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DEVELOPMENT OF OPERATIONS MANUAL AND RESTORATION OF LOW-GRAVITY
SIMULATOR MAGNET

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This thesis is dedicated to all my friends and family.

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ABSTRACT

The simulation and study of fluid properties and dynamics in a microgravity environment on the Earth's surface is of great interest due to the high costs associated with conducting research in space. Cryogenic fluids like liquid helium, hydrogen, and oxygen have numerous applications in space travel and research. An understanding of their sloshing motion and general dynamics in microgravity is extremely valuable for maintaining proper orientation of spacecraft and proper cooling. The ability to study fluids with no surfaces in contact with their container allows greater flexibility in the study such fluid dynamics, and enables deeper research into quantum turbulence to be conducted. Simulating this microgravity environment on the earth is highly desirable, and may be achieved by using drop towers, acoustic levitation, laser levitation, zero-g planes, and superconducting magnets. The stable magnetic levitation of diamagnetic fluids against the pull of gravity on the earth's surface may be obtained by generating a strong magnetic field and specific magnetic field gradient. Stability of a levitated liquid drop requires a potential minimum to hold the drop along a central axis, with a field magnitude increasing with increasing radial displacement. The NHMFL cryogenics lab obtained a unique cryostat containing such a magnet, which was used for hydrogen and helium levitation, but which was in disrepair. The Low Gravity Simulator Magnet arrived with dozens of leaks and no documentation explaining its structure, functions, or the procedures necessary to use it experimentally. The purpose of this thesis work has been to develop a complete understanding of this cryostat's structures and functions, as well as the refinement of procedures to properly cool it down and safely operate its magnet. A user manual has been produced to fully document the experimental setup and all necessary procedures. Because this cryostat was received in such bad condition, much time was also committed to the identification of all its mechanical failures, vacuum and gas leaks, and their full repair. Additionally, all supporting pumping systems and gas handling systems were designed, constructed, and documented to facilitate proper and safe operation.

CHAPTER 1

INTRODUCTION

The Cryogenics Lab at the National High Magnetic Field Laboratory received an older experimental facility consisting of a cryostat with a 16.3 T superconducting magnet used for the magnetic levitation of cryogenic fluids. The facility was received in poor condition and with no documentation, schematics, or labels detailing its usage. Before performing any of the many possible studies opened up by this facility, it was imperative to fully document the structures and functions of the cryostat, develop its procedures, generate a detailed manual, and repair/replace all broken or leaking components. While the idea of levitation may seem distant from rigorous scientific inquiry, the study of fluid dynamics and properties without the interference of the earth's gravity is quite valuable. Spacecraft may experience alteration of their heading when fluids in their fuel tanks slosh about and understanding the damping motion of such sloshing will prove increasingly useful as efforts to expand manned missions to mars and elsewhere gain momentum. Studies of quantum effects on macroscopic scales are often impossible or greatly hindered by the limitations of the experimental apparatus being used. This facility opens the door to studying the formation and decay of quantum vortices in a rotating drop of superfluid liquid helium. Quantum turbulence experiments with quantum vortices involving towed grids or thermal counterflow, for example, do not present the same degree of experimental freedom as an apparatus where the liquid being studied is not bound to interact with its container.

Numerous types of research may be performed using this facility, as discussed in Chapter 2. In Chapter 5, a discussion may be found regarding testing conducted for the ability to cool down correctly as well as to safely operate the magnet at low currents. Full levitation experiments have not yet been performed, but will soon be possible as a result of these developments.

1.1 Goals

When the cryostat was delivered to the Cryogenics lab, no manuals, instructions, or labels/diagrams were included. This device was essentially a black box and the first priority of this work was to conduct a full and thorough examination to determine its structures and functions. The top flange of the cryostat features a wide array of blank pipes, electrical connections, level

detectors, valves, and pipes with compression fittings, each connected to the various internal systems. Several of these were visibly damaged or outright broken from the start. The internals of the cryostat are considerably more complex and required a great deal of analysis to fully understand. Communication with the researchers who used this facility in the past offered some insight, however no documentation could be found to assist this setup. After a large amount of study, most of the cryostat was understood. The manuals for the 16 T superconducting magnet were ultimately found by prior groups and sent to this lab. They contained useful information regarding the specifications of various components within the magnet. In completing this first objective by examining the cryostat, several personnel were trained both in the fundamentals of cryogenic and vacuum systems, and in the proper use of this facility. A full analysis of the components can be found in Chapter 3.

Once each component was studied and understood, the next goal was to locate and modify, repair, or replace all the damaged and malfunctioning components. Extensive leak checking used a mass-spectrometer helium leak detector, and several differing methodologies to identify problems. Leaky and damaged components were repaired to restore to functionality. These and other methods are detailed in Chapter 4.

To demonstrate that the cryostat was functional, a test was devised to cooldown and examine the magnet. A new positive lead was needed to safely operate this device. Testing safe operation included attaining a superconducting state for the magnet, testing the resistance of the magnet and new lead at liquid helium temperatures, and analyzing the quench protections for safety. By testing the magnet at low currents, it was shown that using the cryostat body as the negative lead was still functional. Cooling the magnet, and testing its electrical properties from room temperature to 4.2 K showed that a superconducting state may have been successfully achieved by a proper cooldown procedure and these conditions were maintained for over an hour. Long-term experimentation is vital studying steady-state effects and phenomena with longer durations of effect. The results of this test are presented in Chapter 5. Through the refinement of the cooldown processes, several leaks were generated and these required additional work to repair.

The ultimate goal for this work was to create a user operations manual detailing all the structures, procedures, and functions of this cryostat. It includes detailed schematics and more specific explanations and instructions than some of the discussions here. A large percentage of this manuscript is present in the manual, which is given in Appendix A.

CHAPTER 2

BACKGROUND

Scientific research conducted on earth is subject to the gravitational pull of the planet's mass. At sea level, all matter experiences a downward acceleration of about 9.81 m/s^2 . This ubiquitous gravitational potential well affects both experimental parameters and material properties. For example, experiments in fluid dynamics must deal with the effects of the liquid being bound to its container, and the effects of the viscous boundary layer caused by contact between the fluid flow and these solid surfaces. To study pure free flow effects, a microgravity environment, as in low earth orbit, is desirable yet expensive [1]. This cryogenics lab obtained a facility with a superconducting magnet to permit a deeper study of cryogenics used in spacecraft as fuels [2] and as coolants in space-based observational facilities and satellites. Understanding microgravity fluid dynamics can drastically enhance efforts to conduct research, or even aid the exploration of other planets and astronomical bodies [3].

Microgravity research is very costly and difficult to access. However, several methods of simulating microgravity environments have been developed. The cryostat obtained by this laboratory utilizes magnetic levitation via a superconducting solenoid. Other methods have significant disadvantages by comparison. Examples of such methods and drawbacks include: acoustic levitation and the need for a surrounding gaseous medium [4], laser levitation and heating issues [5], and drop towers with very short experimental time frames (~ 4 seconds or less) [6]. In contrast, a superconducting solenoid, can maintain a microgravity environment almost indefinitely if continuously supplied with cooling and vacuum insulation.

2.1 Magnetic Levitation

A magnetic dipole moment is induced in an object with a non-zero magnetic susceptibility when placed in a magnetic field \vec{B} ; the force per unit volume experienced is expressed as:

$$\vec{F}_m = \frac{\chi}{\mu} (\vec{B} \cdot \nabla) \vec{B} = \frac{1}{2} \frac{\chi}{\mu} \nabla B^2, \quad (1)$$

where χ is the magnetic susceptibility of a material and μ is its magnetic permeability [7]. A diamagnetic material with negative susceptibility will be repelled from a region of high field. The

upward force of the magnetic field in a levitation experiment must balance the downward gravitational force, as seen below:

$$B \frac{dB}{dz} \geq \frac{\mu \rho g}{|\chi|}, \quad (2)$$

where g is the acceleration of gravity on the earth's surface, and ρ is the density of the material. Magnetic levitation has been achieved for materials such as graphite ($\mu \rho g / |\chi| = 1.3 \text{ T}^2/\text{cm}$) [8], bismuth ($\mu \rho g / |\chi| = 7.31 \text{ T}^2/\text{cm}$) [9], and water ($\mu \rho g / |\chi| = 13.6 \text{ T}^2/\text{cm}$) [10]. For liquid helium, a high value of $|B(dB/dz)|$ of about $20.7 \text{ T}^2/\text{cm}$ is needed [5]. Such a high field-gradient product is produced in this facility's specially-designed 16.3 T superconducting magnet [11]. The coil geometry was designed to enhance this gradient. The calculated potential energy is shown below in Figure 1.

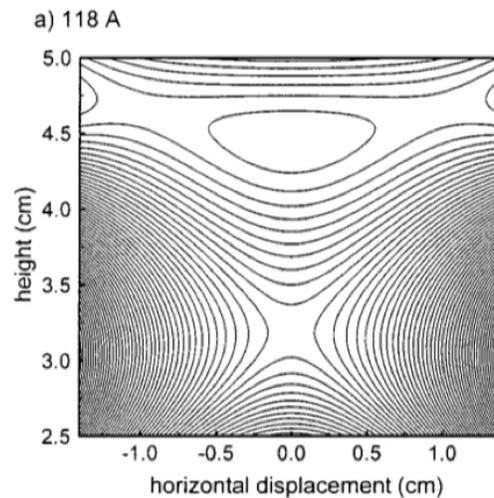


Figure 1: The calculated potential energy contours in the cell at full field strength [5].

In order to achieve stable levitation, a potential minimum in which the liquid may levitate must be attained. To achieve stability in the bore of a solenoid magnet, a combination of gravitational and magnetic fields can produce a stable levitation point [5]. Without a region of stability, a drop cannot be studied in a controlled manner. The desired potential minimum is located on the central axis of this magnet, and has an increasing magnetic field magnitude with radial displacement from the center. The potential well for this magnet is positioned with no radial displacement and at a height of 4.5 cm from the center of the magnet.

2.2 Research Significance

In past experiments using this cryostat, levitation experiments were conducted with liquid helium [5] and liquid hydrogen [12]. These studies largely focused on shape oscillations in levitated drops [13] and the non-coalescence of drops in apparent contact [5]. The experimental cell in this cryostat contains four electrodes (not currently set up for use). These were used to excite and study surface vibrations in a charged drop of liquid helium. The non-coalescence observed by previous groups was largely explained by a thin layer of evaporated vapor preventing contact between two drops in a manner similar to the Leidenfrost effect.

This cryostat was designed for the specific purpose of levitating cryogenic liquids. Thus, many phenomena may be studied by a fully-functioning apparatus. Using the existing electrodes in the cell or mechanical methods like a piezoelectric transducer, it is possible to induce sloshing motion of a fluid in the simulated microgravity environment to study the hydrodynamics and fluid dynamics of these cryogens. When such liquids in microgravity slosh within a container, they can significantly affect the heading of spacecraft [14].

The ability to study such fluid dynamics in freely floating microgravity environments at temperatures below the lambda transition (about 2.17 K [15]) from normal He I to superfluid He II, will allow unique experiments visualizing quantum vortices [16] to be conducted. Rotating a free superfluid drop has not been performed before and the ability to study and visualize the quantum vortices in a rotating drop permits many interesting inquiries. Visualization experiments are already conducted in this laboratory, however combining such research with this magnet will allow for a greater flexibility in the study of quantum turbulence, and may prove significant in explaining diverse subjects from the observed rotational phenomena in the emissions of neutron stars [17], to exploring cosmic strings and the early universe [18].

CHAPTER 3

EXPERIMENTAL SETUP

The cryogenic facility used for this research is a model 16CNDT Janis Dewar [19] containing a large liquid helium bath surrounded by an annular liquid nitrogen jacket. The core of the facility is the experimental cell. Inside this isolated chamber, cryogenic liquids may be levitated and manipulated for various studies at extremely low temperatures. To simulate microgravity, the cell is suspended in the bore of a superconducting magnet that possesses a geometry designed around the magnetic susceptibility of helium. To hold cryogenic liquids, the cell must be kept cold and insulated. Additionally, levitation requires a means to balance the earth's gravity. Here, a superconducting magnet provides the needed upward lift on an induced magnetic dipole from a 16.3 T magnetic field with a carefully designed field gradient to enhance stability in the levitated fluid.

As expected, superconductivity for this magnet can only be achieved at extremely low temperatures. In this facility, liquid helium must continually be supplied as a coolant from a large liquid helium bath to several systems. If the correct temperature is not maintained, the magnet will quench and rapidly boil all cryogenics nearby. Cooling the system down requires ports to introduce liquid cryogenics to their respective baths and the bath and magnet each have their own fill port.

Cooling down is, however, just half the work in levitating a liquid in the cell. Keeping the cell and superconducting magnet cold requires effective thermal insulation. A diffusion pump is therefore connected to maintain a pressure low enough to curtail conductive and convective heat transfer. Radiation can transit a vacuum and in this facility, it is reduced by several concentric radiation shields located around these chambers, which reflect and conduct this heat to a cold reservoir: the liquid nitrogen jacket.

While liquid nitrogen is effective at precooling the system, and liquid helium will reduce the temperature to 4.2 K, superfluid He II, with its remarkably high effective thermal conductivity and other interesting properties, cannot be studied or used to cool the magnet to its operating temperature without cooling below the lambda transition of 2.17 K. Evaporative cooling is an effective means to attain lower temperatures in liquid helium, so pumping ports are needed to facilitate a pressure reduction. Reducing the magnet's temperature allows it to carry more current and remain cold. A vapor-cooled current lead connects through the main bath into the magnet and

uses liquid helium and cold vapor to reduce its electrical resistance and the resultant joule heating. Cooling the cell below 4.2 K requires additional components, notably the 1K pot, attached to an internal heat exchanger. This pot, like the magnet, is continuously filled by the main helium bath and has a pumping port to allow for evaporative cooling of its liquid helium to lower temperatures.

Once the thermal and magnetic field requirements have been met, levitating a fluid requires the fluid be introduced into the cell, where the magnetic field provides lift and balances the earth's gravity. To that end, several fill lines are present and connect to the cell. Only one is required to introduce fluid, but different lines have slightly different properties. One fill line is vacuum insulated from the bath and can be used for hydrogen levitation. Two more each consist of a thin tube with a 0.01 cm diameter wire protruding from its end. One uses a stainless steel tube and stainless steel wire and the other has a glass tube and glass fiber to avoid electric discharges when using the electrodes. Ultra-high purity helium gas is supplied from a gas cylinder to a given fill line. That gas transits the main helium bath, heat exchanges, and condenses into the liquid phase. After exiting the bath and entering the cryostat's vacuum space, the liquid is cooled to lower temperatures via a heat exchanger on the 1K pot. Finally, the capillaries carrying the liquid enter the cell and deposit the liquid into the magnetic trap. If the drop temperature is not low enough, the cell has a pumping line to facilitate additional evaporative cooling.

Of course, levitating the liquid in the cell is not particularly useful without a means to study the levitated drop. To allow observations, the cell has top and bottom windows, an optical window on the bottom of the cryostat, and a side window that points to a mirror above the cell. These allow photographs to be illuminated and taken of the experiment as it occurs. In the event that some liquid makes its way to the bottom of the cell, past the magnetic levitation trap, it may remain in a pool. Such a pool can obscure the imaging of a levitated drop, so a second, smaller pumping line is connected to the bottom of the cell and allows this pool to be removed as needed.

Maintaining all of these components in their operating states requires not just vacuum pumps, but instrumentation including thermometers, heaters, liquid helium level meters, and pressure gages. All internal instrumentation passes through one of several ports into the vacuum space, which allow the wires to carry these signals (through non-conductive ceramic seals) while also maintaining the vacuum insulation inside. To maintain the necessary pressures in each component, and supply the gas to make the drop, specific gas handling and vacuum systems were designed and constructed around the cryostat. All issues with these components and

modifications/repairs to them are detailed in Chapter 4. The summaries given here are in-depth, but complete details and procedures are located in the user manual in Appendix A.

3.1 Initial Observations

3.1.1 Piping Structure

Before operation of this facility, it was important to generate numerous schematics for this system. These are detailed in Section 3.2. Because it is not possible to disassemble the main helium bath without significant disassembly time, a few non-invasive methods were employed. The understanding of the cryostat grew with each new test. Ultimately, both methods detailed below created a robust understanding these connections.

The simplest method to see if two pipes are connected is to pressurize or pull a vacuum on one (with a gauge pressure difference well within the range of the sensors used), and observe what happens in another. This method does have its disadvantages. For example, an attempt to pull a vacuum on the magnet can would be difficult if the helium bath were not also in vacuum as the two are connected through a fill line and originally, a valve. So to start, all ports were closed and sealed. An older photo of the top flange is shown for reference in Figure 2.



Figure 2: An older photo of the top flange of the cryostat is shown. Note the visible damage to some parts.

The system being investigated was connected to a rotary roughing pump and reduced to a low pressure. Next, proceeding from port to port, an attempt was made to open each port. If it

could be easily opened, there was likely no connection to the chamber in vacuum. If the plug in that port was being pulled down by the vacuum, it would be very difficult to remove. A slight dislodging of the plug would also cause an audible stream of air to rush into the evacuated chamber. This method was not entirely successful due to the large number of leaks in the various components.

Inside, everything is located below the base of the main helium bath. Many capped flanges protrude downward as shown in Figure 3, and other pipes connect to specific systems inside. Visually tracing the location of these lines from the top, to directly below the bath inside, gives an initial guess, but no certainty regarding which port on top corresponded to each of these internal caps and pipes. To confirm these visual guesses, an easy method was utilized where liquid nitrogen was poured into each open pipe on top of the cryostat, and whichever cap or pipe formed frost on its surface was clearly connected to that port. Still, some structures required a great deal of thought and discussion before their purposes were understood.



Figure 3: The internal top flange supports several downward facing capped pipes and pipe connections. Most caps correspond to spare ports on the outer top plate of the cryostat

3.1.2 Electrical Structure

Originally three electrical ports were located on top of the cryostat, a broken one with two 32-pin connectors, another with a working 32-pin connector, and a port containing four BNC connectors for the electrodes in the cell. Inside, the electrodes had a clear path to the cell. The other electrical connections differed significantly. The broken port was connected to several banks of pins and wires. The working port connected to a bundle of cords, each terminating in two sockets.

By testing each pin on top alongside a socket inside, using the continuity feature of a multimeter, it was straightforward to generate a mapping of pins to sockets. Nearly all of the pins

on the broken port were severed inside, so the port and its cables were removed entirely. The working port's connections were determined with the exception of six pins. Testing in the magnet can showed that four of those six pins were hard-wired to the magnet can's helium level meter, and while the connections of the last two pins appear unconnected. The switch heater was also allocated two pins. Because the number of sockets was limited, not all sensors or heaters were connected for operation. More electrical ports are planned for future addition.

3.2 Cryostat Structure

The cryostat can be described in terms of its internal and external components, both electrical and piping-related. Electrical systems include the cell electrodes, thermometers, heaters, level meters, and magnet leads. External meters and computer software monitor these readings. The vacuum and gas piping system required extensive planning and design based on limited vacuum pumps and available pipes and valves for construction. A general overview is presented prior to the details and illustrations of individual systems in the following sections.

3.2.1 Vacuum, Gas, and Liquid Piping

This cryostat contains many inter-connected systems. The following subsections offer detailed schematics and descriptions of these structures and their respective functions. To control which systems receive gas for purging, back pressurizing, or simply drop formation, an existing gas board was modified to suit this facility's needs. Finally, a system of vacuum lines to each vacuum pump was designed and constructed to accompany this experiment and fulfill each component's intended functions.

3.2.1.1 Top Plate. The top plate of the cryostat features every port, wire, or pipe connecting to the interior of the cryostat. Figure 4 shows a detailed presentation of each port, and each system is color-coded to indicate which pipes are connected directly or indirectly to each other. Specific mentions of each port are written in parenthesis in this text, and refer to this figure. Ports for the bath and magnet systems are labeled using the upper case Latin alphabet. Ports for the cell are labeled using Arabic numerals. Ports for the vacuum space are labeled using Roman numerals, and finally, ports for the isolated spares, and for the ^3He system are labeled using the lower case Latin alphabet. The labels with an asterisk show the ports that connect to the helium bath internally.

The main helium bath has a port for liquid nitrogen and helium transfers (A), one for pumping and He recovery (B), as well as a port vertically above the socket in the base of the bath for the positive magnet lead (F). It has a pumping line (H*) for the 1K pot (also used for back pressurizing during liquid nitrogen precooling), a port for the liquid helium level meter (E), and a small spare port that is not currently used (G). Lastly, two concentric pipes with a unique “T-shaped” compression fitting are connected and have been allocated for the exhaust and purging of the bath on the center pipe (C), and a connection to the gas board on the outer pipe (D). These may not have been their original purposes, however.

It should be noted that while the 1K pot is listed as part of the main helium bath, it is by merit a different system on its own. It is continuously filled internally by the bath via a capillary, and its pumping port (H*) is what is visible externally. It is included in blue for simplicity as the distinction is less important than it is for the similarly connected magnet system, in pink.

The isolated ports include two pipes connected below the bath to each other by a copper “U-shaped” pipe (d) and (c), a spare pipe (a), the thermal switch (b) capable of about 80° of rotational freedom (eventually severed and capped off inside due to a leak), as well as a gold-colored port with a removable rod inside (e). The purpose of port (e) remains unknown. It does not connect to any internal systems, and the cap it corresponds to inside showed no hidden features when opened and examined.

The vacuum space contains nearly every electrical port for instrumentation inside the cryostat. Currently all instrumentation passes through a port in the front of the top plate (III) with a newer 32 pin electrical connector. The original old electrical port (IV) was eventually sealed off externally and internally. Likewise, the port for the cell’s electrodes (I) was plugged externally. A small port (II) with a very thin wire inside (whose purpose remains unknown) was plugged externally. The pumping port for the vacuum space (VI) is a blank pipe connected to a valve on the cryostat and is connected to the molecular diffusion pump for maintaining the vacuum insulation. A viewport is located in the center of the top plate and offers an additional channel for imaging, illuminating, or a laser into the cell. The mirror and its support structure need to be removed for such use. It features an O-ring, and is sealed under a removable plate.

The cell features four ports used for filling different gasses for levitation, which connect to a system of capillaries and heat exchangers internally (Figure 5). Two fill lines have small colored flags taped where they enter the cell. The line with a red flag (6) is the line used to levitate the

helium drop and connects to a glass capillary inside the cell. The yellow line (5) is a backup fill for the same purpose but is not currently used. A fill line for the bottom of the cell (4) can be used for injecting liquid below the magnetic trap, if desired. This capillary connects internally to the vacuum line for the bottom of the cell (2), which can be used for removing liquid from the bottom of the cell if it obscures the imaging of a levitated drop.

