High Intensity
Gamma Source

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HIγS Frozen Spin Target

HiFrost Manual

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Preface

HiFrost is nuclear polarized target apparatus consisting of a dilution refrigerator, internal magnetic coil, microwave guide and NMR coil. External components of HiFrost include a polarizing magnet, microwave generating EIO, pump and vacuum system to run the dilution refrigerator, and the Q-Meter/Yale Card set up for running the NMR.

To polarize a target, chemically doped or irradiated beads are placed in the inner-most chamber, the fridge is assembled around the beads and the whole fridge is swung to align with the HIGS gamma beam. The beads are cooled with liquid helium and polarized with a large external magnet, with an EIO providing microwaves necessary for dynamic nuclear polarization.

To achieve frozen spin mode, the internal magnet is ramped on as the external magnet is ramped off, so eventually the external magnet can be powered down and removed while keeping the polarized beads in a steady magnetic field.

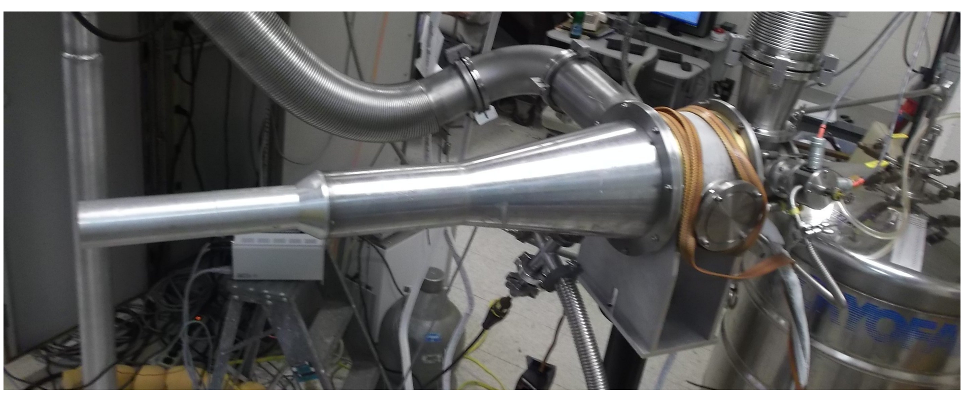

Figure 1: The HiFrost refrigerator during a test cooldown at UVa.

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Chapter 1

Theory of Operation

1.1 Nuclear Polarization

In the presence of a magnetic field, spin-$1/2$ nuclei tend to align themselves along the axis of the field. The polarization of the ensemble of particles is defined by

$$\mathcal{P} = \frac{N_\uparrow - N_\downarrow}{N_\uparrow + N_\downarrow}, \tag{1.1}$$

where $N_\downarrow$ ($N_\uparrow$) is the population of spins with m_z equal to $-1/2$ ($+1/2$). For simplicity, the remainder of this section assumes particles of spin-$1/2$ unless otherwise stated.

1.1.1 Zeeman Splitting

The effect of a constant magnetic field B in the vicinity of an electron, i.e., a simple spin-$1/2$ particle, on its Hamiltonian is

$$H = -\vec{\mu} \cdot \vec{B}, \tag{1.2}$$

where μ is the magnetic moment of the electron and the vector multiplication accounts for the direction of the field relative to the axis of the magnetic moment. Taking both to be in the z-direction, we write the energy shift caused by this change in the Hamiltonian in terms of $\vec{\mu} = \mu\sigma_z$ as

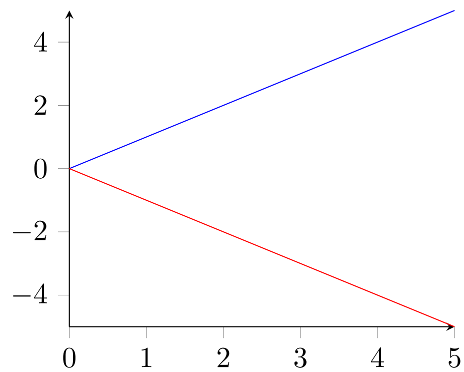

Figure 1.1: Zeeman splitting: TODO label plusorminus lines

$$\begin{aligned} \langle H \rangle &\propto \langle \mu B \sigma_z \rangle && (1.3) \\ &\propto \mu B \langle \sigma_z \rangle && (1.4) \\ &\propto B m_z, && (1.5) \end{aligned}$$

where m_z is the eigenvalue of the σ_z operator and is positive (negative) when the spin is (anti-)aligned with the field. For the spin-$1/2$ particle, the absolute value of m_z is $1/2$.

Figure 1.1 shows the graph of this energy shift as a function of magnetic field. The energy shift is a linear increase or decrease with field strength depending on whether the particle is aligned or anti-aligned.

1.1.2 Thermal Polarization Derivation

A macroscopically sized target has N particles, where N is very large. While any spin has exactly equal probability of being $\uparrow$ or $\downarrow$, it is not given that exactly half of N are $\uparrow$. This is easy to see when considering ten coins dropped on the ground: there is a ≈25% chance that half of them will be heads and a ≈75% chance of another outcome.

The relative odds of the system having a particles $\uparrow$ to the system have b particles $\uparrow$ is interesting. To simplify notation, we give the name M_x to number of ways of getting x particles $\uparrow$ and call it the *multiplicity* of getting x. Now it becomes apparent that the probability P_a of the system having a spins $\uparrow$ is related to the probability P_b of having b spins $\uparrow$ is

$$\frac{P_a}{P_b} = \frac{M_a}{M_b}, \tag{1.6}$$

since a higher multiplicity is associated with a higher probability for each state.

Using $S = k \ln M$ and other thermodynamic relationships (TODO come back and actually work all this out) we arrive at

$$\frac{P_a}{P_b} = \frac{e^{-E_a/kT}}{e^{-E_b/kT}}, \tag{1.7}$$

which is known as the Boltzmann factor that relates P_a to P_b. For the spin-$1/2$ system this gives us the ratios of populations

$$\frac{N_\downarrow}{N_\downarrow} = \frac{e^{E_\downarrow/kT}}{e^{E_\uparrow/kT}}. \tag{1.8}$$

We are now in position to rewrite the definition of polarization (Equation 1.1) in terms of thermodynamic variables:

$$\begin{aligned}
\mathcal{P} &= \frac{N_\uparrow}{N_\uparrow + N_\downarrow} - \frac{N_\downarrow}{N_\uparrow + N_\downarrow} && (1.9)\\
&= \frac{1}{1 + \frac{N_\downarrow}{N_\uparrow}} - \frac{1}{\frac{N_\uparrow}{N_\downarrow} + 1} && (1.10)\\
&= \frac{1}{1 + e^{-\Delta E/kT}} - \frac{1}{1 + e^{\Delta E/kT}} && (1.11)\\
&= \tanh\left(\frac{\mu B}{kT}\right) && (1.12)
\end{aligned}$$

where $\Delta E \equiv E_\uparrow - E_\downarrow = 2\mu B$, the typical difference in energy levels from a system undergoing Zeeman splitting.

1.1.3 Thermal Polarization Values

Equation 1.12 gives the degree of polarization for a proton in magnetic field B and temperature T. With modern cryogenic technology, it is relatively straightforward to cool a target sample to 1 K and, using a superconducting solenoid magnet, magnetic fields as high as 7 T.

