on the bridge control box, and verify that the pen carriage goes up and down. If you do not see this motion, you may need to adjust the sensitivity of the Y-axis input, the zero-adjust knob, or make sure that the zero-adjust is on “OPER”.

Push 11 x 17 sheets of paper under the left-hand stop and fix the paper in place either by using the electronic “hold-down” switch or tape down both sides of the sheet with masking tape, depending on which recorder you are using.

After each down-sweep, lift the pen (use the switch) and wait until the pen reaches the right hand edge of the paper before putting pen down for the next trace.

2.1.3 Check out of the magnet current sweep circuit.

Check that the power supply, function generator, power transistor, current meter and magnet are wired as shown in the Fig. 4. When you are satisfied that the wiring is correct, set the voltage control on the power supply to about 20 volts (indicator on outer black knob straight up), and set the current control to 0 amps (outer black knob fully counterclockwise). Turn on the power supply and the function generator, and turn the current control knob to about 2 amps (indicator on outer black knob straight up). Check that the current sweeps between 0 amps and about 1.1 amps maximum, and if necessary, adjust the amplitude and offset of the function generator to achieve these limits.

2.2 Plumbing and Cryogenics

1. Pump out the inner dewar jacket. The inner dewar is the one that holds the LHe bath (refer to Fig. 2). It has a vacuum space, usually called a jacket, between the inner and outer walls, and any gas in this space must be pumped out before liquid helium is transferred so as to minimize the heat conduction between the LHe bath and its surroundings. The latent heat of LHe is very small; 0.7 watts of heat flowing into a LHe bath will boil away 1 liter in an hour.

With the apparatus at room temperature, start the smaller of the two mechanical pumps (plug in power cord—one with purple tape near the plug) and opening the toggle valve (flip handle up) on the pump-out line. After pumping on the dewar jacket 10–15 minutes (pump should no longer be making gurgling noises) close the toggle valve (flip lever down). Leave the small mechanical pump running for the duration of the experiment.

2. Zero the pressure sensor. This is done by pumping out the LHe bath space. Start the larger mechanical pump (one with orange tape near the plug) and open valve #1. Make sure the rubber stopper is in place in the transfer port (bright green band on it) on top of the cryostat. After a minute or two the pump should no longer be gurgling and the pressure in the LHe bath space will be well below 1 mm Hg. Zero the pressure sensor output with the OFFSET control on the Pressure

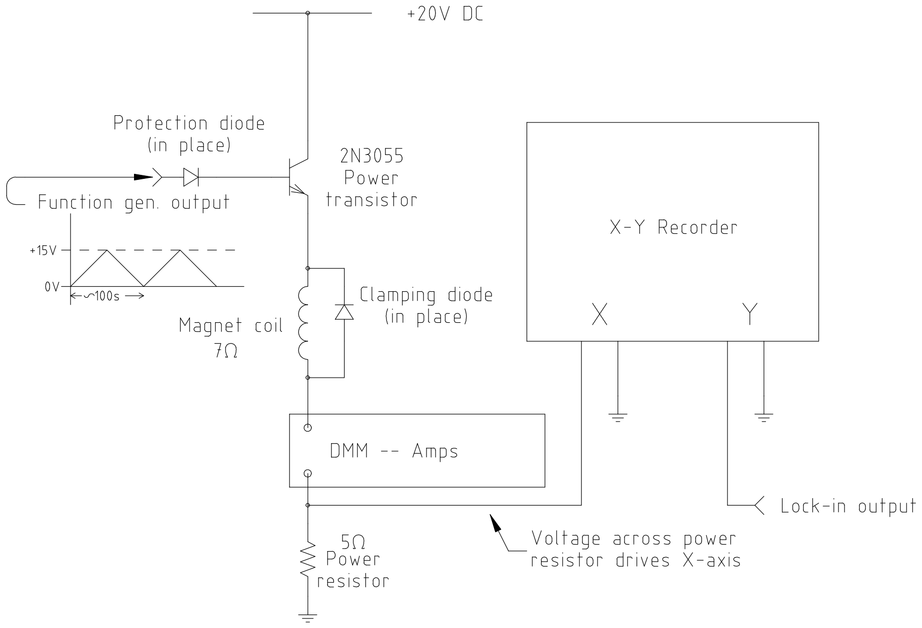

Figure 4: Schematic of the circuit used to drive the magnet coils. Compare this to Fig. 2. Question to ponder: Why is 20V used to power the circuit? Why not, say, 40V?