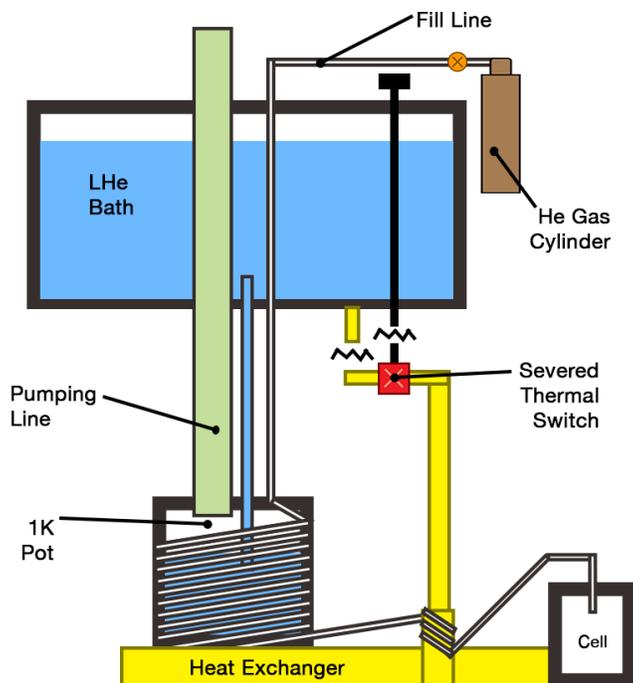


Figure 5: The liquefaction and route of ultra-high purity (UHP) He gas on the way to the cell is shown. Room-temperature gas leaves the cylinder and liquefies on transit through the helium bath. It cools below 4.2 K by heat exchanging with the 1K pot, and may be evaporatively cooled within the cell.

A fill line for hydrogen levitation (3) is currently capped off externally and disconnected from the cell, but could be re-opened for future experiments. It is a double pipe that is insulated from the helium bath, as hydrogen will freeze at liquid helium temperatures. Inside, it connects to a small capsule-like structure used for condensing the hydrogen gas into its liquid phase prior to injection into the cell. The pressure in the cell may be controlled by the vacuum port (1), which allows for evaporative cooling of a drop in the magnetic trap, the removal of any gas in the cell, as well as for obtaining a measure of the saturated vapor pressure in the cell. This pressure reading

is more accurate than the thermometers in the cryostat for determining the exact temperature of the drop. The saturated vapor line of helium is well documented in the literature [20].

The magnet can has a fill port (L*) for transferring liquid helium, and a pumping port (J*) for controlling and measuring the saturated vapor pressure and, for evaporative cooling of the temperature of the liquid helium in the magnet. This port was crushed and damaged as will be discussed in the next chapter, and was repaired using a KF-25 flange and steel pipe.

Originally, there was a small “T-shaped” valve (K*) with around 60° of rotational freedom, which allowed the continuous flow of liquid helium from the main helium bath to be adjusted. This valve was leaking and ultimately needed to be replaced by a simple connection to allow a continuous fill. Externally, a copper plug seals its connection.

Finally, the ^3He system has a large port for pumping on the $\frac{1}{2}\text{K}$ pot (f), and a small pipe (g) used for filling the $\frac{1}{2}\text{K}$ pot with ^3He . Due to the high cost of ^3He , these ports are not used in this facility and are simply capped or connected to a valve for evacuating and leaving in vacuum. The heat exchanger was modified slightly inside to thermally link the 1K pot where the $\frac{1}{2}\text{K}$ pot was previously used (it was thermally connected to the cell and fill lines).

3.2.1.2 Internal Structures. Descriptions of the internal components of the cryostat inherently rely on discussion of the external systems they connect to, as these systems are the user’s interface. As such, Figure 6 includes the relevant internal structures as well as the systems they connect to externally. Not shown are all electrical ports, the hydrogen fill, all spare ports, the helium bath level meter, magnet leads, and the back port to the bath (G). Colors are used in Figure 6 for legibility to differentiate piping systems and do not correspond to the coloring scheme used for Figure 4.

The green pumping lines connect to the red lines only where the magnet recovery is indicated, to the white lines only where the two back-pressure lines are indicated, and to the purple lines only on the top, far right near the roughing pump. The purple pumping lines connect to the red lines only to the right of the valve on the bath pumping line. All other intersections of overlapping external lines of different colors should be interpreted as entirely independent.

Individual systems are fully detailed with tables explaining their components. Schematics illustrating their place both physically in the cryostat, and in their role in this facility are available in Appendix A.

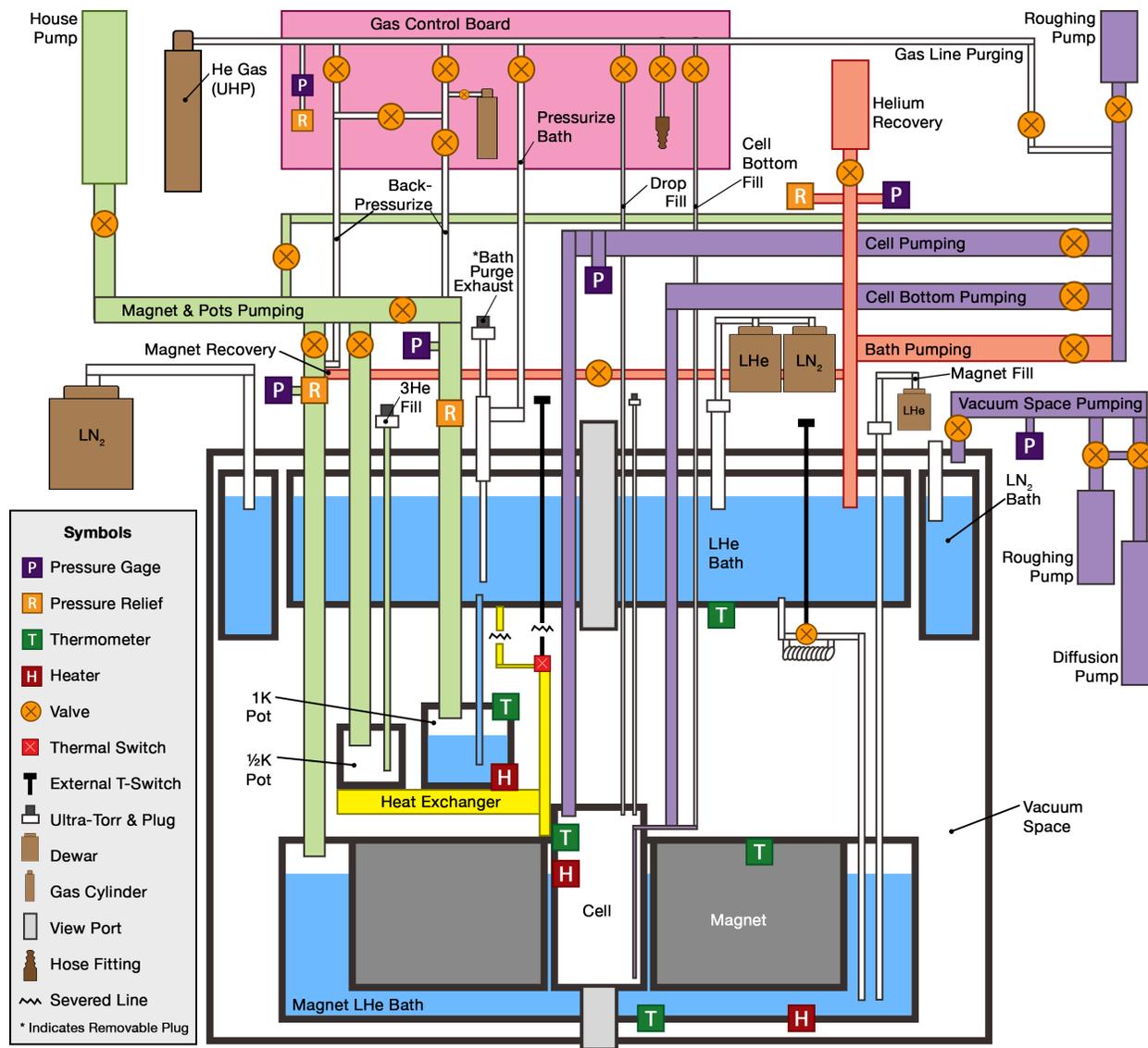


Figure 6: The entire cryostat piping system is shown. The unused ports and those used for instrumentation and the magnet lead are not shown here.

3.2.1.3 Gas Board. The various systems of the cryostat have several different He gas requirements above and beyond the actual levitated drop formation. To these ends, an existing gas handling board was modified and installed onto the cryostat. For example, before liquid cryogens are introduced into the cryostat, it must be purged with helium gas 3-4 times. This process involves pressurizing a given system to around 800-1000 Torr and then evacuating it to 1 Torr or lower, and then repeating both. Helium molecules are monatomic, and thus smaller than other gasses [15] This allows them to flush air and water vapor out of the various internal chambers and surfaces.

Additionally, back pressurizing the 1K pot and magnet can during precooling, and pressurizing the bath while removing liquid nitrogen, each require helium gas at different pressures.

A simple schematic of the gas board with labels indicating which system is controlled by which valve is given in Figure 7. Two UHP helium gas cylinders are connected as indicated and allow two different pressures to be maintained. The 1K pot and magnet can be supplied gas independently of the fill lines and bath for back-pressurizing, which is critical during the pressurized removal of liquid nitrogen from the bath. This gas board features a connection to the roughing pump system so that it can be purged of air when new gas cylinders are connected or when it is exposed to the room for any reason. Finally, a spare line is available with a hose fitting for venting the bath of evaporated liquid nitrogen during precooling and for a convenient helium source during leak checking. The pressure relief valve on the board helps ensure that the pressure from the gas cylinders never exceeds a safe value. Each cylinder has a pressure regulator, so the pressure relief valves are mainly for redundancy and help in the event of vacuum insulation loss and rapid cryogen boil-off. An analog pressure gage is included in the board to observe the pressure being supplied.

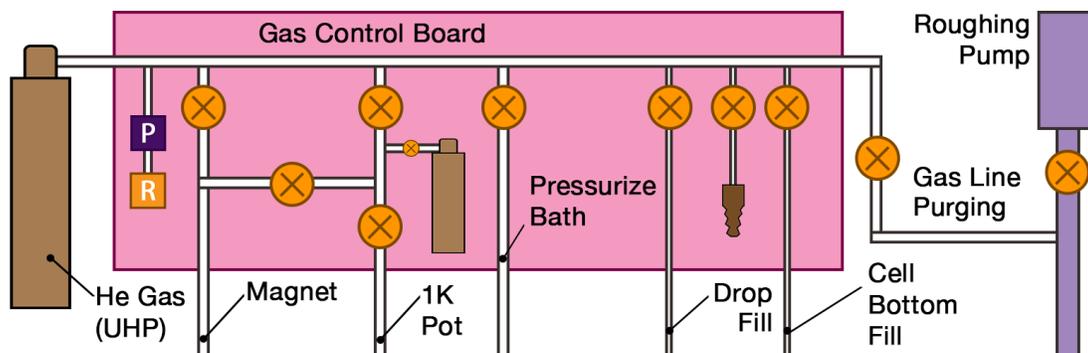


Figure 7: A simple schematic of the gas handling board.

3.2.1.4 External Vacuum System. To control the vacuum and pumping rate in the various systems of the cryostat, it was necessary to design and construct two vacuum boards, each located on either side of the cryostat. Because the main vacuum jacket has a valve built-in, it is connected only to the diffusion. A great deal of thought was put into how to best optimize the limited resources available. The cryogenics lab has a large vacuum rotary pump referred to as the “house pump”, which serves as the end connection for the left board. The right board is connected to a

smaller roughing pump and has a connection to the previously discussed gas handling board, largely for purging.

All relevant connections and valves are included in Figure 6 during the discussion of the internal structure of the cryostat, earlier in this sub-section. Photographs of these boards are given in Figure 8. Diagrams for both of these systems as they appear physically on the cryostat frame are shown in Figure 9 and Figure 10, below. Both diagrams are taped on the front wooden panels of the cryostat's frame to help the user understand and control each system.

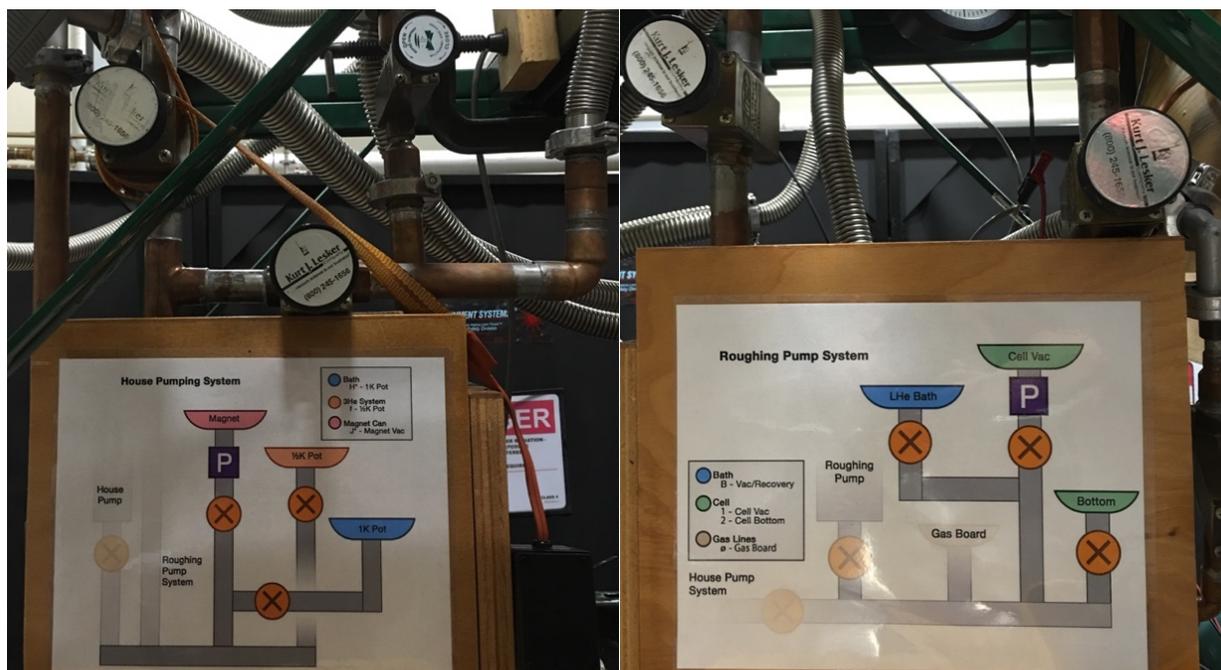


Figure 8: Two photos of the vacuum control boards.

The construction of these systems was achieved largely by repurposing portions of the piping structures supplied with the cryostat, and soldering them into functional systems with fittings and connections compatible with this lab's equipment. After the pipes were soldered and assembled it was necessary to leak check the final assembly. They are mounted vertically into wooden support structures while still being easily removable for future modifications.

When designing two different pumping systems, the main concern was to avoid a connection between the cell and other systems. A connection between the bath and the magnet/1K pot was also undesirable during liquid nitrogen precooling. Because the only time a vacuum is

pulled on the main helium bath is when purging and when removing liquid nitrogen, it made sense to connect it to the same pump as the cell. The cell can be pumped on to reduce the drop temperature, and the roughing pump on the right system can attain pressures below 100-50 mTorr.

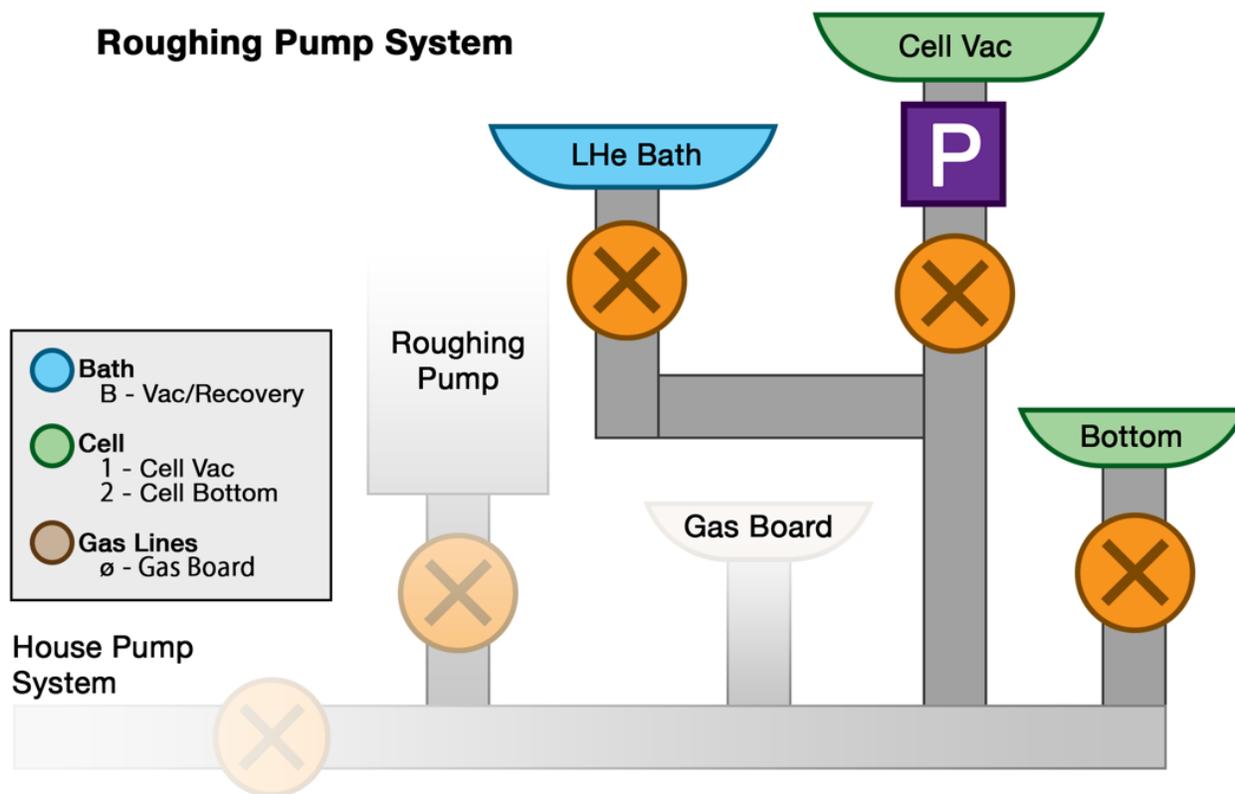


Figure 9: The control board is shown for the roughing pump system (right board). Portions which appear faded are not visible to the end user unless viewed from behind the cryostat.

The magnet and 1K pot similarly use evaporative cooling below 4.2 K by the house pump, however they need to not interfere with the pumping in the cell as the cell's temperature must be precisely controlled during measurements.

The ½K pot is also on this board but only so that it may be evacuated to avoid a thermal link with the ambient environment by gas conduction and convection. Because the house pump is shared between all of the experiments in the cryogenics lab, it is not always available. It is required for magnet operation, but when necessary, the two boards may be pumped by a single roughing pump by means of a back pass-through connection and valve between the two pumping systems.

After the initial purging has been completed, the house pump is not needed until the end of liquid nitrogen precooling and the start of the liquid helium transfers.

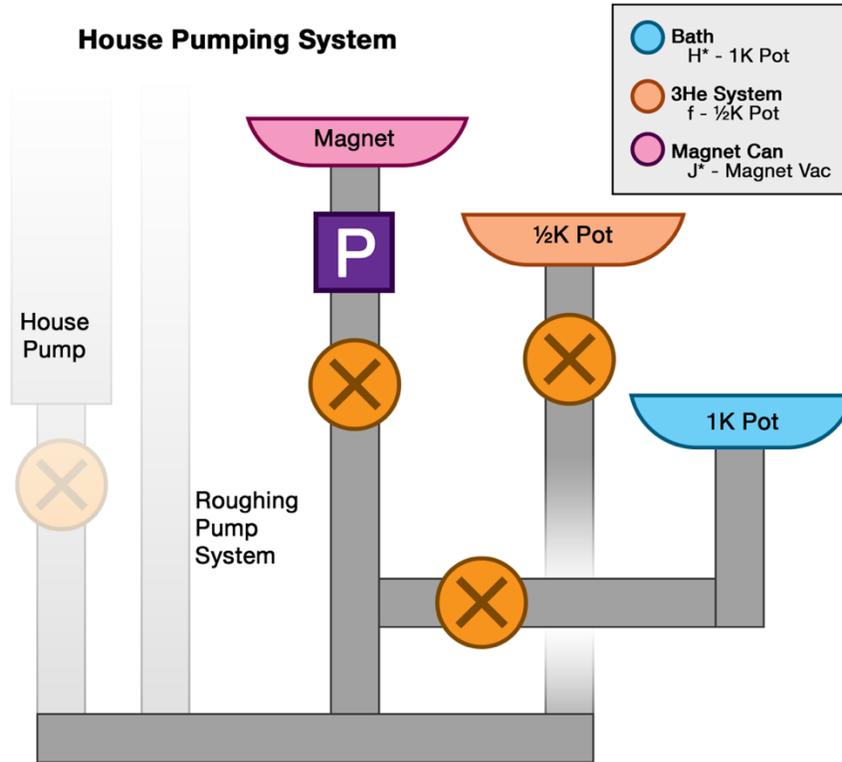


Figure 10: The control board is shown for the house pump system (left board). Portions which appear faded are not visible to the end user unless viewed from behind the cryostat.

3.2.2 General Electric Systems

The instrumentation and electrical connections within the cryostat pass through four ports on the top of the cryostat. The temperature sensors, heaters, and level meter within the magnet can all pass through the new electrical port (III) and are detailed in the following subsections. The main helium bath arrived with a liquid level meter, passing through a dedicated port (E), however it was damaged and subsequently replaced. The superconducting magnet utilizes one positive lead on port (F), and is vapor and liquid cooled by the bath. The negative lead is the cryostat body and connects to the power supply via a copper ring with braided wire connected to the top flange. The cell's electrodes (I) consist of four wires insulated with plastic tubing. They connect to several fittings prior to terminating in a five pin (only four pins are active) connector internally. They plug into a port extending from the cell and connect to four electrodes within the cell. While currently

disconnected, reconnecting them requires simply soldering the existing wires to a new external connector. For consistency, all external instrumentation uses gray wires for the negative wire.

3.2.2.1 Thermometers. There exist two electrical means of determining the temperature of a given system of the cryostat. The first is through the use of one of several Lakeshore Cernox™ [21] thermometers attached to important systems, and the second, for He II, is by measuring the saturated vapor pressure as read by a pressure sensor. The temperature sensors used in this facility were included when it was received. Due to the limited number of electrical pins available on the single functional port (III), only five Temperature sensors are currently in use. They connect to the cell, magnet (internally), 1K pot, gold heat exchanger, and the external plate of the magnet can. The locations of these sensors is indicated in Figure 6.