Plugging these numbers and the magnetic moment of the proton into the thermal polarization equation yields

$$\begin{aligned}
\mathcal{P} &= \tanh\left(\frac{\mu B}{kT}\right) && (1.13)\\
&= \tanh\left(\frac{(1.4\times10^{-26}\,\mathrm{J/T})(7\,\mathrm{T})}{(1.3\times10^{-23}\,\mathrm{J/K})\,(1\,\mathrm{K})}\right) && (1.14)\\
&= 0.75\%. && (1.15)
\end{aligned}$$

The deuteron's polarization, which has a different dependence on B and T due to being a spin-1 system, can achieve thermal polarizations of

$$\begin{aligned}
\mathcal{P} &= \frac{4\tanh\frac{\mu B}{2kT}}{3+\tanh^2\frac{\mu B}{2kT}} && (1.16)\\
&= \frac{4\tanh\frac{(4.3\times10^{-27}\,\mathrm{J/T})(7\,\mathrm{T})}{(21.3\times10^{-23}\,\mathrm{J/K})(1\,\mathrm{K})}}{3+\tanh^2\frac{(4.3\times10^{-27}\,\mathrm{J/T})(7\,\mathrm{T})}{(21.3\times10^{-23}\,\mathrm{J/K})(1\,\mathrm{K})}} && (1.17)\\
&= 0.02\%. && (1.18)
\end{aligned}$$

While the most modern systems can reach lower temperatures and higher fields, the marginal increase in polarization does not warrant dealing with practical issues of such systems. For example, lowering T from 1 K to 10 mK, a very difficult task, translates to a deuteron polarization of 1.9%.

1.2 Dynamic Nuclear Polarization (DNP)

If allowing the spin system to take on its thermal polarization distribution according to Maxwell-Boltzmann statistics is called *static nuclear polarization*, then disturbing the equilibrium configuration to preferentially flip nuclear spins is called *dynamic nuclear polarization*, or DNP.

1.2.1 Solid State Effect

One DNP process, called the Solid State Effect, uses microwave radiation to optically pump electron-proton pairs to energy states departing from thermal equilibrium (i.e., polarization states). The relaxation time for the electron is much shorter than that of the proton, so the proton remains polarized while the electron is able to be paired with other protons for polarization.

The electrons available for optical pumping cannot be ordinary bound electrons, but must be so-called "free radicals", i.e., excess electrons present in the target material. The two basic ways of introducing free radicals to the target sample are irradiation and chemical doping. The method of irradiation involes putting the target in an ionizing beam line (TODO look up NIST specs for ionizing beam). Chemical doping is adding a measured amount of a special chemical, sometimes a large molecular structure which hosts a single radical, to the target material before freezing into beads with liquid nitrogen.

The nuclei closest to the free radicals will couple more strongly and are more likely to flip during the solid state effect. Nuclei-nuclei dipolar coupling then allows the polarized nucleus to transfer its polarization to another nucleus and become available for polarization by the free radical again.

1.2.2 Equal Spin Temperature Theory

Another DNP process, called Equal Spin Temperature, takes place when free-electron dopants are densely scattered throughout the nuclear sample. When electron spin-spin interactions are no longer negligible, there exist distinct thermal reservoirs for the Zeeman and spin-spin interactions simultaneously. Cooling just the spin-spin system, again with microwave radiation, puts the warmer nuclear reservoir in thermal contact with the cooled electron reservoir and subsequently cools down.

1.2.3 Cross Effect

The Cross Effect occurs when two electrons have differing spin-flip transition frequencies due to inhomogeneities in the target sample (TODO: learn more about what causes these inhomogeneities). If the frequency difference is exactly equal to the spin-flip transition of a nearby nucleon, conservation of energy allows a simultaneous electron "flip-flop" with the flip of a nuclear spin.

The microwave frequency can either be the sum of the proton and electron splitting frequency or the difference, depending on whether the nucleons are to be aligned with or against the magnetic field.

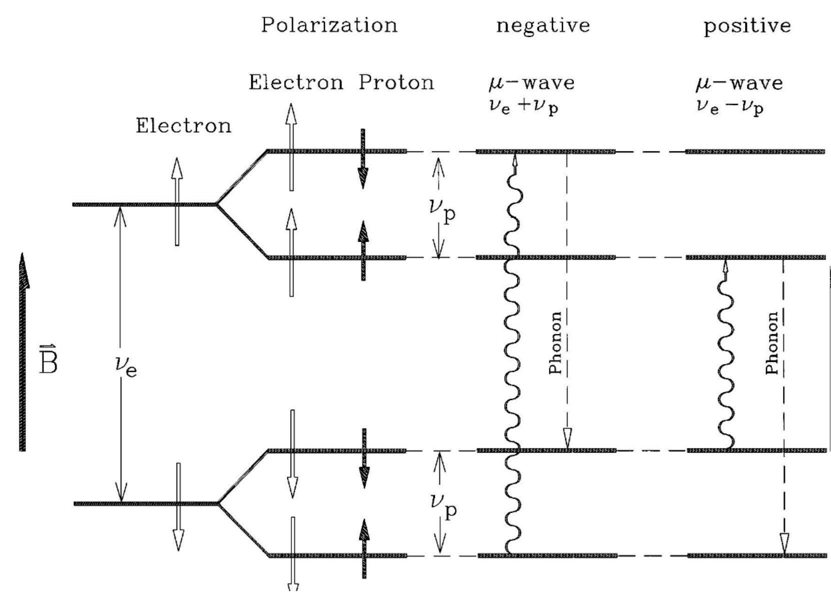

Figure 1.2: DNP diagram [CM97]. The white arrows are electron polarization and the black arrows are proton polarization. Microwaves at a frequency difference $\nu_e \pm \nu_p$ flip the spins of both particles, and the electron's superior lattice coupling ensures it will flip back before the proton.

1.3 NMR

Nuclear Magnetic Resonance (NMR) is the method we use to measure the polarization of the target material. Essentially, a coil in the immediate vicinity of the target acts as the inductor in an LCR circuit, the inductance of which changes as a function of the magnetic susceptibility of the nuclei in the coil. Since the magnetic susceptibility and polarization are related, the resonance frequency of the LCR circuit tell us information about the polarization of the target material[CG80].

1.3.1 Liverpool Q-Meter

The Q-Meter houses the capacitor and resistor for the LCR circuit, as well as detectors that transform the signal into usable data representing polarization[1]. The Q-Meter is entirely encased in gold-plated brass to keep out RF noise from the lab, and all connectors and cables carrying RF signals that connect to the Q-Meter must also be similarly shielded.

LCR Circuit

The *Liverpool NMR Module* is a system for measuring the polarization of polarized nuclei using Q-Meter measurements of their resonant frequency. It uses an LCR circuit with the polarized material contained in the inductor. The resonant frequency of the circuit is calibrated to be the same as the magnetic resonant frequency of the material, given the value of the base magnetic field. The polarized material affects the inductance of the circuit and therefore produces a change in the quality factor, Q, as measured by a device called a Q-Meter, as well as impedance. This change is measured by applying a frequency swept RF voltage to the tuned circuit and detecting the change in impedance as a change in current or voltage. The different elements of the Liverpool Module are arranged in the *Q-Circuit* (Figure 1.3).

Q-Curve

A plot of the RS frequency on the x-axis and the max signal (in volts) seen by the scope is called a *Q-Curve* (Figure 1.4).