Sensor Amplifier.

After zeroing the sensor, it is necessary to adjust the gain so that the pressure sensor reading matches the local atmospheric pressure, which can be read from the mercury barometer mounted on the wall near the experiment. Close valve #1 and open valve #4 to allow atmospheric gas back into the LHe bath space. With the bath space at atmospheric pressure, adjust the GAIN control on the Pressure Sensor Amplifier so that the reading matches the pressure indicated by the barometer. For example, if the barometer pressure is 758 mm Hg, the gain should be adjusted for a reading of 7.58 volts output. After adjusting the gain, close valve #4 and pump the LHe bath space out again by opening valve #1.

The pressure sensor is a SenSym Model SCX15AN. This sensor measures absolute pressure, and has a typical deviation from linearity of ±0.1% of full scale output over its output range (0 to atmospheric pressure) and a worst case deviation from linearity of ±0.5% of full scale output over this range.

3. Introduce He gas from the cylinder into the LHe bath space. Consult the professor, lab manager or TA for assistance with this procedure. First connect the rubber hose from the regulator (below valve C) to the copper tubing above valve #4. Next open valve A on top of the cylinder (counterclockwise) and then slowly turn valve B on the regulator clockwise to adjust the delivery pressure (gauge 2) until the needle indicates about 2 PSI (black mark on glass). Open valve C and slowly open valve #4. Watch the pressure and when it nears atmospheric pressure, close valve #4 and remove the rubber stopper from the transfer port. Re-open valve #4 and flow He gas through the dewar for 10–15 seconds before closing valve #4 and replacing the stopper in the transfer port.

This procedure is called a "flush," as it flushes out most of the atmospheric gases that would otherwise freeze inside the dewar when it is filled with LHe.

Caution: Any contact of liquid nitrogen and bare skin can cause severe injury. Please handle the liquid nitrogen very carefully.

4. Fill the outer dewar with liquid nitrogen (LN2). This dewar will need to be topped off several times while you are doing the experiment—it should be kept at least 2/3 full of LN2 for the duration of the experiment.

5. Wait for apparatus to cool. The apparatus should cool down with LN2 for at least half an hour before LHe is transferred. While it is cooling down, configure and adjust the electronics so that the magnetic field is swept appropriately (see notes on instrumentation and schematic of experiment in section 2.1). It is important to have all the electronics operating properly before transferring LHe, since the LHe only lasts a few hours.

6. Top off He gas. Just before transferring LHe, add any He gas needed to bring the LHe bath space up to atmospheric pressure. Open valve #4 and then close it when the pressure reaches 1 atmosphere.

7. Transfer LHe. The professor, lab manager and/or TA will do this, as some practice is required to efficiently transfer liquid helium. After the transfer is complete, the valve between the mechanical pump and the LHe dewar jacket (smaller pump) can be turned off.

8. Adjust the He pressure. The temperature of the LHe bath is controlled by adjusting the vapor pressure above the bath; this is done by adjusting the pumping speed with valves #1 (coarse adjust) and #1a (fine adjust). More pumping speed (valves open wider) implies a lower vapor pressure and a lower LHe bath temperature. For all but the lowest pressures, pumping just through valve #1a will provide adequate pumping speed. The lowest pressure that can be achieved with both valves open is approximately 6 torr.

To go from one pressure to a lower one, slightly increase the opening of the valve you are pumping through and wait for the pressure to equilibrate. Subsequent minor adjustment of the valve may be necessary to achieve or stay at the pressure of interest. What is important is not to achieve a rock steady pressure, but to note what the pressure is exactly when the sample transitions from normal to superconducting. Some coordination within the group is required for this.

Graphs and a table are provided to make the conversion from pressure to LHe bath temperature; see Figs. 6–8 at the end of this write-up.