Each temperature sensor interfaces with the instrumentation by 4 wires as depicted in Figure 11 and each wire was soldered to an adapter with 4 sockets. These adapters each connect to two of the numerous double socket cables, numbered 411-423. For each sensor, two wires supply the current to power the sensor, and two wires measure the voltage across the sensor.

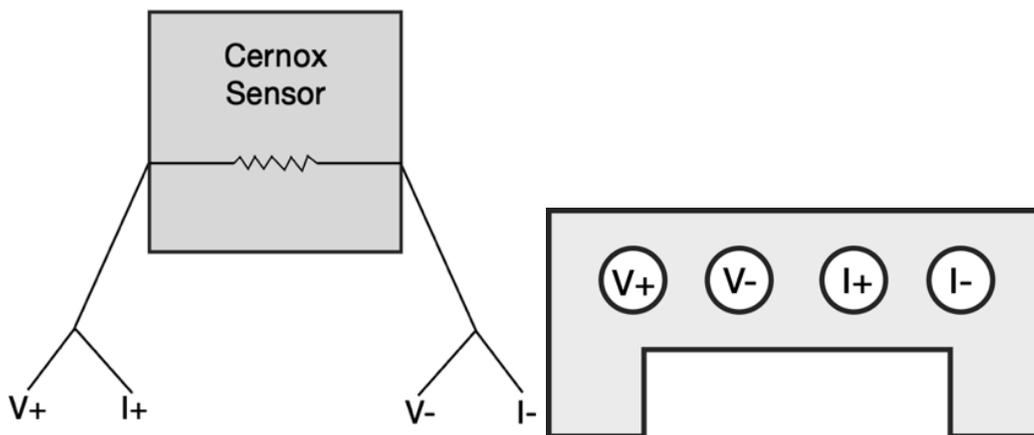


Figure 11: The wiring of a Cernox™ thermometer is shown on the left, and the orientation of these wires as soldered on their internal connectors is on the right. The two downward protrusions represent the shape of the plastic on the internal connectors.

Table 1 gives the current wiring for voltage pairs on each sensor, and Table 2 gives the wiring for the currents. The channel column refers to the number of each connection to the Keithley. The sensor column indicates the number on a small label attached to each sensor. The location column indicates the present location of the sensor in the cryostat. The socket column

denotes which double socket internal connector is attached to two of the four pins (as described on the right side of Figure 11). The pins column shows which external pins correspond to these wires, and the +/- column shows which of those pins is the positive and which is the negative.

Table 1: The pin mapping for voltage pairs on each sensor.

Channel	Sensor	Location	Socket	Pins	+/-
1	37	Cell	422	KL	+/-
2	34	1K Pot	423	PR	+/-
3	35	Gold Plate	421	JH	-+
4	33	Top Magnet	416	ab	-+
5	32	Bottom Mag	417	cd	-+

Table 2: The pin mapping for current pairs on each sensor.

Channel	Sensor	Location	Socket	Pins	+/-
1	37	Cell	414	XW	-+
2	34	1K Pot	418	CB	+/-
3	35	Gold Plate	412	gf	+/-
4	33	Top Magnet	413	UV	+/-
5	32	Bottom Mag	415	ZY	+/-

Additionally, the 32-pin diagram on (III) is given in Figure 12. In most resistive temperature sensors, as the temperature decreases in the sensor, the resistance increases. A calibration is used to correlate a given resistance with a specific temperature. A temperature calibration was performed for each sensor where a calibrated sensor was used to correlate the temperature to the resistance of each sensor from roughly 300 K to around 1.2 K. Four overall graphs of four sections of temperature range are given in Figure 13. Care should be taken to avoid excess current in the sensors as this can cause self-heating from the sensor. Typically, 10 μ A was applied until the helium transfers, then this was changed to 3 μ A and ultimately 1 μ A.

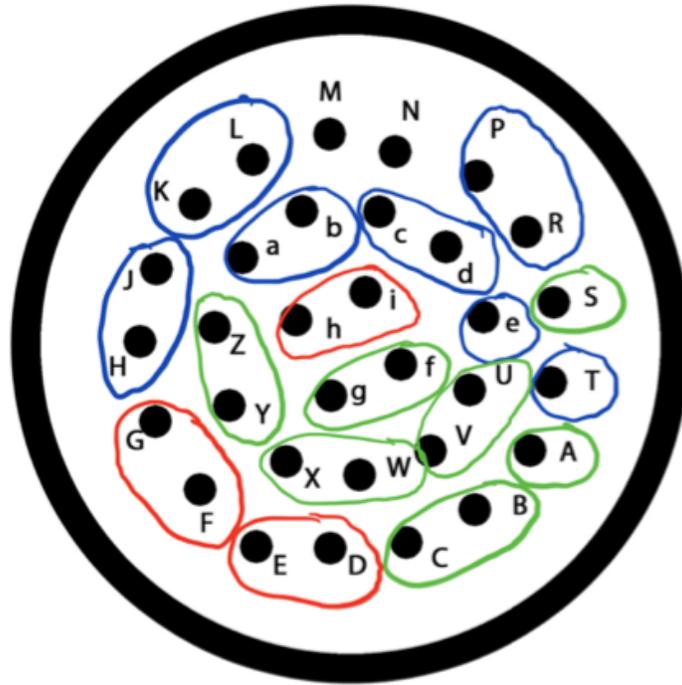


Figure 12: The pins on the external 32-pin connector. Blue denotes voltage pairs, green denotes current pairs, and red denotes all other pin pairings. Note that pins M and N have unknown wiring.

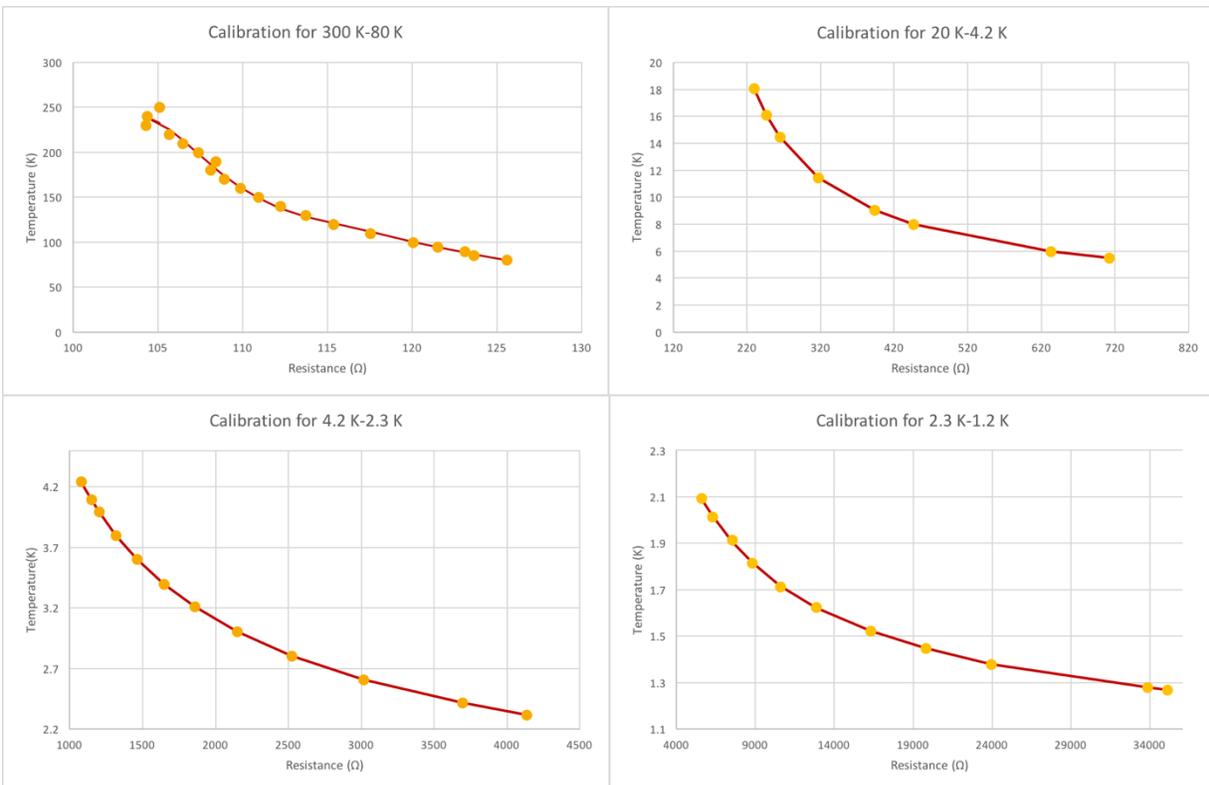


Figure 13: Graphs of the temperatures recorded by sensor 37 (orange) are shown for 300 K-80 K, 20 K-4.2 K, 4.2 K-2.3 K, and 2.3 K-1.2 K. The red line is the calibrated curve plotted for each R.

Table 3: Thermometer calibration curves and regression output for 300 K-80 K are shown. Note, the correlation between 80 K and 20 K has not been included here as the cryostat remains in this range only for a short time during the helium transfer. However, all curves are used while running any experimental tests.

Range	Variables	Correlation Output
300 K – 80 K	r = resistance (Ω) f(r) = Temperature (K) Polynomial degree: 10 20 x,y data pairs $R^2 = 0.9900077323752038$ Standard error: 5.705639784602685	f(r) = $-9.6709411258304026e+006 * r^0$ + $2.7530519457230729e+005 * r^1$ + $-1.5543050471627248e+003 * r^2$ + $-1.6760025235553591e+001 * r^3$ + $1.1726089076001214e-001 * r^4$ + $4.5838021100763854e-004 * r^5$ + $8.9828483906990794e-006 * r^6$ + $-1.5410035867010072e-007 * r^7$ + $1.5570026596851128e-010 * r^8$ + $4.4994228056661034e-012 * r^9$ + $-1.4955427973970537e-014 * r^{10}$
20 K – 4.2 K	r = resistance (Ω) f(r) = Temperature (K) Polynomial degree 6 8 x,y data pairs $R^2 = 0.9999986017070769$ Standard error: 0.0060340045457180115	f(r) = $2.1101075244444547e+002 * r^0$ + $-2.3930082773486787e+000 * r^1$ + $1.2323191936059165e-002 * r^2$ + $-3.4524377629159148e-005 * r^3$ + $5.4512020359851424e-008 * r^4$ + $-4.5599003358531868e-011 * r^5$ + $1.5712300231101784e-014 * r^6$
4.2 K – 2.3 K	r = resistance (Ω) f(r) = Temperature (K) Polynomial degree 6 12 x,y data pairs $R^2 = 0.9999987639811583$ Standard error: 0.000778830426916147	f(r) = $1.1017622753948004e+001 * r^0$ + $-1.3467509953314555e-002 * r^1$ + $1.0609944071454078e-005 * r^2$ + $-4.8430931307784218e-009 * r^3$ + $1.2755306662452724e-012 * r^4$ + $-1.7979244669306914e-016 * r^5$ + $1.0489745106912751e-020 * r^6$
2.3 K – 1.2 K	r = resistance (Ω) f(r) = Temperature (K) Polynomial degree 6 11 x,y data pairs. $R^2 = 0.9998644578696086$ Standard error: 0.0035607127493622382	f(r) = $3.0437570562635625e+000 * r^0$ + $-2.4981736538463921e-004 * r^1$ + $1.7397697344311141e-008 * r^2$ + $-6.4448616844648754e-013 * r^3$ + $1.0895643406911195e-017 * r^4$ + $-3.1015070581990936e-023 * r^5$ + $-7.7805711102622100e-028 * r^6$

3.2.2.2 Pressure Sensors. Pressure sensors used in this cryostat consist of three Baratron pressure transducers, two TC gages, a self-contained analog gage, and a cold cathode gage. Two of the Baratron sensors have a range of 1000 Torr and are located on the magnet pumping line and

the 1K pot. The third has a range of 100 Torr for increased accuracy at low pressures and is located on the cell’s pumping line. This third sensor may be used to obtain a measure of the saturated vapor pressure in the cell, and thus the temperature, if He II is present. All three connect to digital display boxes to show their respective pressures. The self-contained analog gage is connected to the pumping line on the main helium bath and is used to observe relative pressure changes. It is not accurate for an absolute reading and bottoms out at 200 Torr, regardless of the actual pressure.

The two TC gages are connected to the diffusion pump, one on the roughing pump and one the main line from the diffusion pump to the cryostat, past the high vacuum, “HV” valve. The first is read from an analog meter box, and the second on a digital box that also reads the pressure from the cold cathode gage. The cold cathode gage reads extremely low pressures below 1 mTorr between the HV valve and the vacuum space.

3.2.2.3 Liquid Helium Level Meters. Two liquid helium level meters are present in this cryostat: one in the main helium bath (E), and one in the magnet can. Each sensor used four wires colored red, blue, black, and yellow, connected to a superconducting filament. By measuring the change in electrical properties of the level meter, the AMI 134 signal display box [22], once setup for the specific length of the meter it is connected to, can indicate how many inches of the meter are submerged in liquid helium.

In the magnet can, the meter is 12 in. long and is hard-wired to pins e, S, T, and A on the 32-pin electrical port (III). The pin mapping for these wires is given in Table 4. Because of this, it was necessary to create a custom cable capable of feeding the individual signals into the correct pins on the connector of the AMI meter box. Its manual [22] offers the specifications of the pins and the signals they carry, and Figure 14 shows the ends of this new cable. Adjustments to the settings on the meter box are necessary when measuring the level in a LHe dewar due to limited numbers of such boxes within the lab.

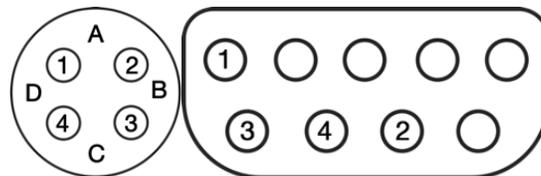


Figure 14: The two ends of the level meter cable for the magnet. The left is the side facing the cryostat and the right is the side connecting to the AMI box. The letters A, B, C, and D are markings on the connector and do not signify anything else.

It is important to not continuously power the level meter, instead taking measurements as given time intervals, as this can generate significant heat and increase the boil-off rate of the liquid helium being measured. Additionally, the bottom and top 0.5 inches of each level meter are not actually measured by the meter. Typically, it was assumed that until the meter read 1-3 inches, the liquid level was not accurate, and could just be an indication of cold helium gas rather than liquid being present. The main helium bath was considered full when a reading around 15 inches was observed. The original broken meter was replaced with a meter from the cryogenics lab.

Table 4: The pin mapping for the liquid helium level meter in the magnet can is shown. The columns follow the same conventions as previous tables, however the color column indicated the color of the insulation on each wire as it attaches to the level meter.

Color	Number	Pin	V or I
Yellow	3	e	V-
Red	1	S	I+
Blue	2	T	V+
Black	4	A	I-

3.2.3 Superconducting Magnet

The magnetic levitation achieved in this cryostat is achieved with a NbTi and Nb₃Sn superconducting magnet [5]. The magnet accepts a current of 118.6 A at an operating temperature of 2.2 K or lower. Its specifications are provided from its manual [11] in Appendix A. It produces a maximum magnetic flux density of 16.3 T in its bore, and a maximum field-gradient product of 22.5 T²cm⁻¹. The cell is suspended and surrounded by vacuum in the 32mm bore of the magnet. A potential minimum is located on the axis of symmetry for the magnet, with the field magnitude increasing with radial distance from this axis. The combined gravitational and magnetic fields, along with the optimized coil geometry, allow a stable magnetic trap to be generated to hold the liquid drop for study. When properly supplied, this system can levitate liquids almost indefinitely. More detailed descriptions of the magnet are in Chapter 5 and the manual. Surrounding the plate with all these ports is a large O-ring, sealed under an annular steel plate. On this plate is a copper ring with braided wire used as the negative lead for the magnet.

CHAPTER 4

METHODS

In the previous chapter, the structures of the cryostat and surrounding experimental setup were discussed with their respective methods of construction. This chapter will focus on the development and execution of the experimental procedures as well as the processes needed to ready the cryostat for actual experimentation. The single largest obstacle in this development was the search for and repair of a great number of vacuum and gas leaks in the cryostat. Once all leaks were sealed at room temperature, a procedure was developed for cooling down the cryostat from 300K to as low as 1.5K in some components. These attempts at cooling down produced their own leaks. Once at 2.2K, the superconducting magnet can begin receiving current from the power supply, and a test was devised to demonstrate that the magnet works and is safe.

4.1 Leak Checking

When a cryogenic system leaks, a number of issues prevent the experiment from functioning properly. If the vacuum jacket leaks, it can no longer maintain a sufficiently low pressure to impede heat transfer from the surrounding environment at 300K through gas conduction and convection to the 4.2K (or lower) cryogenic environment [23]. Rapid cryogen boil-off and dangerous internal pressures can generate great damage as a result of high heat transfer. If a pipe to an internal system leaks, air and water vapor may leak inside and freeze onto cold surfaces. If this happens in a capillary, for example, it may block the flow inside. Because this cryostat has had an extended life serving several different experiments before arriving at the NHMFL Cryogenics Lab, several components were outright broken and many leaks were present. These issues required significant time and brainstorming to locate and repair.

A large number of the leaks on the cryostat were the result of more than one problem. The most common sources (in any number of combinations) were due to O-ring failures, soldering issues, thermal cycling [24], structural integrity issues, window issues, or indium seal failures. When the faulty component was identified, it almost always required a replacement (in part or in whole) of something related to that leak. A common example of such a combination might be a pipe going from the outside into the vacuum space leaking air due to cracks in a solder joint and a

dirty or cracked O-ring. Finding one cause of a leak independently of the others can make repairing that leak difficult. If an attempted fix still leaves the pipe leaking, it may appear as though the attempt had failed.

4.1.1 Broken Equipment

From the very start, it was obvious that several components of the cryostat were broken. The old electrical port (IV) was completely broken off and was only being held onto the top of the cryostat by the wires inside. Likewise, the cell bottom and red fill lines, and the cell bottom pumping line all showed visible damage. Several attempts were made to repair these parts but nearly all required eventual complete or partial replacement. These methods are described in section 4.1.4, specifically stycast and soldering.

4.1.2 Methods for Finding Leaks

Perhaps the quickest way to determine if a system leaks is to generate a pressure difference between the system and its surroundings, and monitor how it changes. In this case, the first step is to either pressurize a given system above atmospheric pressure (usually to around 900-1000 Torr) or pull a high vacuum within the system (usually below 1 Torr). Next, stopping the gas supply or vacuum pump, and observing the resulting pressure rise or drop over a set time interval will provide an indication that a leak is present. This method is not particularly accurate but it is simple and provides an idea of whether a system is leaking significantly.

The most accurate leak checking was performed using an Aiden ASM142 Leak Detector [25] which is capable of detecting the presence of helium atoms. When connected to a chamber with a high vacuum, spraying helium gas next to a suspected leak will cause a signal to appear on the leak detector. In general, a leak rate better than 1×10^{-8} Torr•l/s is necessary for reliable operation of the experiment. The leak detector's internal leak rate is around 1×10^{-10} Torr•l/s, so leaks rates cannot be detected past that amount. Moderate leaks will generally appear around 10^{-6} or 10^{-7} Torr•l/s, however gross leaks could often go as high as 10^{-2} Torr•l/s. Often if several leaks are near each other, or the precise region in which a leak is located is unknown, spraying helium gas is ineffective. Additionally, if helium gas is present, then a large background noise will be observed on the leak detector. If it is not possible to pump the chamber to a pressure below 50 mTorr, it is often not possible to use the leak detector in this manner. For leak checking between

the internal systems of the cryostat and the vacuum insulation space, the leak detector is connected to the vacuum space, and the internal systems are pressurized with helium gas. Any leak between the two will appear during such a test, however a closer internal examination for a precise location can prove challenging.

There is another mode for leak checking using this device, although it is not as accurate. This so-called “Sniffer Mode” cannot generally detect leaks smaller than 1×10^{-6} Torr•l/s, however it can be useful for finding the region where a gross leak is present. For this mode, the system with a suspected leak is pressurized above atmospheric pressure with helium gas and a small, handheld nozzle, connected to the leak detector by hose, pulls in ambient air for the presence of helium atoms to be detected. The biggest drawback of this method is that leak checking is limited to only as long as it takes for the helium gas leaking out to saturate the air around the cryostat. To continue leak checking, a fan can be used to blow gas away from the cryostat for a few minutes, or using a lower gas pressure can reduce the leak/saturation rate.

When the sniffer mode has indicated there is a leak in a certain area, it can help to use a commercial mixture of soapy water called Snoop® [26], or similar agents. For such a test, the system being tested should be pressurized above atmospheric pressure to around 800-1000 Torr with helium or nitrogen gas. The Snoop® is then applied to the surface being tested, and if a leak is present, the gas leaking out will create a stream of bubbles and foam at the site of the leak. This method does not always work as a high pressure difference is needed to push out bubbles through a leak and some components like capillaries, for example, each have a high resistance to flow, restricting the possible pressure at the site of the leak. Depending on the geometry of the surface (smooth or convoluted), it may not be possible to actually get Snoop® onto the leak and there may not be enough pressure to make bubbles. These issues consistently inhibited the search for leaks on the capillaries and fill lines to the cell.

Occasionally, the leak detector will indicate a “ghost” leak. If a system has been exposed to helium recently, residual gas molecules will remain and will artificially increase the reported leak rate, even if no leak is present. The leak detector cannot distinguish between helium left over inside the cryostat, and external helium gas that is being directed at the surface. Likewise, when helium gas is present near the leak detector, the device’s small internal leak rate of $\sim 1 \times 10^{-10}$ Torr•l/s can admit so much ambient helium that it drowns out the signal generated by actively spraying helium gas at the site of a suspected leak.

Whenever possible, the vacuum hoses and lines between the cryostat and the vacuum pump and leak detector should be capped at the point they would connect to the cryostat and leak checked prior to testing the cryostat itself. This helps avoid issues with O-rings or broken hoses leaking and obscuring the presence of a real leak in the cryostat. If a system has not been evacuated by a vacuum pump for a sufficiently long amount of time, the accuracy of the leak detector is reduced and can again indicate “ghost” leaks. Pockets of gas were observed to last for days (despite being pumped on continuously), depending on how high the initial pressure was. It may also be possible to find a leak by covering a suspected leak in a removable sealing putty. This putty can help seal a leak but only partially and temporarily. If a change in pressure or leak rate is noticed after applying putty, a leak is likely present.

4.1.3 Leaks on the Cryostat

Most systems within this facility either presented leaks right from the beginning or formed cold leaks. Initial attempts to learn about the internal structure of the cryostat typically involved pressurizing or pulling a vacuum on a particular system, and observing pressure changes in other components, thus allowing a connection between two systems to be observed. Many of these attempted tests failed due to leaks that were unknown at the time, but which were subsequently discovered.

The main helium bath generally has had the least effective seal against the external environment. This does not pose a serious problem aside from the loss of helium to the room, as the bath is periodically subjected to exposure to the room during liquid nitrogen or helium transfers and during magnet lead insertion or removal. Whenever a chamber is exposed to the air in the room, it should be pressurized with gas to help prevent air and water vapor from entering.