This curve can be shown on the scope's XY mode by plugging the RS's input to channel 1 and the diode detector on channel 2. A Q-Curve is

[1] Much of this section is from PTGroup's Lab Overview[DS15]

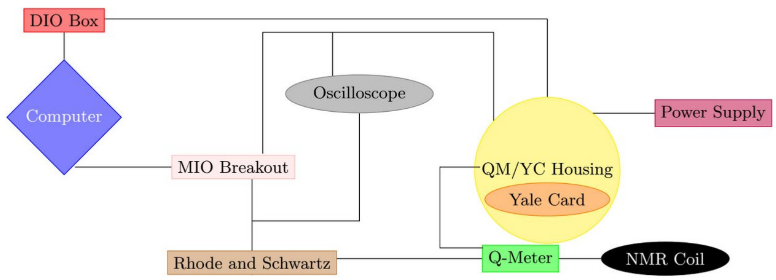

Figure 1.3: Q-circuit schematic

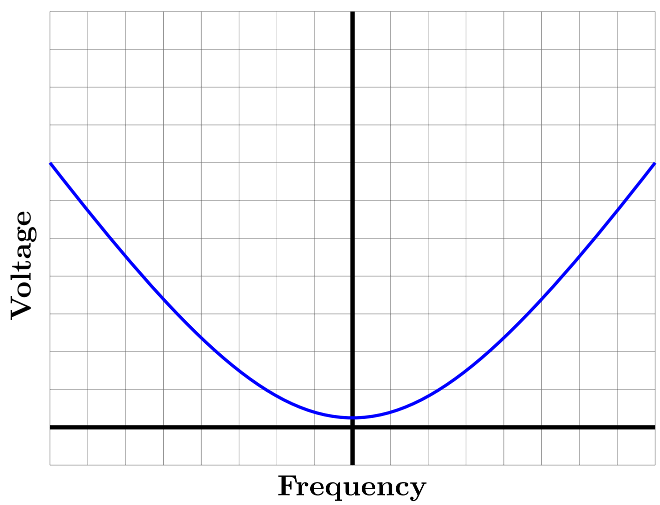

Figure 1.4: A Q-Curve plot with RS frequency on the X-axis and scope (detector) signal on the Y-axis. Compare the left half of this plot with the above triple plot (as RS frequency goes down, scope signal goes up).

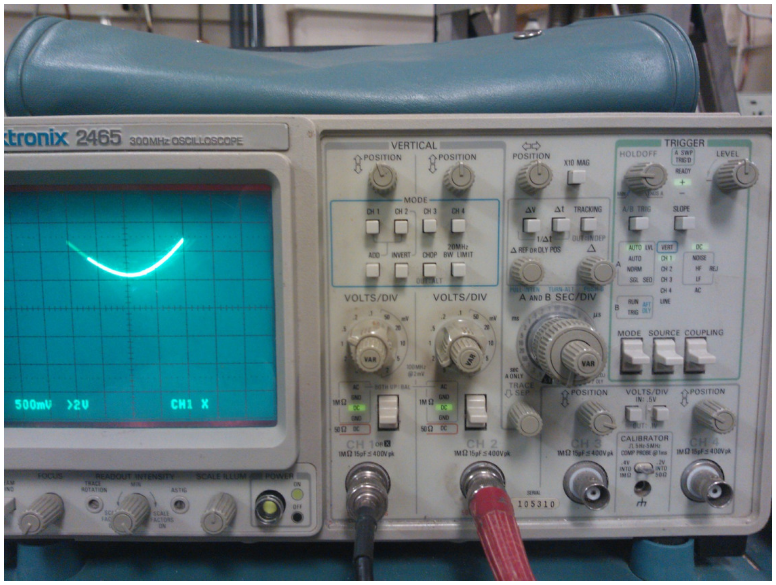

Figure 1.5: The Q-Curve as it is seen on the oscilloscope

referred to as *tuned* when the central frequency of the RS sweep is at the center of the Q-Curve. Since the central frequency is fixed, this is achieved by adjusting L and C of the circuit so the LCR resonance equals the desired frequency.

A Q-Curve tuned to the resonance frequency of a polarized target has a very special quality. When something is placed inside the inductor of the circuit, like an actual polarized target or a "dummy" oscillator chip that absorbs EM radiation at the resonance frequency, we see an aberration (TODO: replace Figure 1.5 with one that has an actual aberration) on the scope where the x-axis marks resonance (Figure 1.5). The more polarized a target is, the larger the aberration will be.

The nature of the aberration is dependent on the nuclear species, the molecular structure the nucleus is in (usually an ammonia or alcohol) and some other factors. In all cases, the aberration arises because the resonance frequency corresponds to a photon of energy equal to the nuclear Zeeman splitting that stimulates emission/absorption from the target material (see Section 1.1.1).

The Q-Curve without the aberration is subtracted from the Q-Curve with the aberration (called a *background subtraction*) and what remains is a bump with an area proportional to the polarization of the target material. In this way, an LCR circuit is used in the NMR setup to measure nuclear polarization.

Diode Detector

The output of the LCR circuit is a sine wave of the same frequency as the RS output. A diode detector is basically a full wave rectifier that smoothes out the magnitude of the resulting waveform (Figure 1.6).

The output can be thought of as a DC voltage comparable to the peak signal from the LCR circuit. Tuning always begins by looking at the diode because it makes finding the correct $\lambda/2$ cable length and Q-Meter capacitance simpler.

Phase Detector and Phase Cable

Once a signal is seen with the diode detector, we switch to a more sensitive detector built into the Q-Meter. The phase detector has a core component called a balanced ring modulator (BRM) that takes two inputs, the LCR signal and the RS signal, and provides one output, an phase-dependent signal that indicates target polarization.

Generally, a BRM multiplies the two input signals and outputs a signal proportional to the phase difference between them. When no polarized target is present, the relative phase between inputs is adjusted to zero by adding electrical path length with a so-called phase cable. When the phase difference is set to zero, the BRM output is proportional to the real part of the LCR signal. For this reason, the phase detector is also sometimes called a "real part" detector. The phase cable is adjusted just like the $\lambda/2$ cable, except it is shorter and both ends are connected to the Q-Meter (see PTGroup's Q-Meter Tune" document for tuning details).

1.3.2 $\lambda/2$ Cable

Because polarization requires that the target material be kept at a very low temperature and the lab's electronics are inoperable at such temperatures, the inductor coil must be coupled to the circuit by use of cables of length comparable to the wavelength of the RF (that is, λ). In this way, the