9. Take data. After LHe is transferred to the experiment, you can begin taking data straight away. Start taking data at higher temperatures (pressures) and then go to lower temperatures (pressures). The reason to move in this direction is that once you have cooled the LHe bath down, it warms up very slowly, and you may not be able to get desired data at higher temperatures before the LHe is completely gone.

You will likely need more than one sheet of graph paper to record your data. When you start a new sheet, make sure that it is fixed securely in the same position as previous sheets so that the X axis calibration taken on the final sheet applies to all data sheets. (One can be very clever and get all the data on one sheet, but this is not necessary.) The first indication of a transition from the normal-to-superconducting state will occur at the extreme left side of your graph paper and will correspond to about half an atmosphere in the dewar. Near the lowest pressure of about 5 Torr, the transition will be near the right-hand edge of the paper. You can estimate from the roughly 400 Torr of pressure change, that you may wish to make steps of about 25 Torr at a time until you get near the lowest pressure.

A record of the normal-to-superconducting transition for at least 10 to 20 different temperatures, roughly equally spaced over the accessible temperature range, should suffice to nicely map out the phase boundary.

10. Calibrate the X axis. After taking data on the transition temperature vs. magnetic field it is necessary to calibrate the X axis of your graph. For this calibration to be valid the X axis settings (zero and gain) on the x-y plotter must be the same for data collection and for the calibration procedure. To perform the calibration:

1. Turn off the function generator (Krohn-hite 2000) and the power supply (HP 6290A).
2. Remove the red and orange wires from the power transistor. (It is attached to a black heat sink with three banana-plug connectors on top.)
3. Connect the red and orange wires together. (One plugs into the other.)
4. Turn on the power supply and adjust the current to near maximum at 1.1 amps. Put the pen down and move the Y-zero adjust knob up and down to make a tic mark. Then, label the current found on the ammeter for that tic mark (be careful not to change any X-axis settings).
5. Repeat this last step for currents near 1.0 amp, 0.9 amps, . . . , 0.0 amps.

Note: it is important to decrease the current monotonically so as to avoid any hysteresis effects which could invalidate your calibration. See Fig. 5.

If you have several sheets of data, the calibration can be done on one sheet and then transferred to the other sheets at your convenience. To convert magnet current to magnetic field values, consult Fig. 5. Note that there are separate data for increasing and decreasing current which illustrates the hysteresis in magnetization of the electromagnet's iron pole pieces. The curve fits indicate the hysteresis by the quadratic term, which shows positive curvature for increasing current and negative curvature for decreasing current.

11. Shut down the experiment. When you are done with the experiment the professor, lab manager, and/or TA will help with the shut-down procedure, as follows. Close valves #1 and #1a, and flow He gas from the cylinder through valve #4 into the LHe bath space to bring it back up to atmospheric pressure. Replace the rubber stopper in the transfer port with the grooved rubber stopper (orange dot on top) so the He gas can escape from the bath space as the LHe boils off. Turn off (unplug) the mechanical pumps. Do not open the toggle valve on the LHe dewar jacket pump-out line. Turn off all the electronics.

Graphs of the vapor pressure of liquid helium follow. You need to use these to convert your pressure data to temperature. Tables of these data are available in the lab.