The most egregious leaks affected the vacuum space. These leaks prevented a pressure below around 30 μ Torr from being maintained, causing a high degree of heat transfer by gas conduction and convection into the cryogenic environment. The primary results of such loss of insulation include the formation of condensation and frost on the outer surfaces of the cryostat (and internal when air and water vapor leak into the cryostat), and the rapid boil-off of liquid nitrogen and liquid helium. A leak in the vacuum insulation often can end an attempted experimental run. This has been an issue for this cryostat for the duration of this work.

the pipe that the thermal switch passed through (b), when traversing the helium bath, leaked from the helium bath into the vacuum space.

Another visibly broken set of parts, the cell bottom (4), and red fill (6) lines, and cell bottom vacuum pumping line (2) required iterations of several different approaches to repair, from stycast, to complete replacement with soldering. During the repair of those parts, it was noticed that the capillary on the hydrogen fill line (3) had detached itself from the vacuum insulated pipe that it traversed towards the cell. It was capped off because it would not be needed until far later in the use of this facility and could be returned to a functional state with a small amount of solder modification and/or drilling.

Pipes showing concave radial deformation include the pumping line to the magnet (J*) and the pipe connected to the electrical connections for the cell electrodes (I). When such a leak occurs, the O-ring is unable to properly seal the entire circumference of the pipe and needs to be replaced or bypassed. Figure 16 shows an exaggerated example of such a leak.

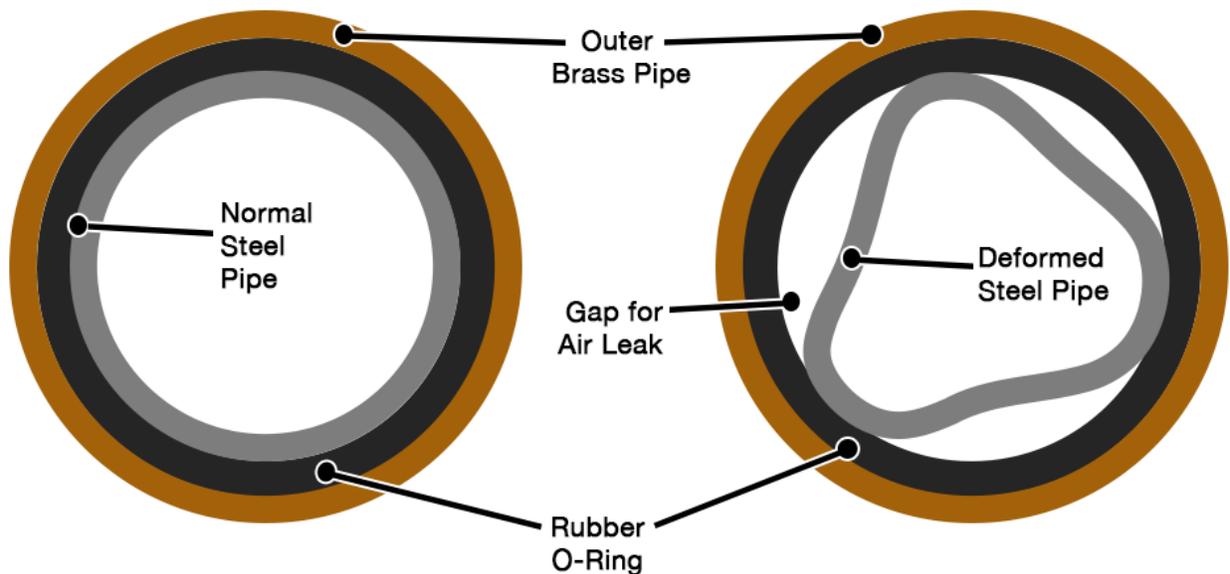


Figure 16: An example of deformed pipe and O-ring is shown. On the left is a normal pipe while on the right is a deformed pipe. For the deformed pipe, air is permitted to pass in the gap with the O-ring.

Small cracks were found at various stages of the leak checking process in the solder on the base of both the bath's double compression fitting pipe (C) and (D), the back lower side of the top viewport (V), and the T-flow valve for the internal magnet fill (K*). A similar crack was found

inside the vacuum space on the pumping line to the $\frac{1}{2}$ K pot. After a few cooldowns, cracks could be seen on the internal portion of the cell bottom vacuum pumping line (2), and on the epoxy connected to the pressure relief port on the vacuum space pumping line (VI). The window on the bottom, outermost window to the cryostat was sealed with epoxy, which also exhibited cracks and was found to leak although the cause of the cracks was never identified, possibly due to their size and visibility.

The internal vacuum space leaks are presented in Figure 17. In addition to the previously mentioned leaks on the $\frac{1}{2}$ K pumping line, thermal switch, cell window, cell bottom pumping line, and old electrical port, internal leaks were found on a few joints along the capillaries around their connection to the gold plate heat exchanger. Leaks were also detected on the connection and valve between the main helium bath and the magnet can.

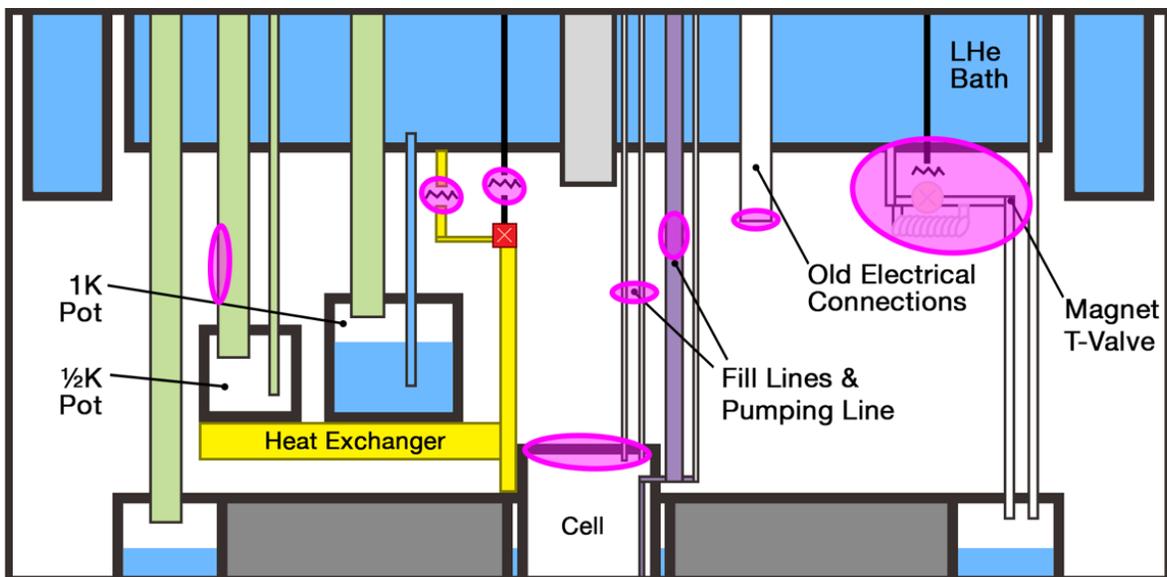


Figure 17: Internal leaks were found within the cryostat where circled in magenta. These leaks required soldering, indium wire replacement, window replacement, or simply cutting and capping.

O-rings are used as room temperature vacuum seals where two pieces of copper, aluminum, steel, or brass are positioned to apply a constant compressive force onto a circular piece of rubber coated in a thin layer of vacuum grease. Issues that have adversely affected the performance of O-rings have been due to dirt or dust on the rubber, lack of vacuum grease, becoming frozen and rigid, or cracks from thermal cycling and fatigue.

Nearly every room-temperature seal in this system relies on an O-ring at some stage. It's important to periodically double check the tightness of each clamp or compression fitting, and that each O-ring is in good condition. When the O-ring is in a compression fitting, the proper sequence of parts should be checked. If the O-ring is making a seal but leaving a path for air to enter (generally through the threads in the fitting), it won't matter how well the fitting is tightened or greased.

The shape and size of the O-ring should also be appropriate for the seal it's attempting to generate. A circular cross section of a piece of O-ring works best against metal surfaces that are slightly curved to ensure greater contact surface area. A rectangular notch in the metal will not seal well with an O-ring unless the geometry of the rubber's cross section is square or rectangular. If the fitting has been cooled by liquid cryogenics or cold gas, the O-ring often becomes brittle and frozen and cannot function. The proper removal of cold gas exhaust from pressure relief valves and transfer ports is a serious consideration. Too much cold gas nearby can easily interfere with the function of a rubber seal.

When a seal in a cold system is required, it is predominately an indium wire seal. Such a seal (or alternatively, a CF flange) is also required any time superfluid He II is present, as a superfluid leak may very easily occur in places where normal He I or other viscous liquids and gasses would not leak significantly. The unique properties of He II allow it to enter regions that a classical viscous fluids cannot [27]. Indium wire has several desirable qualities in cryogenic environments. It is very soft and can be extruded in the laboratory with wire diameters of 1.1mm and 2mm. Indium remains soft at milli-Kelvin temperatures.

Being soft and malleable allows indium to be arranged based on the geometry of the needed seal, concurrently with its installation, but also makes it very easy to tear or damage. Generally, but not always, a groove in the steel surface is present so that the indium wire may be compressed and thus seal across a greater surface area of the steel. The groove also aids in keeping the indium wire from sliding around on the steel surface, potentially preventing the plates from properly sealing with the wire.

Indium seals that leaked in this facility included the seal on the top fused-quartz window of the cell (also exposed to low temperature), as well as the top and bottom indium seals of the magnet can. Figure 18 shows a simple schematic of the indium seals in the cell. The original fused quartz window shattered upon removal, and needed to be replaced entirely. Additionally, when the

cell was opened for internal examination, its indium seal (distinct from the seal on the window) needed to be replaced.

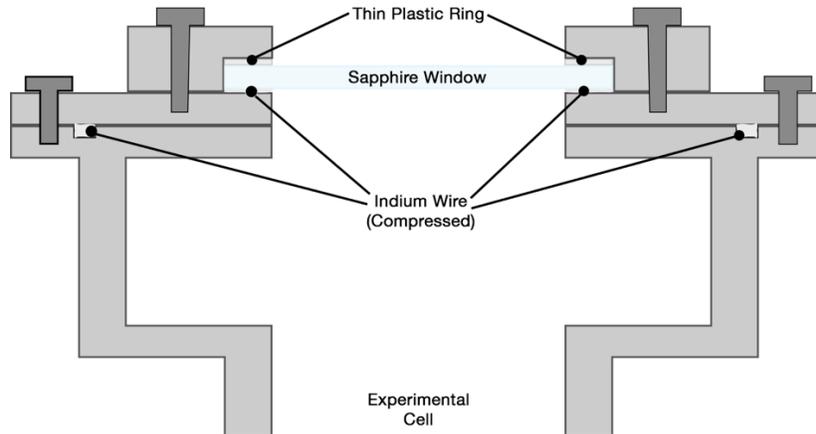


Figure 18: These two indium seals are used to seal the cell.

4.1.4 Solutions for Leaks

The many leaks on the cryostat required several different methods to repair. Those leaks which could be easily fixed, such as those from O-rings, were remedied quickly. Many others ultimately required soldering to replace or seal the leaking fitting. The top of the cryostat required extensive repairs, but soldering was not the first method tried. It was feared that the heat required to solder stainless steel would damage the cryostat, so stycast was employed but proved ineffective. Soldering was the only other option and it did eventually seal each leak. Leaks on the cell window, and the indium seals on the magnet were replaced several times before perfect.

Stycast. Stycast is a resin similar to epoxy that can be formed by a two-part mixture. It needs to be de-gassed with a vacuum pump to prevent the formation of internal bubbles during hardening which can cause leaks. Leak detection around Stycast can be complicated by helium diffusion through the resin, even when leaks are not present [28]. Stycast makes a good vacuum seal but it needs to be placed in areas where it will contract onto the desired seal when cooled down. If any mechanical stress is exerted on the stycast once it has set, micro fractures can form and result in leaks into the cryostat. Ultimately, all stycast needed to be replaced with soldering, except for the bottom window on the outer cryostat shield. The pipes and fill lines that were sealed

using stycast, albeit unsuccessfully, were the cell bottom and red fill lines (4) and (6), the vacuum pumping line for the bottom of the cell (2), and the old electrical connections (IV).

A schematic of how the stycast was applied top plate pipes and fill lines is given in Figure 19. For each leak, the broken pipe was removed and the hole it previously occupied was drilled out and cleaned. A piece of heat-shrink tube was attached to the base of the original fitting, to act as a reservoir to hold the stycast in place and help it contract onto the desired seal. The replacement pipe was inserted, and the stycast was generously applied around the gap with the replacement pipe. The heat-shrink tube was similarly filled. Once the stycast had cured overnight, the seal was finished. Unfortunately, each stycast-sealed pipe leaked. As a result, each of these pipes needed to be replaced, and all stycast removed before soldering could be performed.

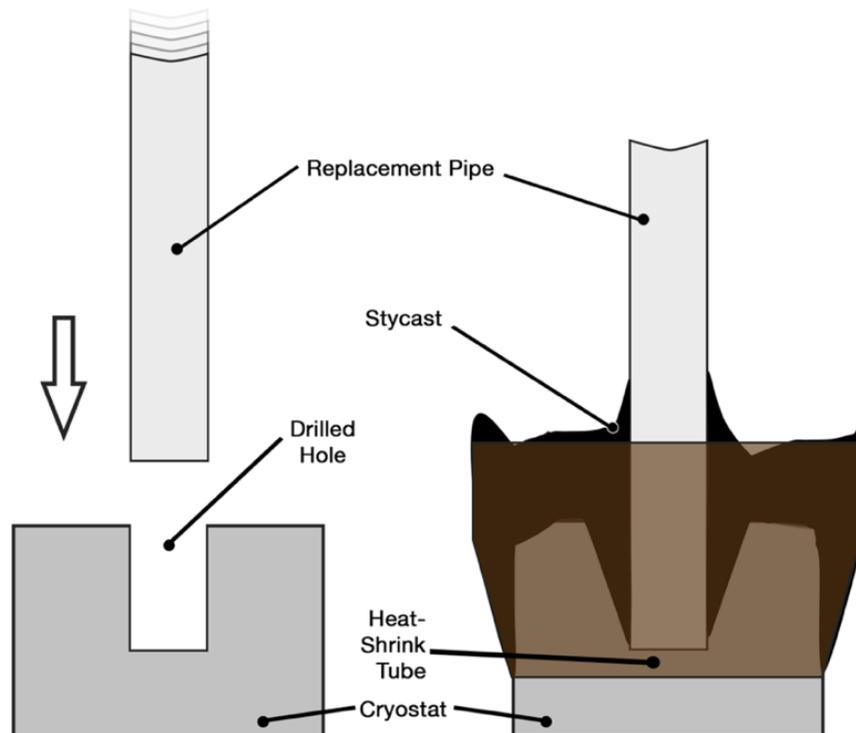


Figure 19: Example of a leak in a pipe on the top flange being repaired with stycast. On the left is a clean hole and the replacement pipe being inserted. The right image shows shrink tube sealed on the original base of the pipe to hold the stycast in place while it cures. This allows the steel and stycast to contract onto each other when cold.

Soldering. The final, successful method for fixing broken and leaking parts was soldering. Soldering here used a solder of tin and lead along with temperatures above 180°C [24] to melt the

soft solder and join two pieces of metal. Before it can be performed, the surfaces must be sanded with sandpaper and cleaned with acetone. Then it is necessary to apply an appropriate flux to the joint. This flux chemically etches the surface of the metal, removing any oxides or debris, and capillary action allows the melted solder to enter the gap between the two pieces of metal and fully wet, making a large contact angle, with these surfaces. It is important to use the flame or soldering iron to heat the metal surfaces and not the solder directly. When heated sufficiently, the metal will instantly melt the solder upon contact.

For these repairs, acid flux was used as it is capable of removing the tough oxide layer on the surface of the steel. This flux was comprised of HCl and zinc chlorides. Soldering copper or brass does not require such a strong flux, but will still work with that flux. Steel is quite difficult to solder because it has a comparatively low thermal conductivity, causing heat to stay right next to the flame, low heat capacity, causing it to cool too quickly for the solder to melt, and a tough oxide layer, preventing the solder from wetting the surface. Aluminum can be many times harder to solder than steel because of its oxide layer. If too much heat is applied, the flux may boil off before the surfaces are hot enough to melt the solder, and several reapplications may be needed. Adequate ventilation must also be maintained for toxic fumes generated by heating the flux.

Where possible, a soldering iron may be used for small touch-ups or repairs, but it cannot heat a large surface or heat anything below the surface, so it is ineffective on large repairs. When copper or brass were soldered, a propane torch was used to heat the metals. For the top of the cryostat, it was necessary to use an acetylene torch (and on one occasion, hydrogen) with the flame maintained as small as possible to avoid heating nearby pipes. Adjacent areas and pipes were shielded by aluminum plates and were kept cool by wrapping with wet napkins or paper towels. A common issue with soldering results from too much heat melting joints nearby, and generating new leaks on those pipes. Additionally, insufficient heating or flux can leave large droplets of solder on the metal, as the solder is unable to wet the surface and its surface tension keeps it in a semi-spherical shape on the surface. After a pipe is soldered, it needs to be cleaned with acetone, some neutralizing agent, and a wire brush. If no pinholes or gaps are visible upon inspection, and no leaks are detected, the soldering was successful.

Externally, a complete replacement was required for the cell bottom fill (4), red fill (6), the cell bottom pumping port (2), and the magnet pumping line (J*). A major issue with these is the mechanical stress they experience during maintenance and operation (likely why they were broken

from the start), so support pipes were soldered around them to add mechanical strength. These all required extensive use of the acetylene torch and were the most difficult to fix. The hydrogen fill (3) was capped and the leak on the vacuum space pumping port (VI) was fixed by installing a new flange with this torch. A soldering iron was used to seal leaking cracks on the viewport (V), the magnet flow valve (K*), and on the T-shaped double compression fitting (C) & (D).

For the internal leaks (Figure 17), only a soldering iron could be used due to the cramped space and possible damage that a torch would cause. It was necessary to solder leaks on the pumping line to the ½K pot, capillaries to the cell, the hose for the pumping line to the cell bottom, and the connection between the magnet and main helium bath. The internal leaks with the helium bath in the thermal switch and old electrical port required caps to be soldered. This created “cold fingers” and undesirable heat loads, which extend below the bath.

Indium Seals. There are a total of four main indium seals, which were opened and replaced during the setup and repair of this facility. Nearly every pipe going into the bath from the top of the cryostat has a corresponding pipe exiting the bottom pipe or cap. These caps are indium sealed but were not modified for this experiment. The connection between the magnet can and the bottom of the bath, which maintains vapor cooling for the magnet leads is also sealed by indium near the base of the bath to keep separate the main bath and magnet bath.

Each indium wire in this cryostat and the two steel surfaces that it will seal together were all cleaned with acetone prior to installation. Any debris on the wire, or even scratches on the metal surfaces, will likely cause the indium seal to fail. When the wire is compressed between two steel plates, the wire needs to be gradually compressed by tightening the screws around the plate in an even alternation (across the diameter of the seal). With each pass, this sweeping motion across them tightens each screw just a bit more. If this is not done evenly, the wire may be compressed in one part of the seal, but may move or separate in another.

Almost every time an indium seal is opened, it needs to be completely replaced with a new wire, although occasionally it can be reused if particularly pristine. When preparing the surface or groove where an indium wire is to be replaced, it is necessary to clear all of the remaining indium from the broken seal. The indium in a groove may be dug and cleared out with a thin, pointed piece of wood, as the wood will not scratch the steel but will be rigid enough despite its low thickness to scrape the soft indium. It is also necessary to ensure the two ends of the wire cross over each

other tightly within the groove of the seal. When the seal has been fully tightened, it should be allowed a day or so to relax under compression, then retightened with a similar procedure.

The indium on the window of the cell needed to be replaced several times to achieve a leak tight window. Whenever indium is installed, leak checking is required to ensure the seal works. A room-temperature leak will only worsen at cryogenic temperatures. A cold leak or a superfluid leak from any system or indium seal, can completely stop an attempted experimental run.

The magnet can has a slightly more involved double indium seal. This chamber is subjected to temperatures below the lambda transition and needs to be superfluid leak tight. The top indium seal has a groove in the steel, but it has a large diameter and needs a wire with a diameter of 2 mm. The lower seal requires smaller 1.1 mm diameter wire and involves compression of the bellows structure that extends below the bore. It is necessary to seal both wires simultaneously otherwise one wire will shift position or not be compressed evenly. The most effective method involved connecting two or three long screws through some of the screw holes on top of the can's shield, while partially sealing the bottom indium wire. In this case, a partial seal means that the wire is compressed and the screws tightened, but not to completion. The top wire was then sealed similarly, and the sweeping sequence of tightening screws was performed on the top wire and bottom wire, alternating one complete pass for each. These seals, while eventually adequate, were very difficult to perfectly seal each time the magnet or wiring near the magnet needed to be accessed or modified.

Optical Windows. The window to the cell was found to be leaking early on in the examination of this facility and shattered when a repair attempt was made. A sapphire window was ordered and used to replace this window [29].

The window on the bottom of the vacuum space was similarly found to be leaking, but it was not removed or replaced. Instead, stycast was used to seal the gap around the window's mount. This proved effective since the window does not experience mechanical agitation and is not subjected to temperature changes unless the vacuum insulation fails.

4.2 Cool-down Procedure

Initially three cooldown procedures were proposed to attempt to cool the cryostat to the desired temperatures. The first used radiation cooling with the liquid nitrogen jacket filled, but was

not effective at achieving noticeable cooling. The second involved liquid nitrogen pre-cooling and final cooling with liquid helium. The third proposal sought all cooling with liquid helium, but was never attempted due to the cost of the massive quantity of liquid helium required to cool such a large magnet mass to a low temperature. The final procedure ended up using elements of all three proposals and will be detailed in this section.

Prior to any cooldown attempt it is necessary to leak check every component of the cryostat and double check all equipment for proper seals (cleaning and greasing O-rings, tightening clamps, checking valves) and proper electrical function. This facility uses a molecular diffusion pump to achieve high vacuum insulation, however this pump requires the vacuum space to already have a pressure below 225 mTorr. The cryostat must be pumped down by a rotary roughing pump to such pressures before a high vacuum can be achieved by this diffusion pump. Generally, this should take place overnight to allow sufficient time to remove as many gas molecules as possible. Once the diffusion pump had pumped the space down, a pressure from $\sim 1 \times 10^{-7}$ Torr to $\sim 7 \times 10^{-6}$ Torr was generally observed. Prior to the cooldown, each component was purged of impurities by repeatedly pressurizing with helium gas to around 1000 Torr, and evacuated by a rotary pump to around 10-50 mTorr. These were alternated 3-4 times each, before liquid cryogenes were transferred. Schematics and descriptions detailing the experimental setup were given in Section 3.2. A slow, gradual cooldown is recommended to avoid damage to the magnet or other structures as a result of large thermal gradients and the resultant thermal strains.