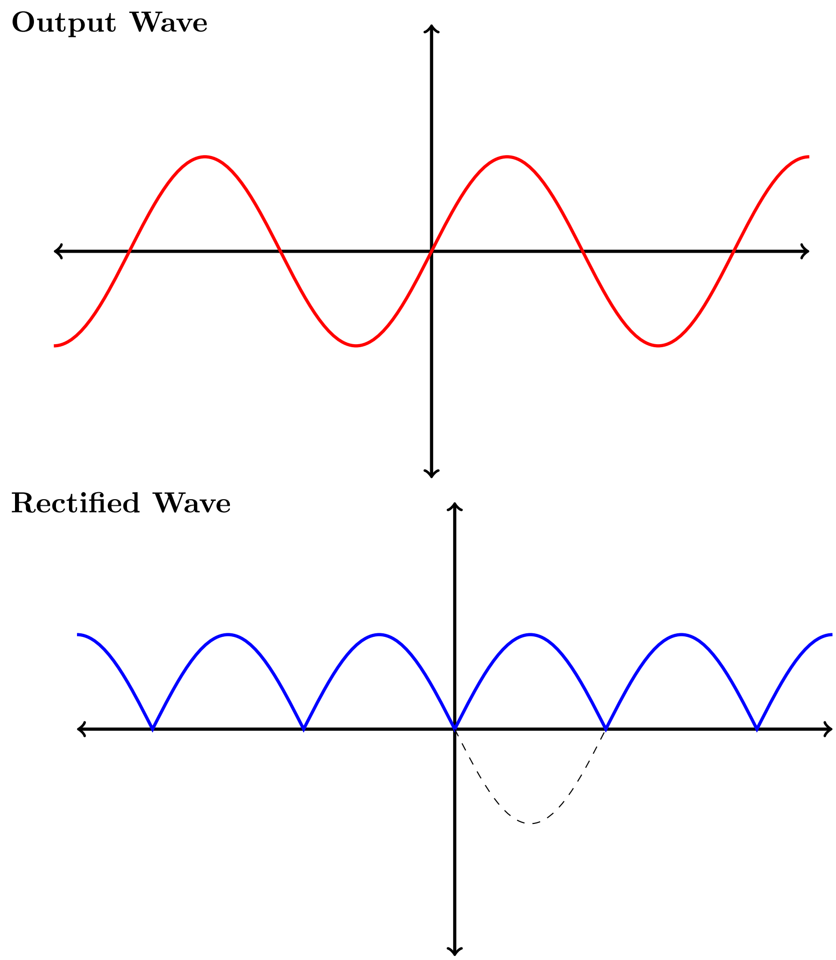

Figure 1.6: The output of the LCR circuit (top) and that same output rectified by a diode detector (bottom)

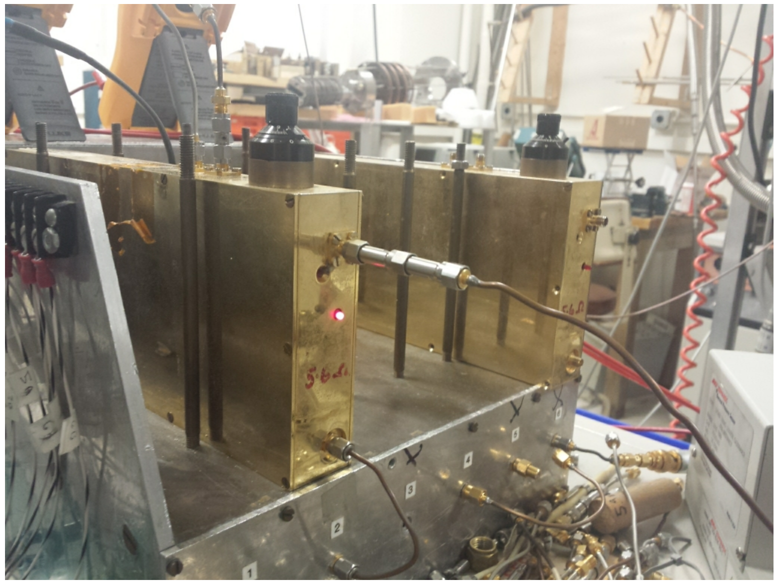

Figure 1.7: A $\lambda/2$ cable attached to the Q-Meter

impedance due to the inductor is the same as it would be if there was no cable attachment.

If the electrical path between the Q-Meter and coil had a length different than n$\lambda/2$, the Q-Meter would see an (input-) impedance larger than the coil's actual (load-) impedance. For our purposes, the load impedance is the actual resistance of the NMR coil, and the input impedance is the resistance the coil appears to be from the Q-Meter. If the Q-Meter is some length l from the coil, then the input impedance it sees is given by

$$Z_{\text{input}} = K\frac{Z_L + K\tanh(i2\pi l/\lambda)}{K + Z_L\tanh(i2\pi l/\lambda)}$$

where K is a constant characteristic of the cable, called its *characteristic impedance*. Clearly, this is at a minimum when the Q-Meter is a multiple of half a wavelength, which is when the input impedance equals the load impedance.

The above is a simplification: the cables are actually lossy so the given equation has an attenuation component, the velocity of light in the cable needs to be taken into account when determining l, fridge temperature and phase-transition temperatures of materials within the cables can play a role, etc. However, these effects are easily corrected for or otherwise tend to be negligible.

In the lab, the length l is already at some minimum value because of the finite electrical path inside the Q-Meter and inside the fridge. In addition to that unchangable length, the Q-Meter is connected to the fridge with a $\lambda/2$ cable. l is adjusted by adding or removing length to this cable, which can be tedious since there is no continuous variable adjustment. Most cables are female-female so must therefore be mated with a male-male barrel, and the RF connectors should be tightened with a special torque-limiting wrench to ensure sufficient electrical contact. Suboptimal connections between the constituent $\lambda/2$ cable components leads to unpredictable and often irrepeatable signals on the scope. Additionally, all cables are "semi-rigid" meaning they have copper shields which make them difficult to bend. The biggest difficulty trying to get the right $\lambda/2$-cable length tends to be finding the right combination of cables that sum to the desired length.

1.4 Frozen Spin

The target is polarized in a 2.5 T field, called the polarizing field, generated from a large, super-conducting magnet (TODO photo). Without this polarizing field, the target immediately loses all polarization due to lack of a quantization axis. Physically, the magnet obstructs the path between the target and detectors, greatly reducing the solid angle available. We wish to employ a method of maintaining target polarization without the polarizing magnet in the way. The method of choice is Hifrost's namesake frozen spin method.

A 0.5 T field, generated from an internal superconducting coil (TODO photo), is ramped up while the target is at maximum polarization. At dilution temperatures, this smaller field, called the holding field, sufficiently maintains nuclear polarization while the polarizing magnet is removed and the detector is placed around the target (TODO diagram).

1.5 Refrigerator

Since the polarization goes like the inverse of temperature, colder environments make for better polarized targets. In our case, we use a dilution refrigerator, one that mixes the two isotopes ^{3}He and ^{4}He for cooling, to reach target temperatures below 0.1 K.

Also, in the case of the frozen spin target, the dilution frigerator is our only recourse to maintain polarization without a large magnet for an amount of time suitable for a nuclear physics experiment.

1.5.1 ^{4}HeCooling (Evaporator and Separator)

1.5.2 Dilution

The dilution refrigerator principle relies on the splitting of a ^{3}He/^{4}He mixture into two distinct phases, a ^{3}He concentrated phase and a ^{3}He dilute phase. The area labeled "Two-phase region" in Figure 1.8 illustrates which mixtures, characterized by ^{3}He concentration, are inaccessible at which temperatures. Since two distinct phases in thermal contact are always in or striving for thermodynamic equilibrium, changing the concentration of the dilute phase (by pumping ^{3}He out of it) will cause atoms from the concentrated phase to cross the phase boundary to restore balance. Since the heat change of mixing (enthalpy difference between the dilute phase and concentrated phase) is positive, the ^{3}He crossing the phase boundary must absorb energy from the surrounding environment, which it does in the form of heat.[Hoc]