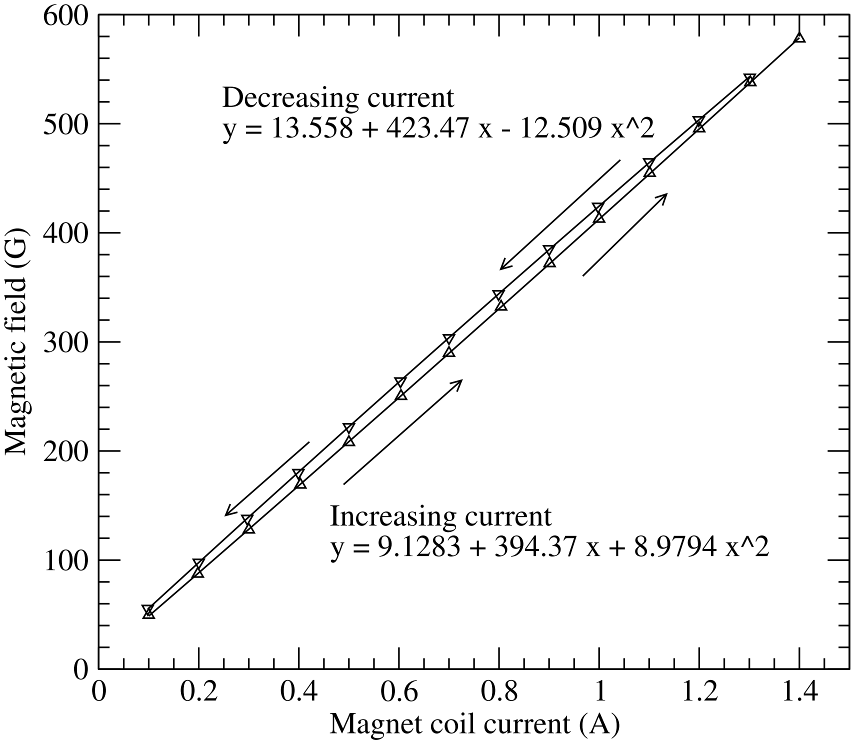

Figure 5: Relationship between magnet coil current and the measured magnetic field of the electromagnet. Note the different data for increasing and decreasing current, as well as the slight hysteresis loop.

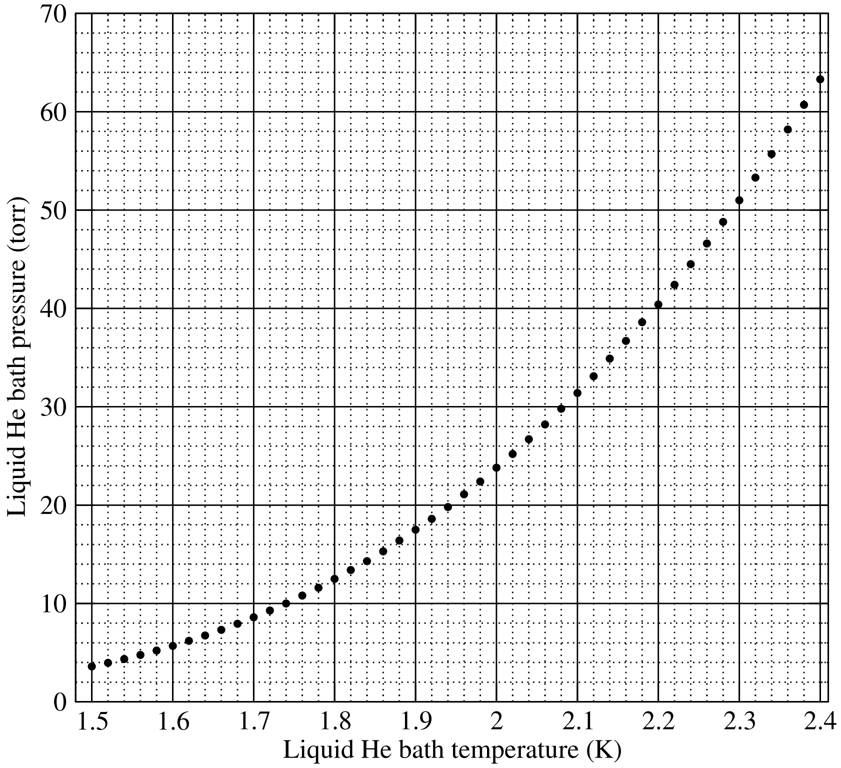

Figure 6: Vapor pressure of liquid helium between 1.5 and 2.4 K.