The liquid nitrogen jacket serves as a reservoir for absorbing incoming radiation and aids in absorbing internal radiation as well. The liquid here boils off continuously as it helps cool and insulate. It needs to be refilled daily, generally several times. A precooling of liquid nitrogen in the main helium bath was used to cool the cryostat to 80-120 K, depending on the component. The liquid helium transfer can be performed at higher temperatures in the precooling process, but larger quantities of expensive liquid helium will be used. Before the bath can be filled with liquid nitrogen, a back pressure of helium gas of at least 1000 Torr must be achieved in the magnet can and 1K pot. The cell and $\frac{1}{2}$ K pot were simply left pressurized with helium gas during precooling, and later evacuated before liquid helium was transferred. These back pressures must always exceed the pressure in the bath, and are required to prevent any nitrogen from entering the fill lines and capillaries connecting the 1K pot and magnet to the bath. When liquid helium is transferred, any nitrogen in those lines may freeze and block these internal flows. Once the back pressure was

attained, the bath was filled with liquid nitrogen and left to gently vent excess pressure to the room. Generally, once the bath was filled, cryo-pumping allowed the pressure in the vacuum space to drop to the order of 10^{-8} Torr. Cryo-pumping is a cryogenic process where gas molecules strike a very cold surface and change phase, adhering to the surface, and reducing the effective pressure. This can help maintain a high vacuum when minor leaks are present.

After several days of refilling and management, the temperatures leveled off and it was necessary to remove all nitrogen from the bath. This was achieved by inserting a pipe into the bath to just a few millimeters above the bottom surface. Helium gas was then pushed into the bath with enough pressure to force the liquid nitrogen out, while maintaining a higher back pressure in the other components. Once the nitrogen was fully removed, the bath was purged with helium gas 3-4 times, as before. The back pressure was not stopped until a sufficient level of liquid helium was present in the bath.

The liquid helium transfer consists of three individual transfers: two to the bath, and one to the magnet. First the bath was flushed with cold helium gas from the helium dewar until liquid helium began to flow. The level meters in both the bath and the magnet can do not take level measurements for 0.5 inch on both the top and bottom portion of the length of each meter. Once the level was read to be around 3 - 4 inches, the back pressure in the 1K pot was stopped and the valve to pump on it was cracked open to encourage the flow of liquid helium. The bath meter is 24 inches long, but begins to stop filling around 15 inches, likely due to a narrowing diameter/neck towards the top of the cryostat. Once the level read a value around 15 in., the transfer was stopped, and a transfer to the magnet was similarly started, while the magnet was still pressurized slightly above atmospheric pressure to prevent air from leaking in once the transfer port (L*) was opened. The magnet level meter is 12 inches long and fills to a level of about 11 inches before stopping. A final transfer to the helium bath was then performed, and the pumping ports to the magnet and 1K pot were opened fully to allow both continuous flow from the bath and evaporative cooling to the desired temperatures. Many different sequences were attempted, but this procedure proved the most effective at reducing helium waste and attaining the desired cooling.

4.3 Experimental Test

To ensure proper function of all components of this facility, it is necessary to properly cool down the structure from 300 K to 4.2 K or lower. The magnet requires 118 A to generate the

desired field, and its quench current is 118.7 at 2.2 K [11]. The positive lead supplied with the cryostat did not appear safe, and was redesigned and replaced. Prior to using the magnet as intended, a test was conducted to ensure that using the cryostat as the negative lead would not be dangerous, and that the quench protection circuitry was still functional. This test, the new lead that was designed for the magnet, and the magnet setup are discussed in Chapter 5.

CHAPTER 5

EXPERIMENTAL TEST

Prior to any active usage of the magnet in this facility, a safety committee of experts in the scientific use of high field magnets and related technology was assembled from scientists affiliated with the NHMFL. This committee raised several concerns regarding the original positive magnet lead, and the fact that the cryostat body was intended to be used as the negative lead. To remedy concerns about the loose, detachable end of the positive lead, a new lead was designed by Dr. Mark Vanderlaan and installed in the cryostat.

For an evaluation of the viability of the negative lead, a test was devised to ensure the terminal voltage of the magnet did not exceed 4 V, as indicated in the manual from Oxford Instruments [11]. This test encompassed supplying a current and measuring the resistance of the circuit for various temperatures. Unfortunately, the heater for the persistent current switch malfunctioned during this test, and the measured resistances may simply represent measurements of the current passing through this switch rather than the coil. This switch is made of a superconducting filament and like the magnet, is superconducting at 4.2 K.

To demonstrate the validity and effectiveness of the cooldown procedures developed for this cryostat, temperature measurements were also taken during the cooldown for the resistance test. It was possible to maintain the magnet at 4.2 K for over an hour. During that time, no significant deviations from the expected pressure in the vacuum space or operation of the diffusion pump were observed. All observations implied that this condition in the magnet can be maintained as long as needed although the magnet is not being continuously filled internally. The test ultimately ended due to vacuum insulation failure when pumping was underway on the magnet during the third helium transfer. The exact cause of the vacuum failure has yet to be identified.

5.1 Magnet Leads

To supply a high current to the magnet, a thick lead connected to the power supply is inserted into the main helium bath and screwed into a fitting on the internal base plate of the bath. The original positive lead for this magnet had a loose detachable tip and questionable soldering

joints along its body. Both are undesirable and possibly dangerous, so a new lead was designed by Dr. Vanderlaan. His CAD drawing for the machining of the lead is shown in Figure 20.

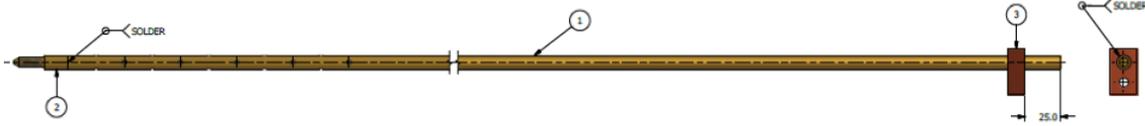


Figure 20: The CAD drawing for the new positive lead as designed by Dr. Vanderlaan.

During operation of this superconducting magnet, the resistive components of the circuit largely encompass the wires and lead from the power supply, which are not superconducting, and the cryostat body, which is stainless steel. While installed in the bath, the lead is partially submerged in liquid helium, and vapor cooled for the remainder of its length. A quick-connect fitting with a non-conductive hose allows helium recovery from the vapor passing through the lead's hollow center. This helps to decrease the resistance and thermal load into the bath. The thin wires connecting it to the power supply for this test do not change temperature, so their comparatively high resistance should be subtracted from the measured resistance of the internal circuit when a four-wire measurement is taken externally. The measurements of just the lead are presented in Table 5, however they do not represent the lead resistance during actual operation as this measurement was at 300 K and was not set up identically to an actual experimental run.

Table 5: The 300 K electric properties of the new positive lead before installation. These values are given for the measurements taken and when adjusted for wires used to measure them.

Supplied DC Current (A)	Measured Voltage (V)	Calculated Resistance (mΩ)	Adjusted Resistance (mΩ)
0.503	0.1241	246.71	1.391
0.753	0.1851	245.81	1.016

Dr. Vanderlaan predicted this resistance to be around 0.78 mΩ. The wires from the power supply are discussed in Section 5.3. Possible explanations for the discrepancy with the lead's resistance may include contact resistance, resistive heating, soldering or other joints on the lead, and simply error in the measurement devices or output of the power supply. Regardless of the

explanation, these resistances are far lower than the resistance of the wires connecting the lead to the power supply.

5.2 Cooldown Test

The magnet is designed to operate while submerged in a liquid helium bath. Its quench current is 109.5 A at 4.2 K and above 118.7 A at 2.2 K [11]. Its protection diodes are designed for operation at or below 4.2 K. In order to operate the magnet, it is necessary for it to enter a superconducting state and at least reach a temperature of 4.2 K. The cooldown procedure is detailed in Chapter 4 and in the manual. Its efficacy was tested by cooling the magnet to 4.2 K and demonstrating the maintenance of superconductivity. The liquid nitrogen precooling procedure was adapted from the magnet manual, and requires a minimum of 5 days of such cooling. Prior to the start of a liquid helium transfer, all liquid nitrogen must be purged or the continuous fill may become blocked by frozen nitrogen. Once in a superconducting state, a test for superconductivity was performed for currents up to 3 A (Section 5.3).

Because the magnet has a large mass, significant quantities of liquid cryogenics are required to cool it from 300 K to the desired operating temperature. A graph of the temperatures in the magnet and 1K pot during precooling is given in Figure 21. Note that the sensors used are not very accurate at room temperature, and the calibration curves typically have higher errors as the temperature approaches 300 K.

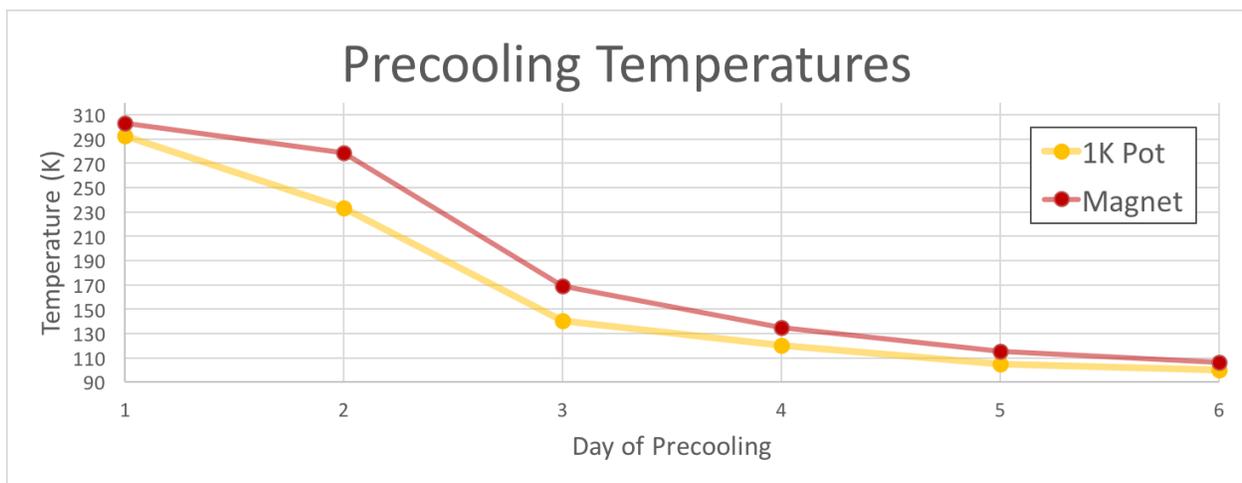


Figure 21: A graph of the temperatures in the 1K pot and the magnet as measured during liquid nitrogen precooling.

During precooling, the main bath is filled with liquid nitrogen (as is the nitrogen jacket), and the 1K pot and magnet are back-pressurized with helium gas at around 1000 Torr. The heat transfer via the gas present in each component's pumping line has proven most effective at achieving this precooling. The 1K pot cools much quicker than the magnet due to its proximity to the bath, and its significantly lower mass.

Once the liquid nitrogen in the main bath has finished precooling the magnet to between 80 K and 120 K, and has been purged, a minimum of three liquid helium transfers are required. First the main helium bath is filled, then the magnet can, and finally a second transfer to the bath is required for a sufficient bath liquid level to maintain the flow of helium to the magnet. Once a level of 2-3 in is observed in the main bath during the last transfer, the flow to the magnet can be encouraged by lightly pumping on the magnet can.

5.3 Magnet Test

The superconducting magnet in this cryostat is composed of eight total windings. The inner three are composed of Nb₃Sn and the outer five are NbTi. These windings vary in height from 8 cm to 14 cm and are arranged to enhance the field gradient in the 32 mm bore. The flying leads connecting to the magnet through its bath are composed of NbTi and run along the bottom of the magnet can to ensure submersion in liquid helium even if the magnet is not fully immersed. To ensure a superconducting state, the magnet must be cooled with liquid helium. To cool it, three transfers are required and the liquid levels in the bath and in the magnet as measured during this test are illustrated in Figure 22.

The starting time of 0.00 indicates the point where level measurements were started: at about 7.8 in. in the bath. The second transfer to the magnet started at 67 mins and the magnet was kept full (~11 in.) from the helium dewar for over an hour during the superconductivity measurements. The second transfer to the bath was aborted when the cryostat's vacuum insulation failed shortly after pumping on the magnet began.

Injecting current into the superconducting magnet requires opening a persistent current switch (Figure 23). This switch consists of a superconducting filament connected across the main terminals of the magnet, and is situated near a resistive heater. This switch provides a short superconducting path for current to flow in a loop if the power supply needs to be shut down or disconnected. When 8 V and a load of 80 mA is applied to the heater at 4.2 K, the persistent current

switch returns to a normal resistive state, and the current then passes into the superconducting coil rather than this resistive path.

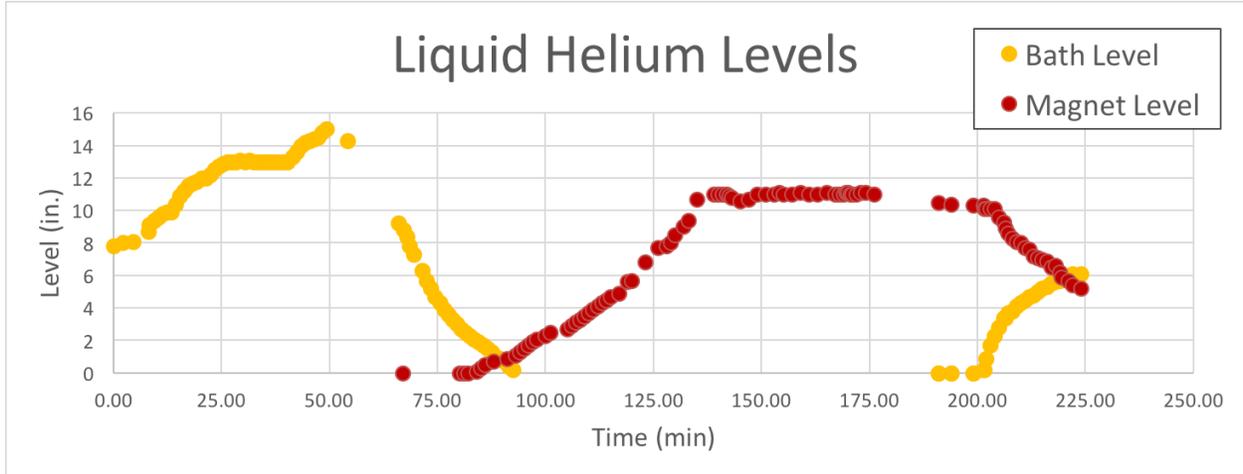


Figure 22: A graph of the liquid helium level in both the main helium bath and the magnet can.

The current is ramped up slowly at around 0.5 A every 10 minutes, until the desired current and field are attained. Normal operation at 2.2 K requires a constant current supply of 118 A for a field of 16.3 T. Once the magnet is fully energized, the switch heater is turned off and the persistent current switch returns to a superconducting state causing the current to remain in the coil.

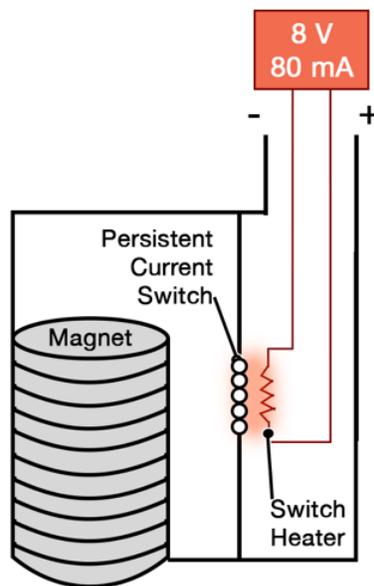


Figure 23: A simple magnet circuit diagram illustrating the persistent current switch and heater.

Throughout the cooldown, resistance measurements were taken for low currents to document the changing properties of the system as it cooled. At 4.2 K, the current was ramped to 3 A, and the voltage was measured across the terminals on the power supply.

During a quench, a superconducting magnet loses superconductivity either locally or entirely, which leads to a sharp rise in voltage as the resistance increases and the current starts to dissipate through resistive heating. The terminal voltage should never exceed 4 V, without activating special diodes inside the magnet to protect it from damage and in the power supply to dump the current into special resistors. For each measurement, a DC current was supplied and the voltage across the two ends of the circuit was measured. From these values, the resistance R of a circuit is calculated by Ohm's law:

$$R = \frac{V}{I}, \quad (3)$$

where V is the voltage and I is the current. At all times, the measured resistance is affected by the resistance in the external wires to the power supply. They do not decrease in temperature (which would change their electrical properties) and as the cryostat cools, their resistance far surpasses that of the internal circuit.

The true power supply for this magnet uses wires with far less resistance. A different power supply with a limited current output was used during this testing to increase safety. To correct for this discrepancy in resistance, measurements were taken for these wires (connected to each other) at various operating currents and subtracted from later measurements in an attempt to correct the measured resistance of the circuit.

For a rough estimate the accuracy of these tests, the uncertainty δR in the calculated resistance was found using the root mean square:

$$\delta R = \sqrt{\left(\frac{\partial R}{\partial V} \delta V\right)^2 + \left(\frac{\partial R}{\partial I} \delta I\right)^2}, \quad (4)$$

where the uncertainty of the voltage δV was estimated to be 0.005 mV and that of the current δI to be 0.5 mA, which leads to a simplified result of:

$$\delta R = \sqrt{\left(\frac{\delta V}{I}\right)^2 + \left(-V \frac{\delta I}{I^2}\right)^2}. \quad (5)$$

The results of some of the tests and the calculated uncertainty of each measurement on just these external wires are given in Table 6. The complete set of values were subtracted from all measurements taken while using this electrical setup during the magnet test. It should be noted that

the uncertainties in the current and voltage are likely much higher as they were measured by hand-held multimeters. That resulting uncertainty in the resistance and in the adjusted resistance implies that the general trend of the resistance changes is likely the real takeaway from these tests, rather than an exact measurement. Likewise, without the persistent current switch in operation, it is uncertain if current actually entered the coil at any time.

Table 6: Shown are several measurements of supplied current, measured voltage, calculated resistance, and calculated uncertainty for the wires connecting to the power supply. This uncertainty should be considered only a rough estimate as questions remain regarding the accuracy of the handheld multimeters used to measure the voltage and current during these tests.

Supplied DC Current (A)	Measured Voltage (V)	Calculated Resistance (mΩ)	Calculated Uncertainty (mΩ)
0.032	0.009	281.25	4.397
0.205	0.0546	266.34	0.650
0.506	0.1352	267.19	0.264
0.746	0.1941	260.19	0.175
1.002	0.2476	247.11	0.123
1.246	0.3073	246.63	0.099
1.497	0.374	249.83	0.084
1.748	0.439	251.14	0.072
2.001	0.505	252.37	0.063
2.25	0.567	252.00	0.056

The results of some of the measurements of the magnet circuit from 1 A to 3 A, while it was continuously kept filled with liquid helium at a level of about 11 in. of LHe, are shown in Table 7 along with the current supplied, the resistance measured, the adjusted resistance, and the combined uncertainty of the original resistance measurement and that of the adjustment. Such low uncertainties might aid but cannot assert the validity of the measured resistances.

At room temperature, the original circuit had a resistance around 48.8 Ω. It was observed that when attempts were made to supply a current over ~0.07 A to ~0.08 A (given a 300 K resistance of 48.8 Ω), the voltage measured would pass 4 V, then began dropping as it appeared

that the diodes in the magnet began to impede this “quench”. Additional confirmation of this observation is recommended during a future test.

Table 7: Some results of the magnet test while it was continuously filled with liquid helium from a dewar and was maintained at 4.2 K.

Supplied DC Current (A)	Calculated Resistance (mΩ)	Adjusted Resistance (mΩ)	Combined Uncertainty (mΩ)
0.216	316.204	49.862	0.997
0.307	317.264	50.922	0.781
0.401	317.706	50.512	0.661
0.499	317.635	50.442	0.583
0.598	316.555	56.368	0.529
0.706	309.632	49.444	0.484
0.801	303.371	56.265	0.454
0.893	296.193	49.087	0.430
1.002	285.329	38.224	0.407

Unfortunately, the persistent current switch could not be used to guarantee current injection into the magnet because the switch heater malfunctioned during the test. It is possible that the observations of resistance changes may simply be due to the persistent current switch, which is also superconducting at 4.2 K. Because the switch is located in the base of the magnet with the lead terminals, it is possible that it was submerged in liquid helium at the start of the transfer. The measurements of the magnet’s temperature were taken from a sensor located on the top of the coil and don’t necessarily represent the temperature at the base, which could be much colder. Repeated future measurements will be necessary to confirm these observations. A sensor in the base of the magnet could be used to confirm the claim that superconductivity in the switch can be observed prior to complete coil submersion. The adjusted resistance of the circuit is plotted in Figure 24 as

a function of the temperature measured on the top of the magnet. Three resistances of note are indicated. A sharp decline occurred around 64 K and began leveling out around 45 K. Both temperatures are far above 4.2 K, although this temperature was measured at the top of the magnet.

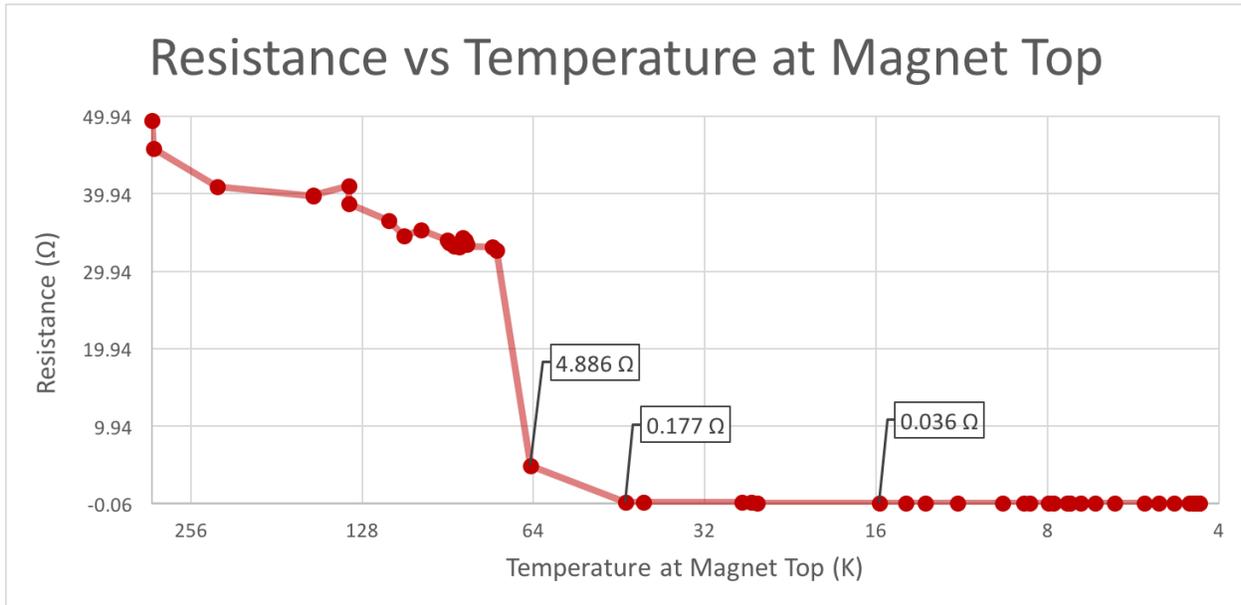


Figure 24: A plot of the resistance of the circuit as a function of the temperature on the top of the magnet. A sharp decline was observed at about 64 K, and began to level out at 45 K. The magnet was maintained at 4.2 K for over an hour.