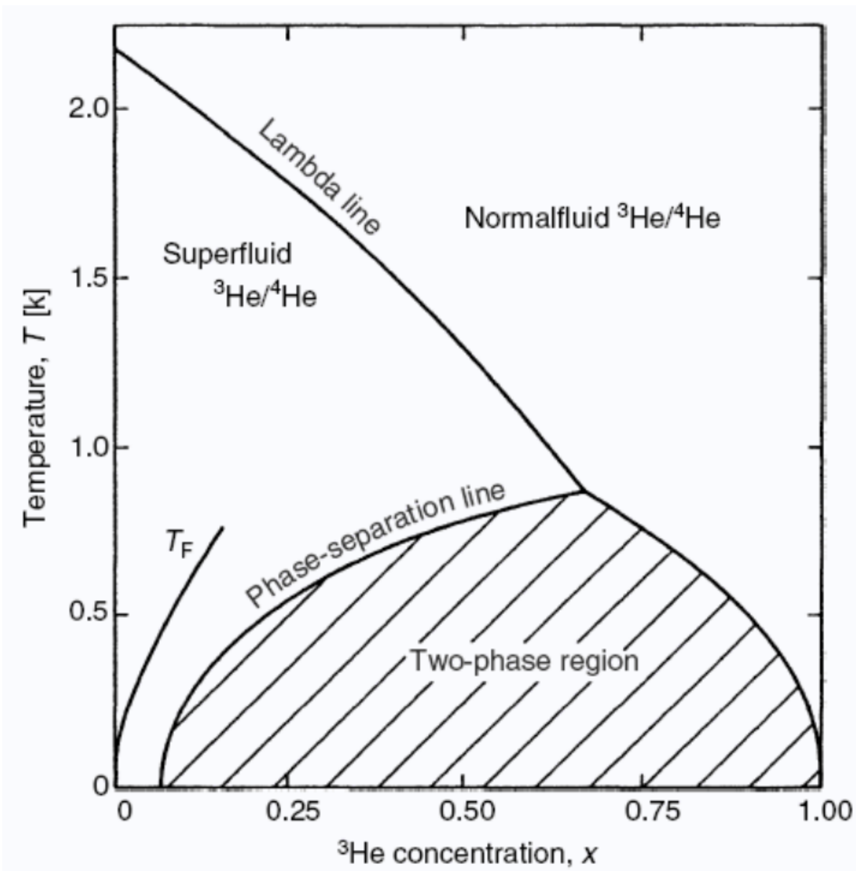

Figure 1.8: The famous diagram that shows the splitting of two distinct phases of ^{3}He-^{4}He mixture. [Bet89]

Chapter 2

List of Hardware

This chapter is an index of all the equipment used in the project. Part and/or serial numbers are included where applicable.

2.1 Fridge

2.1.1 Assembly Tools

OVC

- wire gripping pliers
- 5 mm allen key
- tee pilot pins
- 8x M6x25 triple flange machine screws

IVC

- solder station
- 2.5 mm allen key
- indium waste box
- wooden or hard plastic q-tips
- wire cutting pliers
- isopropyl alcohol
- 6x M3x12 machine screws, 316SS only

Microwave guide support

- flat head screwdriver
- fine tip squeeze-to-open tweezers

Mixing chamber

- mixing chamber customized open wrench
- 1.5 mm allen key
- indium waste box
- wooden or hard plastic q-tips
- wire cutting pliers
- isopropyl alcohol
- 6x M2x12 machine screws and nuts, 316SS only

2.1.2 Heating tapes

- *Briskheat*^{TM} silicone heating tapes, #BSAT051002 (and other various sizes)

2.1.3 Cryogenic Instruments

^{4}He sensors

- 2x *Lakeshore*^{TM} DT-670 silicon diode
- *Lakeshore*^{TM} PT-102 platinum RTD
- *Lakeshore*^{TM} Quad-Lead cryogenic wire, 36 AWG
- *Lakeshore*^{TM} Varnish, IMI-7031

^{3}He sensors

- *Lakeshore*^{TM} DT-670 silicon diode
- *Omega*^{TM} KHLV-0502 Kapton heater
- *Lakeshore*^{TM} RX-202A-AA ruthenium oxide sensor

- *Lakeshore*^{TM} GR-200A-30 germanium diode
- 4-wire Matsushita resistor (model unknown)
- *Lakeshore*^{TM} Ruthenium oxide calibrated sensor, #RX-102B-CB-0.02B

2.1.4 Magnet Leads

- OFHC copper leads
- insulating mylar
- Kapton tape

  Since Chris is out, could you place this Mouser order for three connectors and one roll of cable for me? We need them as soon as possible (each "ships today").
- small flat head screwdriver
- minature flat head screws
- *Solid Sealing*^{TM} KT20678 copper conductor KF feedthrough
- *Solid Sealing*^{TM} FP20106 replacement plug for feedthrough

2.2 Target Material

- cryogenic storage LN_2 dewar
- 1000 mL cold target load dewar (Ted Pella #81740-85)
- open styrofoam tub, about 5-10 liters
- tweezers
- hemostats
- LN_2 transfer dewar
- cryogenic gloves

2.3 Fridge Stand Mounting/Swinging Tools

- 13 mm combination wrench
- long-shaft 3/16″ ball driver
- 10 mm combination wrench
- 5 mm allen key
- fridge lever
- *80/20*^{TM} 5/16″-18 nuts, bolts

2.4 Pump Station Area

- Alcatel or VIC leak checker
- KF-25 plastic blank off
- KF-25 flexible hose
- Adixen 40 HPX oil mist eliminator

2.4.1 ^{3}He Pumps (and oil)

- *Pfeiffer*^{TM} WKP4000 Roots pump (KJLSS20)
- *Alcatel*^{TM} MIV1000 Roots pump
- *Alcatel*^{TM} MIV350 Roots pump (VP1)
- *Alcatel*^{TM} 2063-H mechanical pump (A119)
- *Alcatel*^{TM} ZM2004H mechanical pump (A114)

2.4.2 ^{4}He Pumps

- *Alcatel*^{TM} RSV1002 Roots pump (A120)
- *Alcatel*^{TM} RSV300 Roots pump (A100 or A113)
- *Alcatel*^{TM} 2063-H mechanical pump (A119)

2.4.3 LN_2 Trap

- LN_2 transfer dewar
- cryogenic gloves
- heat gun

2.4.4 Gas System

- 1-1/8″ open wrench for regulator
- 1/2″ fNPT to hose barb fitting for dewar pressurization
- 1/4″ MNPT brass purge valve (Swagelok #B-4P-4M)
- Helium gas

2.5 Vault Pumps

2.5.1 OVC

- *Pfeiffer*^{TM} Hi Cube 80 Eco,PM S03 555 A turbo pump
- *Key*^{TM} flexible 316SS hose, KF25, 77″
- *Pfeiffer*^{TM} PT R25 500 ActiveCold Cathode gauge
- OVC manifold assembly

2.5.2 IVC

- turbo pump
- *Pfeiffer*^{TM} PCR 280, ActivePirani/Capacitance gauge
- *Key*^{TM} flexible 316SS hose, KF16, 78″
- IVC manifold assembly

2.6 Cryogenics

2.6.1 Safety Equipment

- cryogenic handling gloves
- closed toed shoes
- apron
- safety goggles
- ODH monitors

2.6.2 Transfer Lines

Cryofab TLs

- WTL
- UTL
- MTL
- *Cryofab*^{TM} OPE1/2-KF25 SV-9 Operator KF25 pumpout adapter
- LTL

Cern LTL (obsolete March 2015)

- LTL
- LTL pump out adapter
- teflon tape
- flat head screwdriver
- LTL oring *McMaster*^{TM} #9452k26 Buna-N AS568A Dash #114

100LD

- stainless steel QuickDisconnect couplings, .50” (Lesker #410006)

2.7 Vault Electronics Rack

2.7.1 Modules

- 2x *Lakeshore*^{TM} 218S readout
- AVS47 resistance bridge
- *Cryomagnetics*^{TM} Model 1200 S/H level readout
- *Agilent*^{TM} E3646A power supply
- Holding coil power supply
- Polarizing magnet power supply