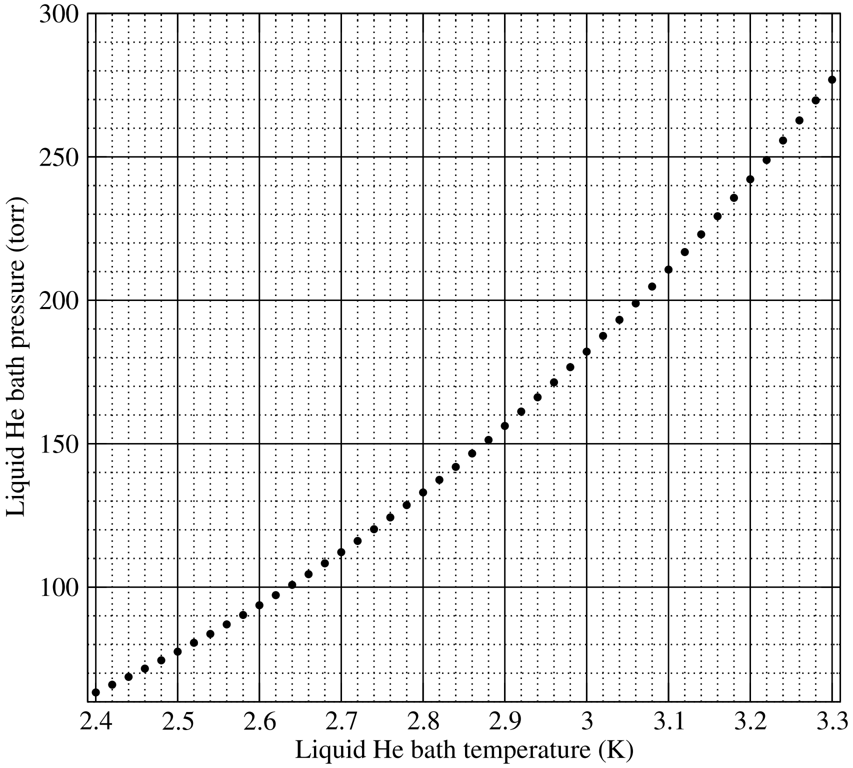

Figure 7: Vapor pressure of liquid helium between 2.4 and 3.3 K.

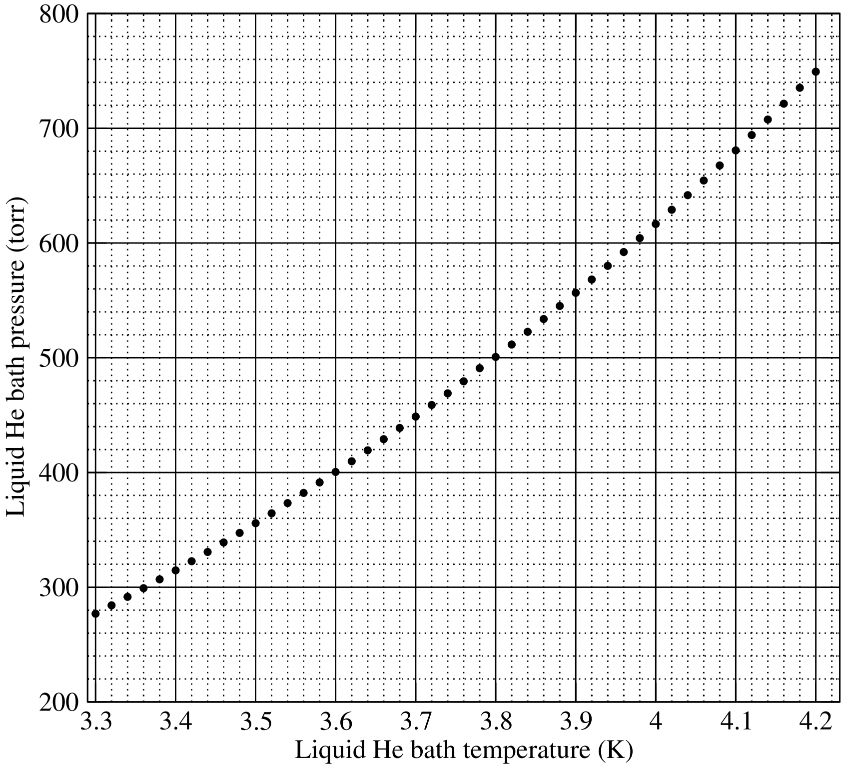

Figure 8: Vapor pressure of liquid helium between 3.3 and 4.2 K.

References

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Soulen, R. J., J. F. Schooley, and G. A. Evans, Jr., Simple instrumentation for the inductive detection of superconductivity, *Rev. Sci. Instrum. 44,* 1537–1539, 1973.

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