The liquid level may to be a better indicator of how much of the magnet had been submerged and thus begun to cool to 4.2 K. Figure 25 shows a plot of the (adjusted) measured resistance and the liquid helium level both as functions of time. Two pairs of concurrent data points are indicated on each plot to emphasize the liquid level and the changing resistance. Because the magnet's quench protections limit it to 4 V at 4.2 K, and its quench current at 4.2 K is 109 A, the resistance should be on the order of 36 mΩ. It cannot be proven that current was injected into the coil, but the adjusted resistance does appear to indicate that at least the persistent current switch may have been superconducting during this test.

The coil was maintained in complete submersion with liquid helium for over an hour, which was likely (although not certain) sufficient time for it to cool to 4.2 K. If the coil was also in a superconducting state, some current may have passed through it in parallel with the persistent current switch, although additional testing is required.

While the persistent current switch's heater malfunctioned, and the vacuum insulation did eventually fail, the magnet was maintained at liquid helium temperatures for well over an hour with no observed issues. All observations during this period supported the idea that the magnet could be kept stable and maintained at 4.2 K. The test and third liquid transfer had to be aborted about halfway through the second transfer to the bath, when pumping on the magnet can began. This may have been due to shaking and vibrations of the diffusion pump and cryostat caused by the vacuum pump, or a likely leak in some internal component of the bath-magnet system. More testing is necessary to verify the cause of the leak. At various points during the cooldown, it was observed that occasionally, even minor agitations of the table supporting this pump could alter the pressure in the vacuum space. The explanation for this observation is currently unknown.

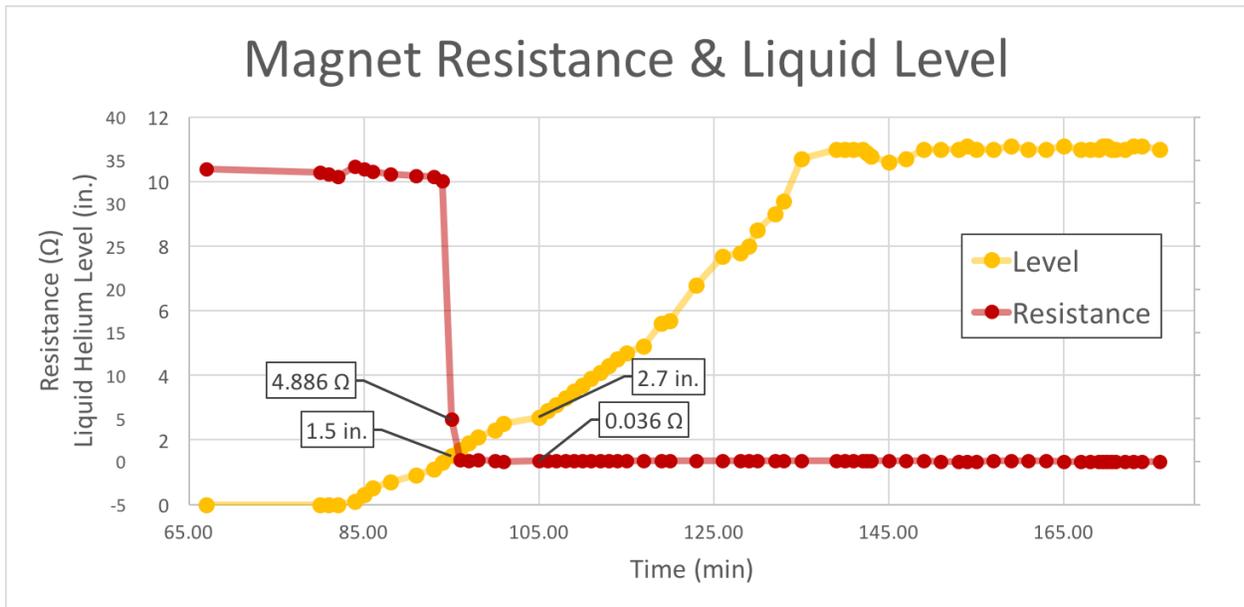


Figure 25: A plot of the adjusted measured resistance and liquid helium level in the magnet can, both as functions of time. It appears that the sharp drop in resistance was observed when 1.5 in. of liquid helium was present. About 2.7 in were detected when the measured resistance approached 36 mΩ. No certain conclusion can be drawn without additional testing, however.

CHAPTER 6

SUMMARY AND FUTURE WORK

When the cryogenics lab received this magnetic levitation cryostat, it had no documentation or manuals. A complete understanding of the facility resulted from close examination and this ultimately produced a complete user manual. While developing this manual, a cooldown procedure was created and refined to effectively attain the proper operating temperature for the superconducting magnet. A new positive lead was designed and tested using a low-current test of the coil. Because experiments using this facility are intended for future work, it was necessary to determine which features of the cryostat were needed and for which purposes.

Significant leaks and damages were present from the onset of this work and needed to be located and fixed before any operational tests could be done. These leaks required several different methods of repair, from stycast, which ultimately did not work, to soldering with an acetylene torch. Ultimately, these repairs imparted an even deeper understanding of the cryostat, and led to the design and construction of the gas and vacuum handling systems. Details of structures and operations are all given in the user manual in Appendix A.

Future research using this facility centers around the study of fluid dynamics and sloshing motion in microgravity, and the study of rotating drops of superfluid He II to study quantum turbulence. Future work will modify the experimental cell to allow a drop of levitated liquid to be rotated and this rotating morphology to be studied. Possible methods to achieve such rotations involve either the existing electrodes in the cell, or the installation of a magnetic quadrupole. Either scenario would make use of opposed alternating fields with a phase difference of 90° . The existence of three potential optical ports (two currently in use, and one sealed on the top flange) allows for numerous laser-induced fluorescence setups to study these and other low-temperature phenomena. A larger facility for conducting such research is being planned and this cryostat serves as a prototype for such future work.

Additionally, new instrumentation and a feed-through for them should be installed. An ideal spot would be the spare gold-colored port (e). Once a second electrical port has been installed, some additional sensors could be placed on the fill line between the bath and magnet (for use during pre-cooling), and the bottom of the helium bath.

APPENDIX A

USER MANUAL

This document is adapted from the user manual produced by this thesis work. To meet all formatting requirements for this manuscript, the presentation of the information here has been modified although the content is largely unchanged. Several portions of this document are duplicated within the main body of the thesis, however the generation of this manual was the primary goal of this work, so including it in its near-entirety was a logical decision. Note, two sections of the manual were written by Dr. Mark Vanderlaan are not included here, however they regard the magnet safety and lead design, respectively and they are his own analysis and design.

A.1 Introduction

The cryogenic facility used for this research is a model 16CNDT Janis Dewar [19] containing a large liquid helium bath surrounded by an annular liquid nitrogen jacket. The core of the facility is the experimental cell. Inside this isolated chamber, cryogenic liquids may be levitated and manipulated for various studies at extremely low temperatures. To simulate microgravity, the cell is suspended in the bore of a superconducting magnet that possesses a geometry designed around the magnetic susceptibility of helium. To hold cryogenic liquids, the cell must be kept cold and insulated. Additionally, levitation requires a means to balance the earth's gravity. Here, a superconducting magnet provides the needed upward lift on an induced magnetic dipole from a 16.3 T magnetic field with a carefully designed field gradient to enhance stability in the levitated fluid.

As expected, superconductivity for this magnet can only be achieved at extremely low temperatures. In this facility, liquid helium must continually be supplied as a coolant from a large liquid helium bath to several systems. If the correct temperature is not maintained, the magnet will quench and rapidly boil all cryogenics nearby. Cooling the system down requires ports to introduce liquid cryogenics to their respective baths and the bath and magnet each have their own fill port.

Cooling down is, however, just half the work in levitating a liquid in the cell. Keeping the cell and superconducting magnet cold requires effective thermal insulation. A diffusion pump is therefore connected to maintain a pressure low enough to curtail conductive and convective heat transfer. Radiation can transit a vacuum and in this facility, it is reduced by several concentric

radiation shields located around these chambers, which reflect and conduct this heat to a cold reservoir: the liquid nitrogen jacket.

While liquid nitrogen is effective at precooling the system, and liquid helium will reduce the temperature to 4.2 K, superfluid He II, with its remarkably high effective thermal conductivity and other interesting properties, cannot be studied or used to cool the magnet to its operating temperature without cooling below the lambda transition of 2.17 K. Evaporative cooling is an effective means to attain lower temperatures in liquid helium, so pumping ports are needed to facilitate a pressure reduction. Reducing the magnet's temperature allows it to carry more current and remain cold. A vapor-cooled current lead connects through the main bath into the magnet and uses liquid helium and cold vapor to reduce its electrical resistance and the resultant joule heating. Cooling the cell below 4.2 K requires additional components, notably the 1K pot, attached to an internal heat exchanger. This pot, like the magnet, is continuously filled by the main helium bath and has a pumping port to allow for evaporative cooling of its liquid helium to lower temperatures.

Once the thermal and magnetic field requirements have been met, levitating a fluid requires the fluid be introduced into the cell, where the magnetic field provides lift and balances the earth's gravity. To that end, several fill lines are present and connect to the cell. Only one is required to introduce fluid, but different lines have slightly different properties. One fill line is vacuum insulated from the bath and can be used for hydrogen levitation. Two more each consist of a thin tube with a 0.01 cm diameter wire protruding from its end. One uses a stainless steel tube and stainless steel wire and the other has a glass tube and glass fiber to avoid electric discharges when using the electrodes. Ultra-high purity helium gas is supplied from a gas cylinder to a given fill line. That gas transits the main helium bath, heat exchanges, and condenses into the liquid phase. After exiting the bath and entering the cryostat's vacuum space, the liquid is cooled to lower temperatures via a heat exchanger on the 1K pot. Finally, the capillaries carrying the liquid enter the cell and deposit the liquid into the magnetic trap. If the drop temperature is not low enough, the cell has a pumping line to facilitate additional evaporative cooling.

Of course, levitating the liquid in the cell is not particularly useful without a means to study the levitated drop. To allow observations, the cell has top and bottom windows, an optical window on the bottom of the cryostat, and a side window that points to a mirror above the cell. These allow photographs to be illuminated and taken of the experiment as it occurs. In the event that some liquid makes its way to the bottom of the cell, past the magnetic levitation trap, it may remain in a

pool. Such a pool can obscure the imaging of a levitated drop, so a second, smaller pumping line is connected to the bottom of the cell and allows this pool to be removed as needed.

A.2 Experimental Setup

To provide a full perspective on the system schematics in later sections, a full schematic of the entire experimental setup is shown in Figure 26. Not shown are electrical ports, the hydrogen fill, all spare ports, the helium bath level meter, magnet leads, and the back port to the bath (G).

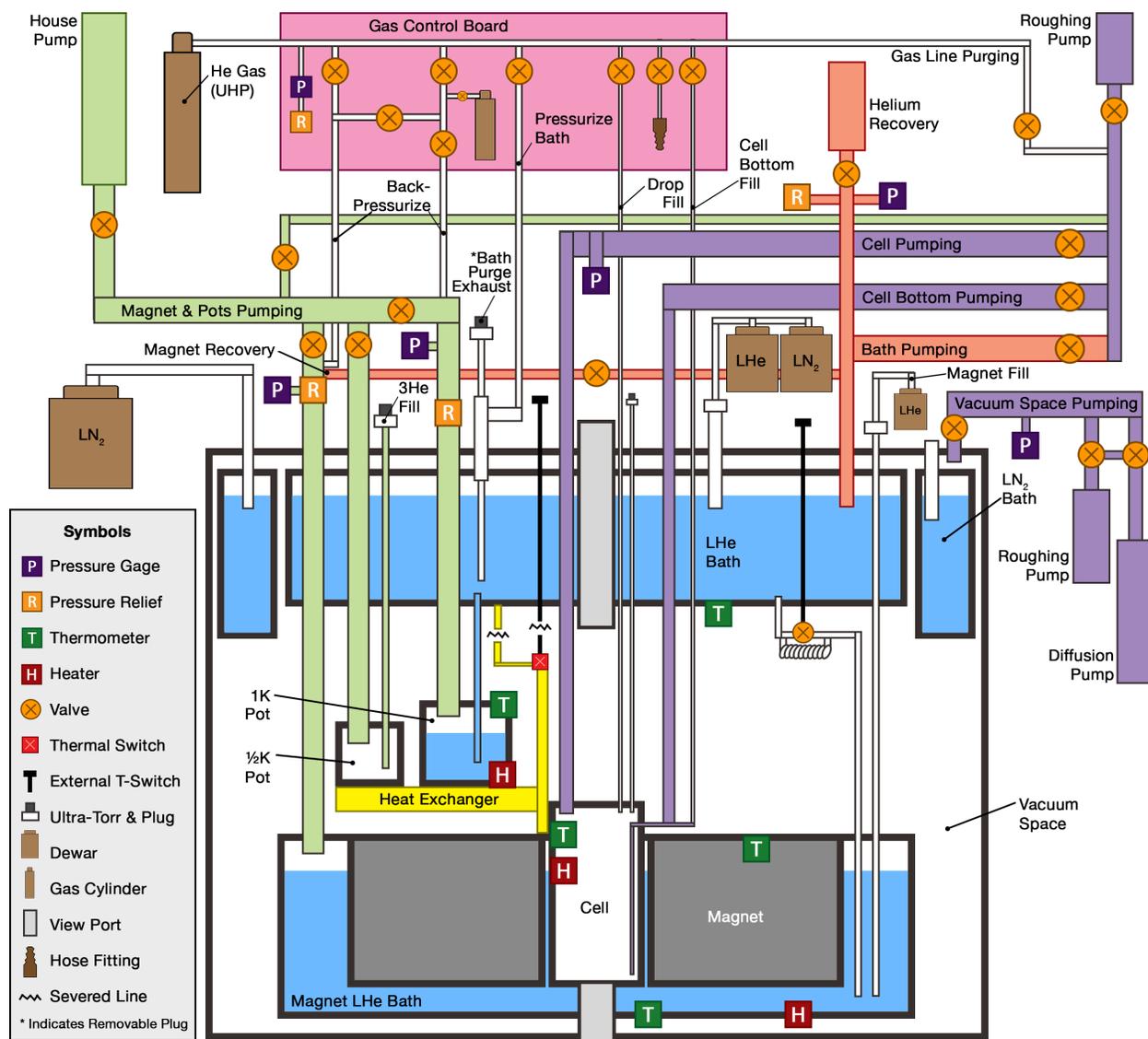


Figure 26: The entire cryostat piping system is shown here. The ports for instrumentation are not shown. Later schematics of the individual systems of the cryostat are simplified from here. The specific spatial orientation and scale of components are not reproduced.

Colors are used in Figure 26 for legibility to differentiate piping systems and do not correspond to the coloring scheme used for the schematic of the top plate. Likewise, a schematic of the top plate of the cryostat is shown in Figure 29. The system colors and numbering schemes for all other diagrams follow from this figure. Ports for the bath and magnet systems are labeled using the upper case Latin alphabet. Ports for the cell are labeled using Arabic numerals. Ports for the vacuum space are labeled using Roman numerals, and finally, ports for the isolated spares, and for the ^3He system are labeled using the lower case Latin alphabet. The labels with an asterisk show the ports that connect to the helium bath internally.

Aside from the isolated (brown) system, and ports with an asterisk, the colors indicate separate systems without connection to each other. Additionally, the gas handling control system is shown in Figure 27.

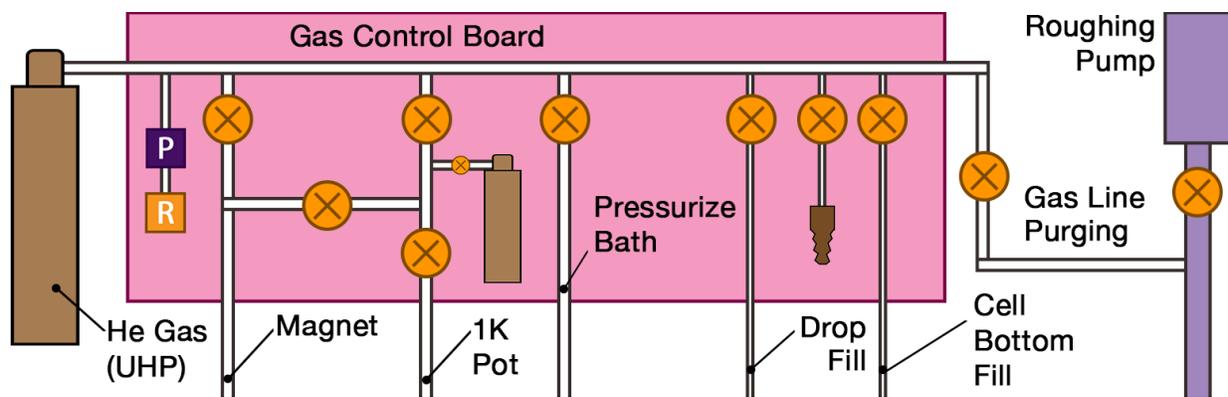


Figure 27: A schematic of the gas handling system as modified for this cryostat.

Two ultra-high purity (UHP) helium gas cylinders are connected as indicated and allow two different pressures to be maintained. The 1K pot and magnet can be supplied gas independently of the fill lines and bath while back-pressurized. This gas board features a connection to the roughing pump system so that it can be purged of air when new gas cylinders are connected or when it is exposed to the room for any reason. This board also supplies the helium gas used to produce a drop in the cell.

Finally, a spare line is available with a hose fitting for venting and as an easy connection for the nozzle used to spray helium during leak checking. Schematics for the vacuum pump control boards are given in in Figure 28. These boards are fully removable from the wooden frames in which they are currently mounted.

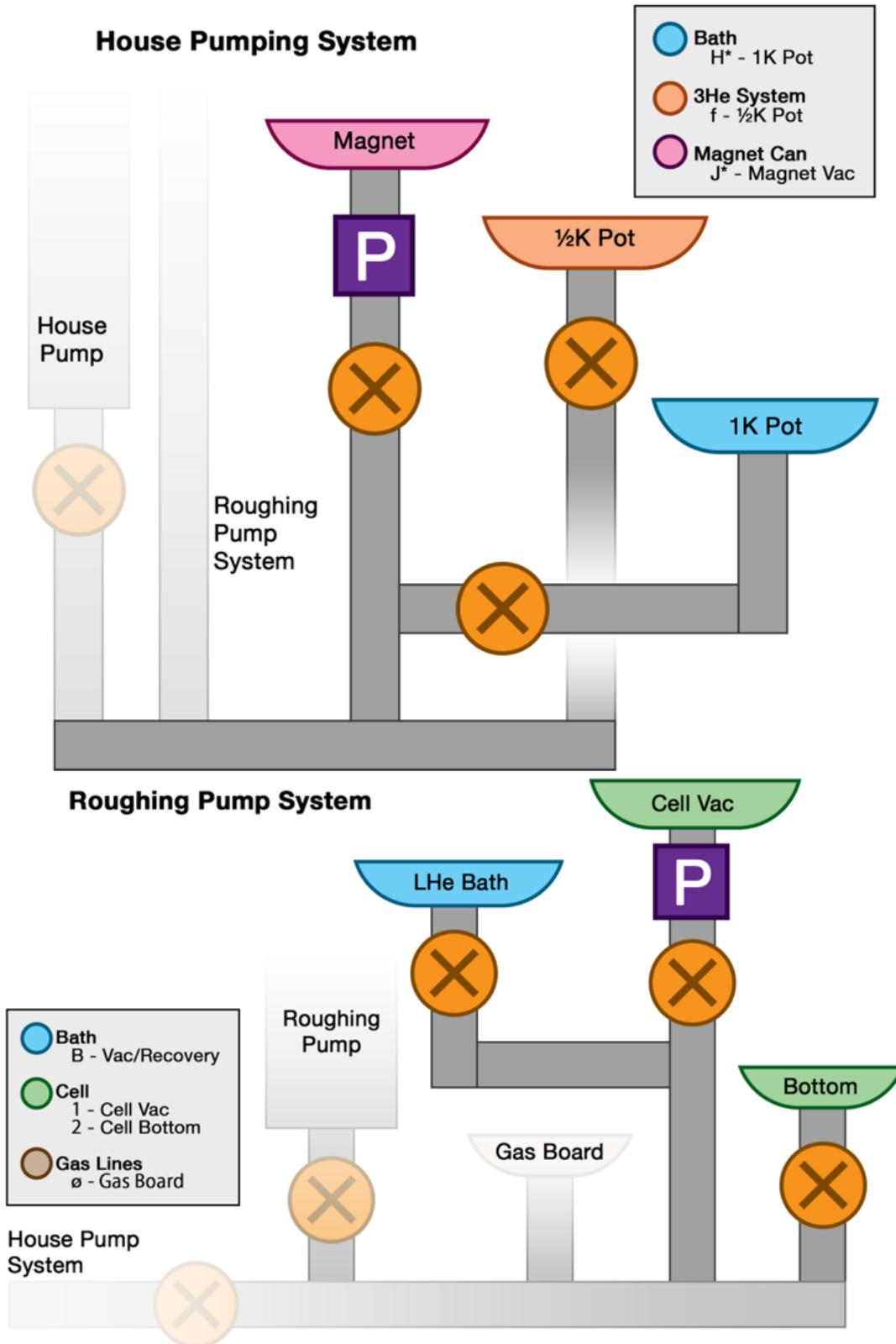


Figure 28: The control board for the house pump system is on the top. The control board for the roughing pump system is on the bottom. Portions which appear faded are not visible to the end user unless viewed from behind the cryostat.

A.2.1 Experimental Cell

The purpose of this cryogenic facility is to conduct experiments on levitated cryogenic fluids. Levitation requires such liquids to be injected into a special magnetic trap inside an isolated experimental cell. The cell needs to be maintained at a unique temperature, so while it is suspended in the bore of the magnet, it is surrounded by vacuum to thermally isolate it within the bore. Its temperature needs to be cooled below the normal boiling point of liquid helium, so it is thermally linked to the 1K pot, and it has a pumping port to facilitate evaporative cooling.