2.7.2 Patch Panel

- small flat head screw driver
- small philips head screw driver

2.7.3 Thermistor System

- *Omega*^{TM} #SA1-TH-44006-T EIO body thermistor
- *Omega*^{TM} #ON-909-44006 water flow thermistors
- *Newport*^{TM} INFCH-011 thermistor meter/controller
- *MCCDAQ*^{TM} 1608G USB DAQ
- *Omega*^{TM} DP25TH thermistor readout
- *Gems Sensors*^{TM} M103005 (product #02H2390 from *Newark*^{TM}) flow meter display
- *Gems Sensors*^{TM} 155421 water flow meter

2.8 NMR

1. computer with PDP installed
2. *NI*^{TM} BNC-2090 rack mounted terminal block
3. water cooled housing

4. *Bay Voltex*^{TM} HT-5000-AC-RC-VR water chiller
5. power supply
6. DIO panel
7. SMA wrenches
8. *Rhode and Schwartz*^{TM} SMA100A function generator
9. cable from R&S to SMA
10. *NI*^{TM} PCIe-GPIB 778930-01 computer card
11. *NI*^{TM}763061-02 cable
12. *NI*^{TM} PCIe-6321 - 781044-01 computer card
13. *NI*^{TM} 192061-02 cable
14. *NI*^{TM} PCIe-6509 - 779976-01 computer card
15. *NI*^{TM} 182762-02 cable
16. *Teledyne Storm Products Co.*^{TM} $\lambda/2$ semi-rigid cable, #423-0004-001
17. *Omni Spectra*^{TM} RF coax connector, #202-2A (bought through Connector Distribution Corporation)
18. oscillator crystals
19. plastic screwdrivers
20. Q-Meters
21. Yale Cards
22. oscilloscope
23. *Mouser*^{TM} SSMC F connector, #501-7002-1571-003
24. *Mouser*^{TM} Coax cable for NMR, #566-83284-100
25. *Field Components Inc*^{TM} Flexible coax phase cable, #SMAM-RG405TB-SM (and other sizes)

2.9 Microwaves

- EIO
- Cober power supply
- *Phase Matrix*^{TM} 578 microwave counter
- *HP*^{TM} 432A power meter
- *Phasematrix*^{TM} ACC093 mixer
- various microwave guides and attenuators
- 5/64″ ball driver
- 3/32″ ball driver
- water chiller
- *Omega*^{TM} SA1-TH-44006-40-T thermistor
- *Omega*^{TM} DP25-TH thermistor readout
- *Gems Sensors*^{TM} 155421 flow sensor
- *Gems Sensors*^{TM} M103005 M103 series flow readout

Chapter 3

Safety

This chapter is about personnel safety, not equipment safety. Learning to use HiFrost equipment properly without damaging anything is the aim this whole manual. Safety of lab users, however, is important enough to warrant an entire devoted chapter, even if many points are reiterated throughout the document.

Ways to die or become seriously injured working on HiFrost:

- Cryogenic explosions
- Cryogenic burns
- Oxygen depravation
- Electric shock
- Falling
- Hot surfaces
- Chemical exposure

3.1 Cryogenic Safety

Both liquid nitrogen (LN_2, 77 K) and liquid helium (LHe, 4 K) are used during HiFrost operation. The two primary cryogenic safety concerns are over-pressurization (explosion) of cryogenic vessels and cryogenic burns. LN_2 and LHe are each at risk of both, and precautions are taken while handling either.

3.1.1 Equipment

Cryogenic gloves

Cryogenic gloves are lined internally with super-insulation and are long enough to cover past halfway between the wrist and elbow. HiFrost equipment includes large and medium sized sets of gloves, and the best fitting size should always be used. Typical activities requiring use of cryo gloves are filling the LN_2 trap, inserting and removing LHe transfer lines, filling LN_2 dewars for the target loading procedure, and making or manipulating target beads.

Goggles

Wearing googles and/or a face shield while handling open LN_2 dewars is recommended. Goggles also protect users in the vicinity of the large helium gas plumes expected during LHe dewar filling procedures.

Closed toed shoes

The general FEL safety regulations preclude anyone from wearing open toed shoes in controlled areas. Still, there are areas outside the radiation perimeter where cryogens may be handled (e.g., the supply dewar just outside the bay doors), and closed toed shoes must be worn when handling LN_2 or LHe vessels.

3.1.2 Cryogenic Explosions

The principle hazard of handling cryogens is an explosion caused by an enclosed volume of liquid warming up without adequate pressure relief for the evaporated gas. For this reason, every container that holds cryogenic liquid has a pressure relief valve, and most large dewars have a non-configurable, one time use emergency burst disk.

An important exception to the pressure relief rule is the ^{3}He circuit in the dilution refrigerator and pumping system. Due to the scarcity and cost of ^{3}He, it is unacceptable to install pressure relief valves venting to atmosphere. Instead, a single internal pressure relief valve, which opens at about 1.2 bar, is installed on the ^{3}He gas rack and should open if the pressure in the circuit rises due to a plug. The pressure relief valve can only open if the valves leading to the fridge (via the condensor and still lines) are opened in the appropriate configuration. Failure to do this could lead to catastrophic damage to the pumping system or, worse yet, the refrigerator itself.

Three valves rule

The 500LD, 100LD and most supply helium dewars have three pathways for helium to escape in addition to the burst disk. The three pathways are:

1. the pressurization port, where external gas is applied to the dewar
2. a pressure relief valve, usually set around 2-4 PSI
3. the inlet/outlet pathways where transfer lines connect through the top of the dewar

In general, all three of these ports may have manual valves to prevent helium from flowing through them. For example, at the end of a 500LD fill, we close the outlet port on the filling dewar to halt the transfer, and the pressure relief valve is already closed to maintain liquid flow to the 500LD (see Section 4.2.2). The pressurization valve is always closed unless specifically venting the dewar or actively applying pressure to it. The three valves rule is

At least one of the three pathways on a cryogenic vessel must be open at all times.

If all three pathways are closed simultaneously, the radiative heat load (present in all dewars) will warm up the trapped liquid, increasing the pressure on the dewar until the burst disk breaks. If for whatever reason the emergency burst disk fails to operate as designed, the resulting pressure will lead to an enormous explosion, easily fatal to any personnel nearby [Mat06].

3.1.3 Cryogenic Burns

Cryogenic burns may happen by touching a liquid cryogen or cold gas plume, touching a bulk mass that was recently cooled by cryogenic liquid, or inhaling cold gas. Use cryogenic gloves whenever handling liquid cryogens or surfaces they have recently cooled (like transfer lines), and always wear closed toed shoes in the lab.

In the event of a cryogenic burn[Epl14]:

- If prehospital warming is attempted, options include placing the affected area in warm (not hot) water or warming it using body heat (eg, placing frostbitten fingers in the axillae).
- You can flushing the area with tepid water but, in order to avoid tissue damage, a forceful flow of water should NOT be used.
- Never use dry heat. Never apply direct heat.
- Do not rewarm frostbitten tissue if there is a possibility of re-freezing before reaching definitive care. This would result in worse tissue damage.
- Do not rub frostbitten areas in an attempt to rewarm them; this can cause further tissue damage.
- Remove any clothing or jewelry that may restrict circulation to injured area.
- Do not cover the area; leave injured area open to air.