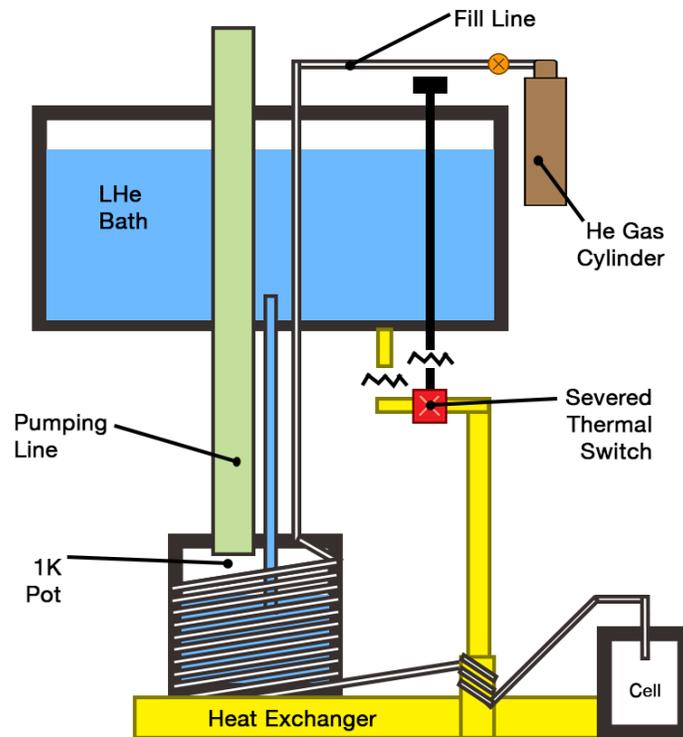


Figure 30: The liquefaction and route of UHP He gas on the way to the cell is shown. Room-temperature gas leaves the cylinder and liquefies on transit through the helium bath.

Liquid is injected into the cell via a system of capillaries. Room temperature gas is condensed while going through the bath, and cools below 4.2 K by heat exchanging with the 1K pot before being deposited into the cell, as seen in Figure 30.

The cell is sealed by 2 indium wires as seen in Figure 31. Details for the connections and functions of each important component of the cell are given in Table 8. A corresponding schematic is given in Figure 32.

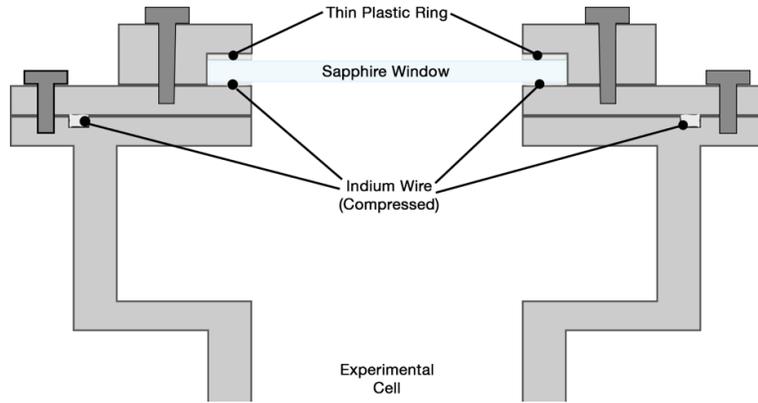


Figure 31: Schematic of the indium seals on the cell.

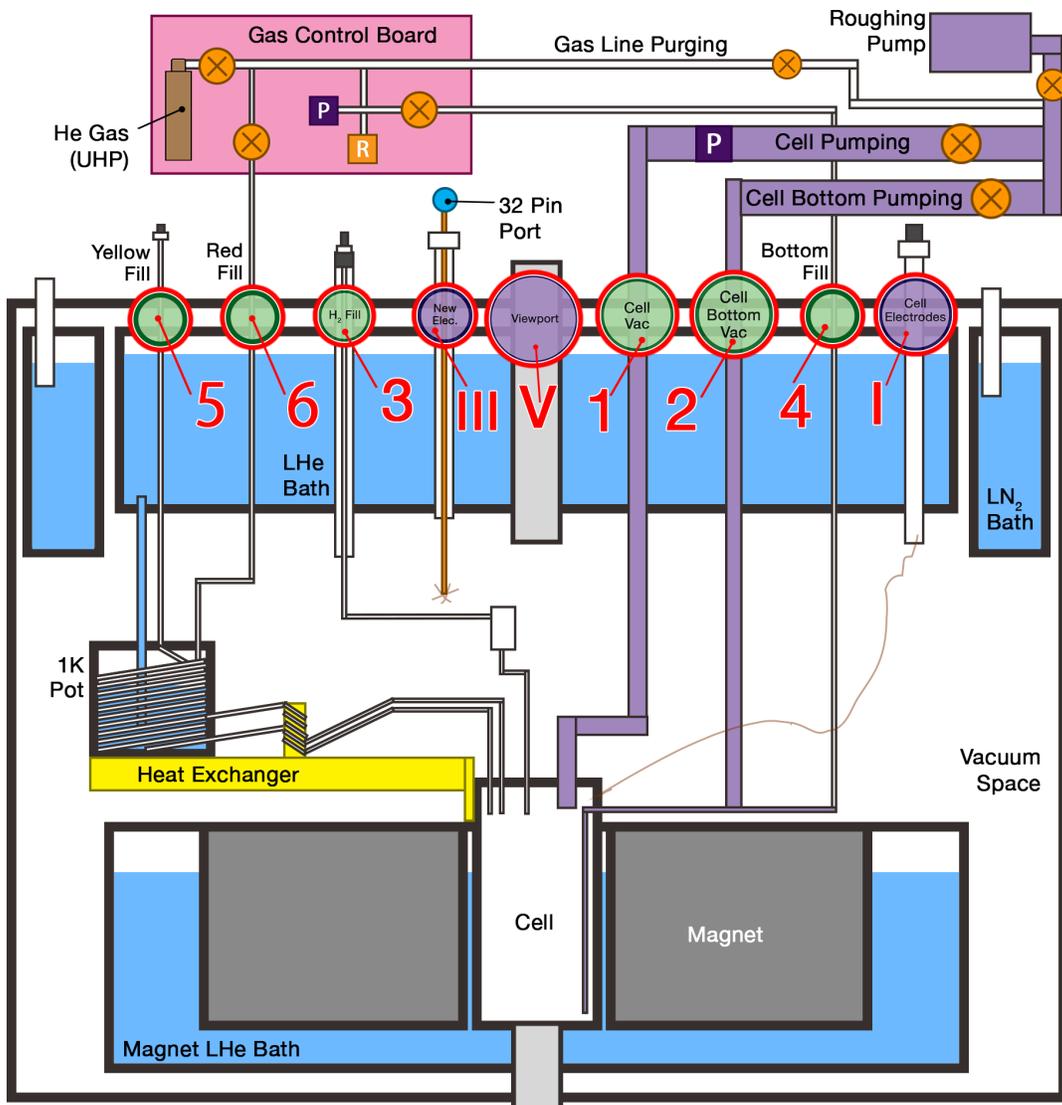


Figure 32: Components of the cell and all relevant connections. Wires for (III) are not shown.

Table 8: Components and connections to the cell.

Component	Location	Functions	Notes
He fill lines	Top plate: (4), (5), and (6)	-Provides the UHP He gas that liquefies in transit to the cell -Yellow fill is unused -Red fill is connected to gas board	-See Figure 30 -High flow resistance
Pumping port	Top plate: (1)	-Allows for the drop to be evaporatively cooled below 4.2 K -Used for pumping out/pressurizing the cell during purging -Used to obtain the cell's pressure	-The portion between the cell and the external port has developed cracks and these have needed repair
Bottom pumping line	Top plate: (2)	-Allows liquid to be removed from the bottom of the cell, if obscuring the image	-Inside, this line connects to (4), then both connect via a capillary to the cell
Electrodes	Top plate: (I)	-Allow charging of the levitated drop, and manipulation of the shape -Allows shape oscillations to be studied, perhaps also sloshing	-The electrodes are disconnected but can be easily fixed on (I)
Instrumentation	Top plate: 32-pin connector (III)	-The signals for the thermometer in the cell pass through (III), and enter the side of the cell	-The thermometer in the cell is not as accurate as measuring the SVP
Viewport	Top plate: (V), cryostat side, and above and below cell	-Facilitates observation of the drop -If condensation is visible on the side port, the vacuum space may have issues insulating	-The window on the top of the cell was replaced with sapphire
Hydrogen fill	Top plate: (3)	-Used to introduce hydrogen gas to levitate in the cell -Disconnected currently	-Vacuum insulated to prevent solidification while passing through the bath
Heat exchanger	Internal: thermal link to 1K pot	-Provides a thermal link to the cell with the 1.5 K LHe in the 1K pot -The cell is lined with copper that continues up into this exchanger -Originally connected to a thermal switch linking to the bath but now disconnected and sealed	-The ½K pot is also linked, but not used

A.2.2 Vacuum Space

A schematic of the vacuum space is shown in Figure 33 and a table detailing all of its components and connections is shown in Table 9. The vacuum space is vital to safe and proper operation of this facility. It provides the necessary insulation and isolation from the 300 K room for every internal component, especially the experimental cell. The magnet cannot levitate unless it is in a superconducting state, at very low temperatures, and this is not possible without vacuum insulation.

Table 9: Components and connections to the vacuum space.

Component	Location	Functions	Notes
Pumping port	Top plate: (VI)	-Allows the vacuum space to be evacuated and to insulate its internal components from heat transfer -Contains a built-in bellows valve -A KF-25 flange is soldered onto the valve, but not currently in use	-Three O-ring seals are present throughout the valve -Ensure no large mechanical stress is applied to the blank pipe from the valve, connecting to the pump
32-Pin connector	Top plate: (III)	-All electrical instrumentation inside passes through this port	-Do not allow this port to freeze or experience any large stress or it will leak
Top viewport	Top plate: (V)	-Can be used as an extra optical channel to the cell	-A small crack in the side was sealed with solder
Cell electrodes	Top plate: (I)	-Originally a BNC port for electrodes -Currently plugged with a copper rod	-The wires for the electrodes are currently severed
Old electrical line	Top plate: (IV)	-Originally housed a double 32-pin electrical port -Significant source of leaks	-It leaks with the bath internally -This pipe is currently sealed externally and internally
Thin wire	Top plate: (II)	-The purpose of (II) is unknown	-Currently just sealed

No cryogenics should be transferred or stored without a pressure below 3×10^{-5} Torr, and ideally on the order of $\sim 10^{-6}$ or $\sim 10^{-7}$ Torr before cryo-pumping. A low pressure prevents heat

transfer via gas conduction and convection. Radiation can still pass through the high vacuum, so several concentric radiation shields absorb and conduct this heat to a nitrogen bath, which acts as a cold reservoir. It is sealed with a large O-ring around its midplane, and a side and bottom window are sealed with epoxy and stycast, respectively. The vacuum space must be regularly maintained and leak checked or most cooldown attempts will fail.

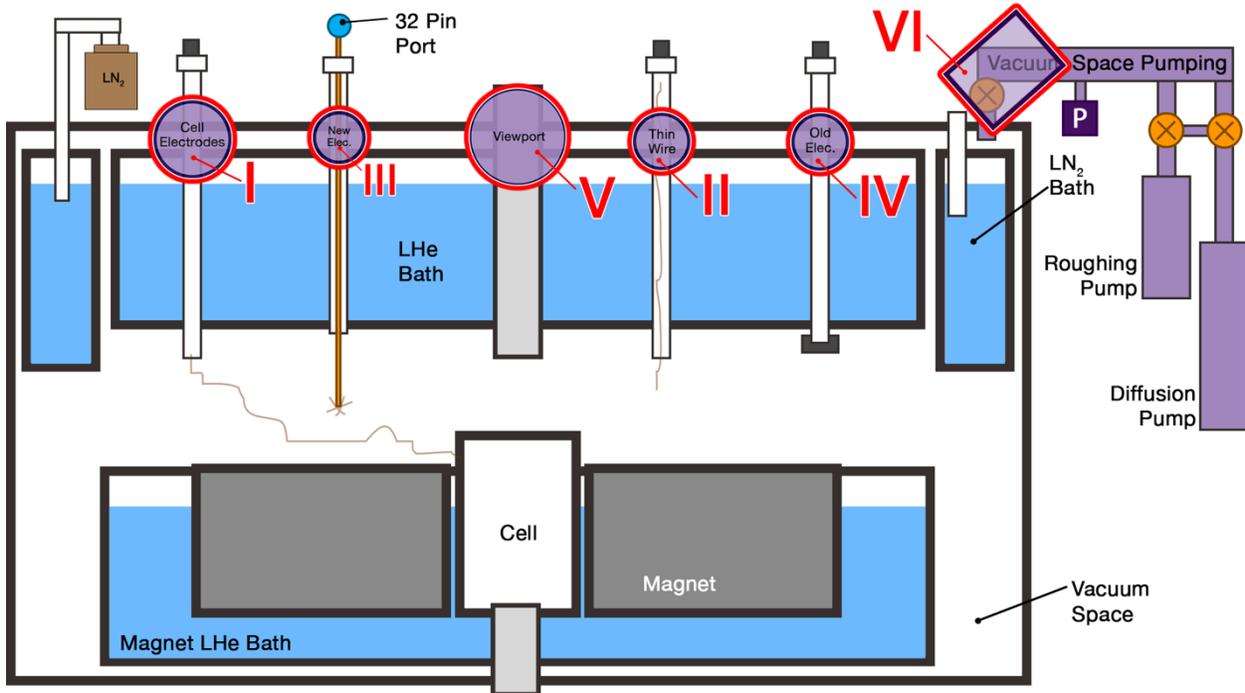


Figure 33: Components of the vacuum space and all relevant connections. Note that any internal system may leak with the vacuum space, even if not shown here.

A.2.3 Magnet

To attain levitation in the experimental cell, this 16.3 T superconducting magnet generates an upward lifting force, which balances the downward pull of gravity at the levitation point. Helium requires a value of $|B(dB/dz)| \geq 22.5 \text{ T}^2/\text{cm}$ for stable magnetic levitation [5].

In order to generate a sufficient field and field gradient, 118 A must be supplied to this magnet by an external power supply, and this can only be achieved in a superconducting state. At 4.2 K, the quench current is 109.5 A, and at 2.2 K, the quench current is $> 118.7 \text{ A}$ [11]. Therefore, this magnet will only function correctly at full current at temperatures below 4.2 K, the normal boiling point of liquid helium. Figure 34 shows a schematic for the magnet system.

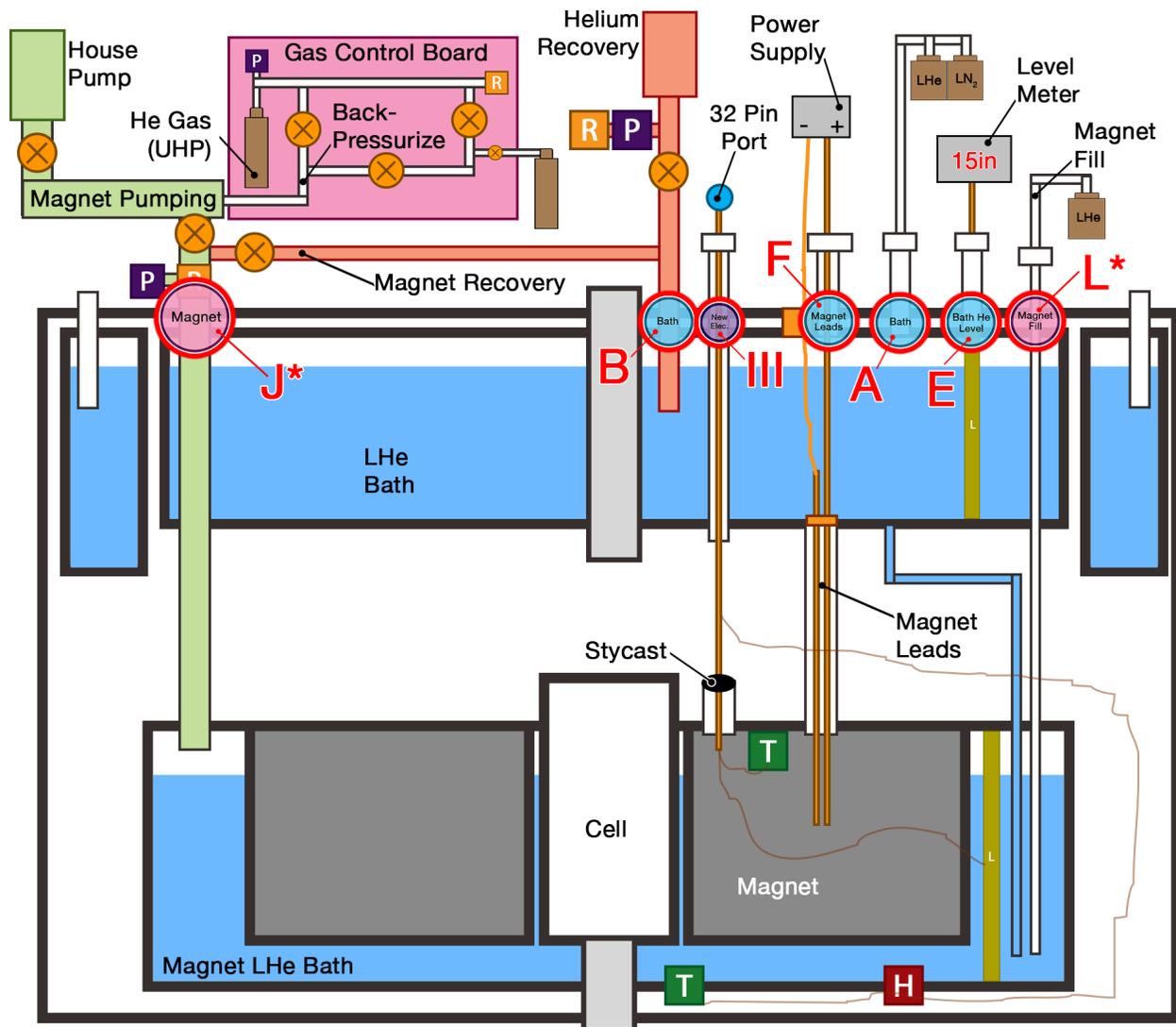


Figure 34: A simplified schematic of the magnet system within the cryostat. Note that the level meter for the magnet is 12 in. long and is full at 11 in. Level meters are shown as yellow rectangles with the letter L in white.

Cooling the liquid helium bath in the magnet can below 4.2 K is achieved by evaporative cooling via pumping on the pumping port (J*). Cooling the magnet from 300 K to such low temperatures requires 5+ days of precooling, and at least one liquid helium transfer directly into its bath (most likely, 2 or more transfers). It is continuously filled during normal operation by the main helium bath. Table 10 details the connections and functions of each important component of the magnet system. Additional details concerning the magnet system and the current injection are detailed in Section A.5. The cryostat body serves as the negative lead and a positive lead must be installed in the bath prior to the cooldown.

Table 10: Components and connections for the magnet system.

Component	Location	Functions	Notes
Pumping line	Top plate: (J*)	<ul style="list-style-type: none"> -Connects to the house pumping system to facilitate evaporative cooling of the LHe bath, allowing the full current to be injected into the coil -Supplies the He gas from the gas board during purging and back-pressurizing -He gas in the pipe while back-pressurizing aids heat transfer between the magnet and the liquid nitrogen in the main bath -Provides pressure relief in the event of a quench and connects to recovery 	<ul style="list-style-type: none"> -The pressure relief plate must be initially sealed by tightening the 3 screws above it -These screws must be loosened once current is injected into the coil
Fill port	Top plate: (L*)	<ul style="list-style-type: none"> -Used to transfer LHe directly into the magnet can 	<ul style="list-style-type: none"> -Generally sealed with a brass plug
Positive lead	Top plate: (E) through the bath to threads in the base of the bath, then through a sealed pipe to the magnet	<ul style="list-style-type: none"> -This port on the bath opens directly above a threaded opening for the positive current lead to be inserted -The lead is hollow to allow for cold He vapor cooling -The top portion of the lead has a quick-connect fitting to connect to the helium recovery line 	<ul style="list-style-type: none"> -Tubes made of G-10 or similar insulating material must be placed around the lead to prevent electrical contact with the cryostat body
Continuous fill	Internal; between the bath and the magnet	<ul style="list-style-type: none"> -This thin connection provides a continuous LHe fill from the main bath when the magnet is being pumped on -Carries the back pressurized He gas during LN₂ precooling 	<ul style="list-style-type: none"> -This connection used to be an externally controlled liquid flow T-valve, but was replaced due to repeated leaks
Stycast cap	Internal: below the 1K Pot	<ul style="list-style-type: none"> -Allows electrically-insulated wires to connect inside the magnet can for thermometers, persistent current switch heater, and liquid helium level meter -These wires exit through the 32-pin connector (III) 	<ul style="list-style-type: none"> -The voltage taps are very thin and frequently detach from the leads

A.2.4 Bath and 1K Pot

The cooling requirements of this facility are the work of the main helium bath and the 1K pot. While the vacuum space provides insulation, the bath provides cooling, and the 1K pot

Table 11: Components and connections to the 1K pot and bath.

Component	Location	Functions	Notes
Pumping port for the 1K pot	Top plate: (H*)	-Facilitates evaporative cooling so the 1K pot can cool the fill lines and cell below 4.2 K -Supplies the gas from the gas board -Provides pressure relief and measurement	-The fitting on this pipe is unique and can damage the pipe if tightened excessively
Vacuum and He recovery port	Top plate: (B)	-Allows the bath to be evacuated during purges -Provides a connection to the He recovery line -Connects to a small valve on the magnet pumping line (J*) for He recovery from the magnet can	-This pipe is bent -Exercise care when manipulating things nearby
Transfer port	Top plate: (A)	-Allows liquid nitrogen and liquid helium to be transferred to the bath	-This port can be used to measure the liquid nitrogen level
Double fitting	Top plate: (C) and (D)	-Used to supply gas from the gas board via (D) -Allows gas exhaust and liquid nitrogen removal through (C)	-This port frequently freezes -Uses a special fitting
Magnet lead	Top plate: (F)	-Allows the lead to be inserted, screwed in, and current to be injected -Provides liquid and vapor cooling for the lead, and connects to recovery	-The lead should be surrounded with G-10 to avoid electrical shorts
LHe level meter	Top plate: (E)	-Measures the level of LHe in the bath	-The bath is full ~15 in.
Continuous fill	Internal capillaries	-One capillary allows a slow, continuous internal transfer of liquid helium from the bath to the 1K pot -The other fills the magnet -Both carry the He gas back-pressure	-The 1K pot's capillary is very thin and very long -The magnet's has been replaced
Heat exchanger	Internal	-This allows heat to be exchanged between the fill lines and 1K pot, and the cell and 1K pot	-The ½K pot is connected but not used

A.3 Operational Procedures

Note that these procedures each assume initial conditions of a fully assembled cryostat entirely at room temperature, opened to the room, and all pumps fully shut down. It is extremely important

to fully read and understand each procedure and all related notes before beginning any steps. Review all relevant schematics for each system periodically and wherever necessary. Prior to starting any procedures, ensure all piping is assembled correctly and tightly. Start LabView to monitor temperature sensors. Check all gas supplies. Close all valves and ensure all plugs are secure. Procedures include: starting up the diffusion pump (Table 12), shutting it down (Table 13), pre-cooling (Table 14), completing the cooldown (Table 15), and warming up (Table 16).