To get help[Epl14], call Duke Employee Occupational Health and Wellness Hotline, available at any time, for assistance with a typical burn, 919-684-8115. A Duke Hospital Operator with answer and then will page the EOHW nurse to your number. During business hours you may also call EOHW main number, 919-681-3136, option 2 and ask for nurse. Call 911 if situation is life threatening. Provide the following information[Epl14]:

- Agent causing cryogenic burn
- Skin tone/color (white, pale, red, or blue).
- Presence of Blisters (yes or no)
- Location and area of injury
- Presence of open skin (yes or no)
- Current treatments

- Any additional past medical information, medications, allergies

- Confirm that the area of liquid nitrogen or hydrogen is secure and safe

3.2 Oxygen Depravation

Work with cryogenics automatically involves an oxygen depravation hazardous (ODH) environment, because the contents any liquid cryogen vessel are capable of displacing many times its volume of breathable air. Generally, liquid helium can displace 1 cubic meter of breathing air for each liter of liquid that is quickly boiled, meaning the 100 L HiFrost dewar can displace 100 cubic meters of air. Additionally, helium is colorless, odorless and tasteless, so it is often impossible for a worker to tell when they are not getting enough oxygen until the symptoms of oxygen deprivation begin to kick in.

The following information and the content of Table 3.1 is taken from Thomas Jefferson National Lab's ODH manual. [TJNL]

Health Effects of Reduced Oxygen Normal air is approximately 21% oxygen and 78% nitrogen. The remaining 1% is mostly argon. Health effects begin at an oxygen concentration of 17%. Oxygen monitors at Jefferson Lab are set to alarm at 19.5%. This advance warning should give ample time to escape the hazard area. The early health effects are difficult to detect so the oxygen monitors are relied upon to give early warning:

3.2.1 Oxygen Monitors in Gamma Vault[Wal13]

Oxygen monitors have been installed in the Gamma Vault, one each at high and low elevations. These measure and display the percentage of oxygen in the atmosphere (20.9% is normal), and will alarm when the percentage falls below 19.5%.

The alarm is not deafening, but is loud enough to be heard by anyone in the room. If the alarm sounds, you should exit by the quickest path available, then prevent others from entering and contact lab staff and me, whoever you can reach most quickly.

Please do not unplug or tamper with these oxygen monitors for any reason. If you think there is a problem, contact the lab staff.

Percent Oxygen	Health Effects
17	night vision reduced increased breathing volume accelerated heartbeat
16	dizziness reaction time for new tasks is doubled
15	poor judgement poor coordination abnormal fatigue upon exertion loss of muscle control
10-12	very fault judgement very poor muscular coordination loss of consciousness
8-10	nausea vomiting coma
<8	Permanent brain damage
<6	spasmodic breathing convulsive movements death in 5-8 minutes

Figure 3.1: JLab ODH manual's list of health effects of oxygen deprivation.

3.3 High Voltage Safety

The EIO power supply (Cober) outputs a maximum 10 kV, 100 mA power across HVlines from the pump station area to the GV. The only interlock on the power supply is on the door that opens the back panel, which immediately turns off all HV.

Figure 3.2: EIO power supply (Cober) and breakout panel rack.

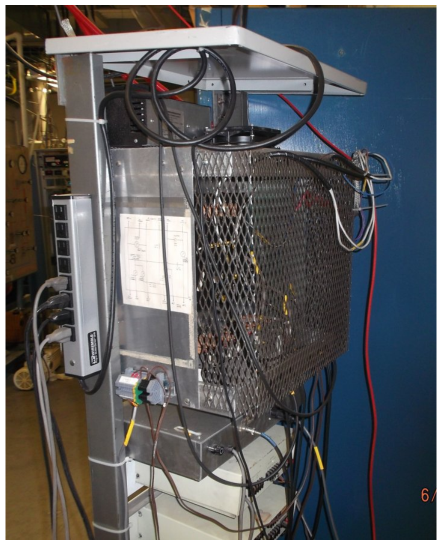

Figure 3.3: Protective cage around EIO breakout panel (rear).

The cage-protected back of the power supply breakout rack is also a risk to personnel, but there is no interlock to kill the HV when the cage is opened. Nothing should be reached inside the cage, and the cage should not be removed, while the power supply is active.

In the GV, the HV wires are enclosed in a plastic red sheath for insulation and visibility. They connect to a breakout panel behind the EIO, which is protected by a transparent plastic cover. There is no interlock on this cover, so it should never be removed while the power supply is active.

The EIO itself is not at HV, and is not an electrical hazard to personnel. However, it is very delicate and should not be worked on with ferromagnetic tools (see magnet safety section below).

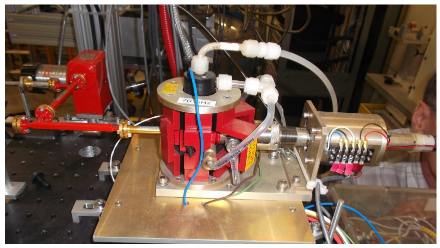

Figure 3.4: The EIO.

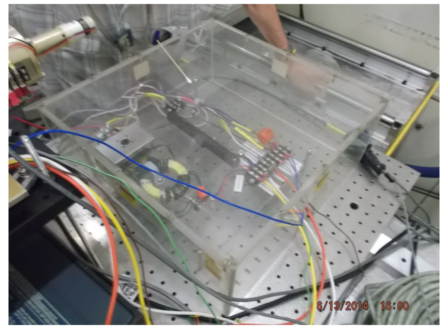

Figure 3.5: The breakout box located behind the EIO in the GV.

3.4 Gravity Safety

No high places should be reached without the appropriate ladder during Hifrost operations or activities. Remember the following 5 rules of ladder safety[OO]:

1. Choose the right ladder for the job.
2. Inspect the ladder before you use it.
3. Set up the ladder with care.
4. Climb and descend ladders cautiously.
5. Use safe practices when working on a ladder.

Additional information ladder safety is provided by OSHA[SA].

3.5 Magnetic Field Safety

Aside from being full of 77 K liquid nitrogen and 4 K liquid helium, the polarizing magnet poses a safety threat by virtue of its 2.5 T field. The 0.5 T frozen spin field is considerably weaker and, due to its localization inside the radiation shield of the fridge, less accessible to personnel and foreign objects.

The most likely danger is a ferromagnetic object (like a wrench) striking personnel as it is pulled towards the polarizing magnet[IC]. The best safety measure against this is making sure Hifrost workers and anyone else in the

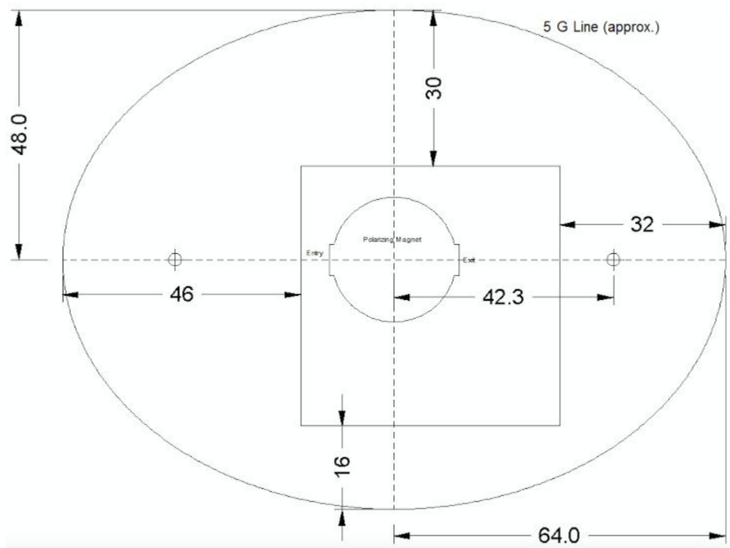

Figure 3.6: Diagram showing the 5 gauss safety line around the polarizing magnet. Units are in cm.