Table 12: The procedure to start up the diffusion pump.

Step & Instructions	Notes
1) Close the valve to the vacuum space (VI), the valve on the roughing pump, and the high vacuum (HV) valve. Place the three-way valve in the backing position and start the roughing pump.	-Double check all clamps and fittings for tightness and proper seals -Grease and clean the O-rings if exposed
2) Open the valve on the roughing pump and evacuate the internal chamber of the diffusion pump until a pressure of 375 mTorr is achieved.	-This pressure is on the analog TC gage
3) Turn on the cold water supply and plug in the heater on the diffusion pump.	-Make sure no cables are exposed to the heater on the pump, or the condensation from the cold water supply
4) Wait at least 20 minutes. Check that the plate is giving off heat, but do not touch it.	-Make sure the diffusion pump is level
5) Place the three-way valve in the roughing position and allow the roughing pump to evacuate the pumping lines to the vacuum space. Slowly open the valve to the vacuum space (VI) until fully opened.	-The roughing pump will make a loud clanging sound if the valve is opened too quickly, and should be partially closed to avoid damage to the pump
6) Once the vacuum space is below 225 mTorr, place the three-way valve in the backing position and slowly open the HV valve. You should immediately see the pressure drop, and the cold cathode gage initialize. Allow these pumps to pump for several hours.	-The base pressure of the diffusion pump at 300 K is around 1×10^{-7} Torr. The vacuum space typically pumps down below 1×10^{-5} Torr, and if no leaks are present, will go to around $5-7 \times 10^{-6}$ Torr. Cryo-pumping will drop to $\sim 10^{-8}$ Torr
7) The vacuum space must stay below 3×10^{-5} Torr. If the pressure exceeds this threshold, abort all cryogen transfers and open all vent ports as the cryogen will rapidly boil as the cryostat loses insulation and begins to collect water condensation and frost	-Always monitor any condensation on the cryostat windows as this gives you an idea of how good the vacuum insulation is

Table 13: The procedure for shutting down the diffusion pump.

Step & Instructions	Notes
1) Close the valve on the vacuum space (VI) and the HV valve. Unplug the heater on the diffusion pump and allow it to cool completely.	-Wait at least 20-45 minutes for cooling before proceeding to step 2
2) Place the three-way valve in isolate position. Close the valve on the roughing pump then switch it off. Slowly open the air admittance valve on the roughing pump to allow air into the hoses.	-Once the analog TC gage shows atmospheric pressure, the air admittance valve may be opened fully
3) Turn off the cooling water supply.	-Condensation may remain for a while
4) The diffusion pump may be opened to the room if needed for changing the oil, but should be left in vacuum if possible. Evacuate it overnight	-If the pump is not left evacuated, it should be pumped for longer in step 2 of the start-up procedure

While precooling, try to periodically check for blockages by stopping the gas supply and ensuring that the pressure in the 1K pot and magnet each begin to drop. A graph of typical precooldown times and temperatures is given below in Figure 36.

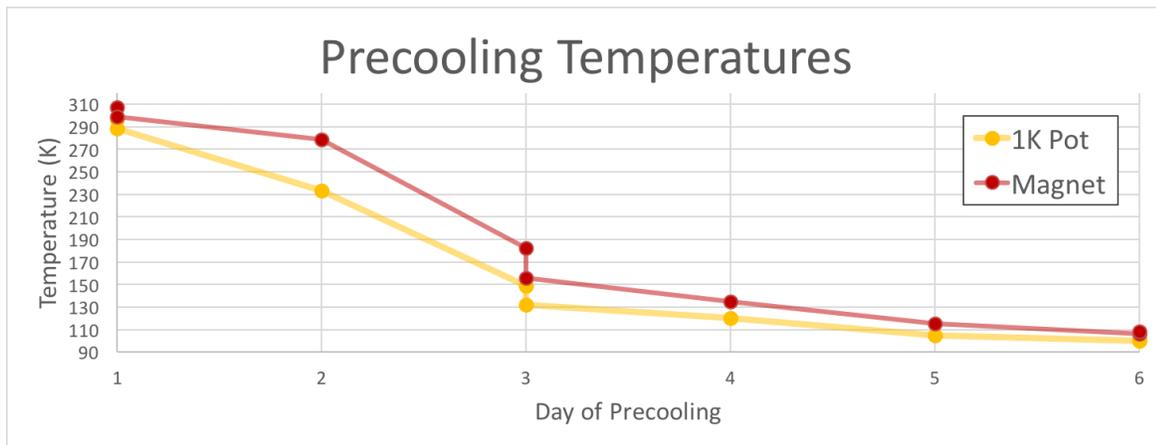


Figure 36: A graph of a typical precooling is shown. It will take 5-7 days before liquid helium may be transferred.

Monitor the pressure in the vacuum space during each transfer. Minor shaking or bumping of the diffusion pump or the cryostat can cause the vacuum to fluctuate. If the pressure is not sufficiently low after pumping down, leak checking is required.

Table 14: The procedure to pre-cool down the cryostat from room temperature to 80 K-120 K.

Step & Instructions	Notes
<p>1) Purge the gas lines, gas board, vacuum lines, vacuum space, bath, 1K pot, magnet, cell, and ½K pot with He gas at least 3 times. Leave everything but the vacuum space and boards pressurized with He gas.</p>	<p>-Allow at least a few hours (preferably, overnight) for purging the fill lines/capillaries to the cell and the capillary from the ³He fill to the ½K pot</p>
<p>2) Begin back-pressurizing the 1K pot and magnet with He gas. Maintain the pressure around 900-1000 Torr. The pressure gauges typically turn off around 1000 Torr. Try to maintain a reasonable balance with the gas cylinder valves, or you will use excessive amounts of helium.</p>	<p>-The gas pressure in the 1K pot and magnet must always exceed the pressure in the bath during precooling -Do not stop back pressurizing the 1K pot until the bath has at least 2-3 in. of LHe and no LN₂ or nitrogen gas remains in the bath or magnet</p>
<p>3) Begin filling the LN₂ jacket. Ensure that the relief tube is correctly attached to a filling port and pointed towards the wall. Make sure the plastic sheet is covering all electronics. Do not allow any components to get wet. Monitor this regularly or damage may occur.</p>	<p>-The LN₂ jacket is full if liquid shoots from the exhaust and if so, remove the fill line and close the dewar</p>
<p>4) Insert the fill line into port (A) on the bath. Attach the gas exhaust pipe to port (C), and point it towards a dewar on the floor. Fill the bath with liquid nitrogen. Allow it to flow and slowly cool the magnet and monitor the temperature.</p>	<p>-The gas in the pumping lines to the 1K pot and magnet will help cool them -The magnet should cool slowly to avoid generating strong thermal stresses</p>
<p>5) Once the bath is full, remove the fill line and seal the port. Remove the exhaust pipe and seal it as well. Monitor the pressure in the bath. Reinsert the transfer line into the nitrogen jacket and leave it.</p>	<p>-Ensure that the vent on the bath is cracked open to allow nitrogen vapor to escape</p>
<p>6) FOR 5-7 DAYS: The nitrogen jacket must be filled at least twice per day, preferably every 10-12 hours.</p>	<p>-It can speed cooling if topped off more frequently for the first few days</p>
<p>7) FOR 2-3 DAYS: The bath should be filled on the first and second day of the cooldown. Sharply diminishing returns will be observed for each subsequent refill. For purging, be sure to keep track of the last bath fill. It will affect how stringent the purging will need to be.</p>	<p>-Make sure all ports are tightly sealed, and all O-rings are defrosted prior to changing any pipes or plugs</p>

Table 15: The procedure for completing the cooldown from liquid 80 K to < 4.2 K.

Step & Instructions	Notes
1) Once the magnet and 1K pot are below 120 K, and preferably closer to 80 K, it is necessary to purge the bath. Insert the liquid purge line into port (C), direct it into a dewar on the floor, and pressurize the bath to between 760 and 1000 Torr. Be careful, as LN ₂ will shoot out of the exhaust. Continue until all liquid has stopped flowing.	-Check the liquid nitrogen level in the bath at any time by inserting a thin G-10 rod into the bath until reaching the bottom -Hold the rod there for a minute, then remove it and quickly wave it through the air several times -A distinct layer of frost will appear on the rod, indicating the liquid level -Keep a higher back-pressure at all times
2) Purge the bath with He gas at least 3 times with pressure slightly above atmosphere but still less than the pressure in the 1K pot and magnet. Evacuate the ½K pot and cell for sufficient time to clear out all capillaries.	-Do not begin transferring helium certain that all the nitrogen is gone -In the event of a blockage, the magnet, bath, and 1K pot must be warmed above 100 K, pumped out completely, and purged with room temperature helium gas several times
3) Once purging has been completed and no blockages are present, close all gas lines except the ones to the 1K pot and magnet and ensure the pumping line to the bath is closed.	-Do not open (A) or transfer liquid helium into the bath if it is not pressurized as this could allow air inside
4) Now LHe can be transferred via (A). As the LHe transfer to the bath begins, open the helium recovery line promptly.	-Monitor the temperature, pressure, liquid level, and vacuum space pressure carefully
5) Once the helium level in the bath has reached at least 3 in., close the gas lines to both the magnet and 1K pot. Next, begin pumping very lightly on the 1K pot.	-The bath will be full around 15 in. -This level ensures no residual nitrogen gas can leak into the 1K pot or magnet
6) Seal the bath and begin a transfer to the magnet, ensuring the recovery valves are open.	-Once the magnet is completely covered in liquid helium (~10 in), the liquid level in the bath will have dropped significantly so move quickly
7) Begin a second transfer to the bath. Once the bath has again gained a significant liquid level, begin lightly pumping on the magnet.	-Monitor the level meters closely -Make sure they are set to sample and not continuous, or helium will be wasted

When the magnet has cooled to 4.2 or 2.2K, depending on the desired field, the current ramp up procedure can be started. Ensure the lead is properly connected and that the persistent current switch heater has 8V and 80 mA so it can be in its open state. Once the magnetic field has reached the desired strength, the valve on the red fill line should be opened to allow helium gas to move to the cell. This gas liquefies while travelling through the bath and cools down further, while

wrapping around the 1K pot. The liquid then travels down a glass capillary into the cell's magnetic trapping potential and a drop is levitated.

Table 16: Warm up procedure for the cryostat.

Step & Instructions	Notes
1) Open all vents to any components with cryogens. Stop topping off the nitrogen jacket. Turn any heaters on and set to low heat, then pressurize any components without cryogens to aid heat transfer.	-If helium is in the bath or magnet, leave the recovery open -A higher pressure in the vacuum space can speed the process, but try to stay in the mTorr range
2) The cryostat will reach ~50-70K within a day of the last liquid helium transfer. It takes a minimum of 3-4 days to warm up to room temperature.	-Make sure the recovery line is closed once all liquid helium has evaporated -The magnet may remain cold for a day or so after it appears to his 300 K
3) Close the valve to the vacuum space (VI).	-Most likely, the diffusion pump will also be shut down, so follow Table 13
4) Place the three-way valve in the roughing position then close the valve on the roughing pump.	-If the diffusion pump is to be left on, the HV valve must be closed, and the three-way valve on roughing or isolate
5) Slowly open the air admittance valve on the roughing pump until the analog TC gage reads atmospheric pressure.	-Opening quickly may damage the gage -Open it fully when at 760 Torr
6) Slowly open the valve on the vacuum space until at atmospheric pressure.	-An audible hiss indicated the rate of air rushing into (VI)
7) The vacuum space is now warm and ready to be opened if needed. Shut down any pumps or instrumentation that is no longer needed.	-Double check the gas cylinders and the pressure on the gas board

A.4 Electronics and Instrumentation

This system has five active temperature sensors. Each temperature sensor interfaces with the instrumentation by 4 wires as depicted in Figure 37 and each wire was soldered to an adapter with 4 sockets. These adapters each connect to two of the numerous double socket cables, numbered 411-423. For each sensor, two wires supply the current to power the sensor, and two wires measure the voltage across the sensor. Additionally, the pins on the external electrical connector are given in Figure 38. To interpret the voltage output of the sensors, a Kiethley 2700 is

used and connected to a computer running LabView 2014. To send current into each temperature sensor, and measure the change in voltage (resistance) with temperature, the pins were mapped as shown in Table 17 and Table 18.

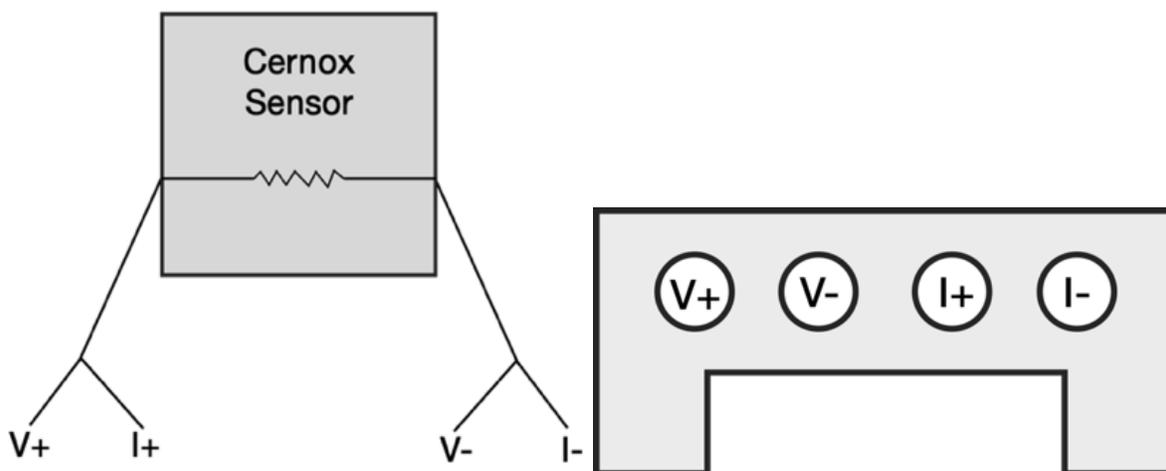


Figure 37: The wiring of a Cernox™ thermometer is shown on the left, and the orientation of these wires as soldered on their internal connectors is on the right. The two downward protrusions represent the shape of the plastic on the internal connectors. Adaptors may be changed as needed.

Pressure sensors used in this cryostat consist of 3 Baratron pressure transducers, 2 TC gages, 1 self-contained analog gage, and 1 cold cathode gage. Two of the Baratron sensors have a range of 1000 Torr and are located on the magnet pumping line and the 1K pot. The third has a range of 100 Torr for increased accuracy at low pressures and is located on the cell's pumping line. This third sensor may be used to obtain a measure of the saturated vapor pressure in the cell, and thus the temperature, if He II is present. All 3 connect to digital display boxes to show their respective pressures. The self-contained analog gage is connected to the pumping line on the main helium bath and is used to observe relative pressure changes. It is not accurate for an absolute reading and bottoms out at 200 Torr, regardless of the actual pressure. The gage on the gas board can be quite accurate however.

The two TC gages are connected to the diffusion pump, one on the roughing pump and one the main line from the diffusion pump to the cryostat, past the high vacuum, "HV" valve. The first is read from an analog meter box, and the second on a digital box that also reads the pressure from the cold cathode gage. The cold cathode gage reads extremely low pressures below 1 mTorr between the HV valve and the vacuum space.

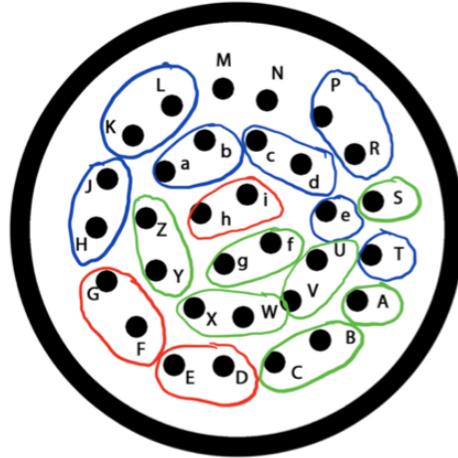


Figure 38: The pins on the external 32-pin connector (III). Blue denotes voltage pairs, green denotes current pairs, and red denotes all other pin pairings. Note that pins M and N appear to serve no purpose. No internal connection for either pin was ever discovered

Table 17: Voltage pins on the 32-pin connector.

Channel	Sensor	Location	Socket	Pins	+/-
1	37	Cell	422	KL	+/-
2	34	1K Pot	423	PR	+/-
3	35	Gold Plate	421	JH	-+
4	33	Top Magnet	416	ab	-+
5	32	Bottom Mag	417	cd	-+

Table 18: Current pins on the 32-pin connector.

Channel	Sensor	Location	Socket	Pins	+/-
1	37	Cell	414	XW	-+
2	34	1K Pot	418	CB	+/-
3	35	Gold Plate	412	gf	+/-
4	33	Top Magnet	413	UV	+/-
5	32	Bottom Mag	415	ZY	+/-

Two liquid helium level meters are present in this cryostat—one in the main helium bath, and one in the magnet can. Each sensor used 4 wires colored red, blue, black, and yellow, connected to a superconducting filament. Their pin mapping is shown in Table 19. By measuring the change in electrical properties of the level meter, the AMI 134 signal display box [22], can indicate how many inches of the level meter are submerged in liquid helium.

Table 19: The pin mapping for the liquid helium level meter in the magnet can. The color column indicates the color of the insulation on each wire as it attaches to the level meter.

Color	Number	Pin	V or I
Yellow	3	e	V-
Red	1	S	I+
Blue	2	T	V+
Black	4	A	I-

It is important to not continuously power the level meter, instead taking measurements as given time intervals, as this can generate significant heat and increase the boil-off rate of the liquid helium being measured. Additionally, the bottom and top 0.5 in. of each level meter are not actually measured by the meter. Typically, it was assumed that until the meter read 2-3 in., the liquid level was not accurate, and could just be an indication of cold helium gas rather than liquid.

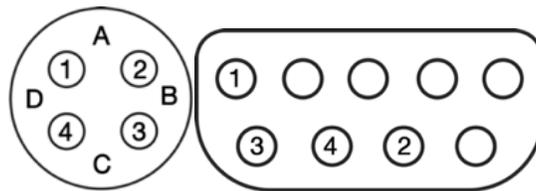


Figure 39: The two ends of the level meter cable for the magnet. The left is the side facing the cryostat and the right is the side connecting to the AMI box. The letters A, B, C, and D are markings on the connector and do not signify anything else.

The main bath’s level meter is 24 in. long, but is effectively “full” when around 15 in. is observed. The original broken meter was replaced with a meter from the cryogenics lab during assembly of the experiment. It feeds through its own port (E).

In the magnet can, the meter is 12 in. long (full at 11 in.) and is hard-wired to pins e, S, T, and A on the 32-pin electrical port (III). Because of this, it was necessary to create a custom cable capable of feeding the individual signals into the correct pins on the connector of the AMI meter box [22] offers the pin details and the signals they carry, and Figure 39 shows the new cable.

A.5 Magnet Information and Operation

The specifications of the magnet from its manual are given in Figure 40.

Measured central field/current ratio	:	0.1378 T/A
Maximum central field achieved at 4.2K during testing	:	> 15.0 T
Quench current achieved at 4.2K	:	109.5 A
Maximum central field achieved at 2.2K during testing	:	> 16.3 T
Quench current achieved at 2.2K	:	> 118.7 A
Guaranteed field gradient product at 2.2K	:	22 T²/cm
Central field for guaranteed field gradient product	:	15.98 T
Maximum field gradient product achieved during testing	:	> 22.5 T²/cm
Resistance values at 300K:		
Magnet resistance Start-Finish	:	48.8 ohm
Superconductor switch resistance (nominal)	:	50 ohm
Switch heater resistance (Ten pin seal "A" - pins J - L)	:	99.5 ohm
Spare switch heater (Ten pin seal "A" - pins K - L)	:	100.5 ohm
Maximum Energisation Voltage	:	4 Volts
(Energisation voltage is limited to 4 Volts by Magnet Protection Circuitry, higher energisation rates will cause the protection circuit to open and shunt current through the protection resistors)		

Figure 40: Magnet specifications from its manual [11].

A.5.1 Current Injection

The superconducting magnet in this cryostat is composed of 8 total windings. The inner 3 are composed of Nb₃Sn and the outer 5 are NbTi. These windings vary in height from 8 cm to 14 cm and are arranged to enhance the field gradient in the 32 mm bore. The flying leads connecting to the magnet through its bath are composed of NbTi and run along the bottom of the magnet can to ensure submersion in liquid helium. To ensure a superconducting state, the magnet must be submerged in liquid helium

Injecting current into the superconducting magnet requires opening a persistent current switch (Figure 41). This switch consists of a superconducting NbTi wire across the terminals of the magnet, which is attached to a resistive heater. When 8 V and a load of 80 mA is applied to the heater at 4.2 K, the persistent current switch returns to a normal resistive state, and the current passes into the superconducting coil. The current is ramped up slowly at around 0.5 A every 10 minutes, until the desired current and field are attained. Normal operation at 2.2 K requires a constant current supply of 118 A to attain a field of 16.3 T. Once the magnet is fully energized, the switch heater is turned off and the persistent current switch returns to a superconducting state causing the current to remain in the coil. Details on magnet and drop procedures will be added as they are developed.

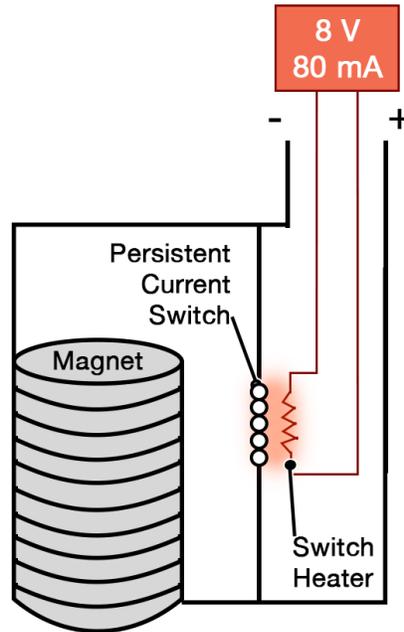


Figure 41: A simple magnet circuit diagram illustrating the persistent current switch and the persistent current switch heater used to “open” and “close” it.

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BIOGRAPHICAL SKETCH

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