GV know when the magnet is on and do not keep loose ferromagnetic objects on their person.

Anyone with an implanted medical device, such as a pacemaker, should alert Hifrost officials before working on or near Hifrost equipment during a polarizing magnet cooldown. Under no circumstances should they get closer than the 10 gauss line while the magnet is energized without speaking with their physician and the director of DFELL[RWDL]. As shown in Figure 3.6, we have measured and marked the 5 gauss line around the polarizing magnet when it is charged to 2.5 T. This line will be visibly marked on the floor and with caution tape. We also plan to install a flashing sign of Magnet-On at the entrance of the Gamma Vault to alert people who enter.

3.6 Chemical Safety

3.6.1 Heavy elements

Indium

Indium is used for sealing two interfacing sets of flanges in the dilution refrigerator. Disassembling the refrigerator necessarily entails scraping indium off these flanges, and assembling the fridge requires cutting and setting the seal from a roll of indium wire.

While no cases of indium poisoning by oral consumption have been recorded, it remains a heavy element and toxic to humans if it enters the blood stream. As a precaution, wearing latex gloves is recommended while handling indium, and hands should be washed immediately after, especially before taking a lunch break or leaving for the day.

Lead

Lead is a toxic metal often used in the laboratory due to its density and availability. Inorganic lead is not readily absorbed through the skin, but once it enters the body (through ingestion or inhalation) it is carried by the bloodstream to the "bone, teeth, liver, lungs, kidneys, brain and spleen" in high concentrations [Sta98]. Children and pregnant women are particularly at risk to damage from lead poisoning [EPA13]

Lead bricks are used at the University of Virginia PTGroup lab to counter the weight of the fridge on the stand. Lead bricks are also present at HIGS around the beam line and UTR, although they are not usually found in the Vault. Work gloves and hard-toed shoes are recommended when carrying or lifting lead bricks. Any skin that was exposed to lead bricks or lead dust should be washed immediately after performing duties requiring lead exposure.

3.6.2 Isopropyl Alcohol

Isopropyl alcohol is used to clean the indium and KF-oring surfaces on the fridge and in the pumping system. It is generally safe to be exposed to, but if there is a large quantity in an open container (like a bath for soaking vacuum parts) make sure the area is well ventilated and anyone working nearby knows about it. Symptoms of isopropyl alcohol inhalation are dizziness, drowsiness and headache, and may cause unconsciousness [Air].

Isopropyl alcohol has a flash point (the lowest temperature it emits ignitable fumes) of 53° F, and care should be taken not to expose alcohol bottles to open flames.

Chapter 4

Practical Operation

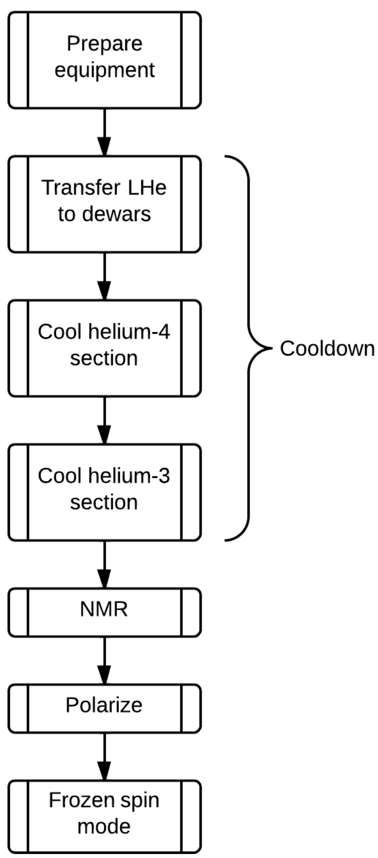

Figure 4.1: Practical steps to polarize target material with HiFrost.

This chapter assumes the reader is familiar with everything safety related. Read Chapter 3 before attempting anything here.

4.1 Cooldown Prep and LHe Filling

4.1.1 Prep

Go down the checklist found in Appendix A, reporting any issues to the senior cooldown scientist.

This step is not optional: the rest of this operational procedure assumes all prep work is successfully completed and equipment bugs are worked out. See walkthroughs for specifics tasks in Chapter 5.

4.2 LHe Transfer

Figure 4.2 is a schematic of the LHe transfer system. More detailed figures of the dewars are found later in the chapter, and complete technical drawings of the TLs are found in Appendix C.

4.2.1 Dewar Filling

The 500LD and 100LD are initally filled before cooling the fridge. The magnet is filled either before or after dilution.

The 250LD has an LHe outlet port, a pressurization/exhaust port and the pressure relief port, shown in Figure 4.3. Each port has an accompanying valve.

The 500LD has an inlet port (for LHe filling and the level probe), an outlet port, a pressurization port, a vacuum pump out port and a pressure relief port, as shown in Figure 4.4. Only the pressurization, vacuum pump out and pressure relief ports have accompanying valves.

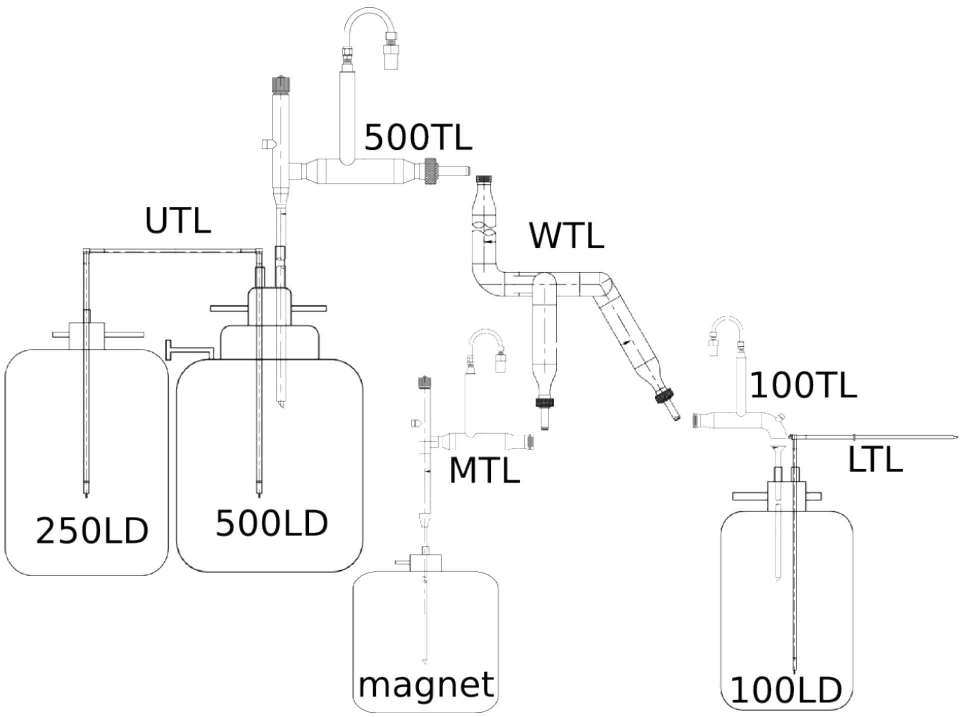

Figure 4.2: Schematic overview of the LHe transfer and storage system.

Figure 4.3: An example of 250LD ports: A) LHe outlet port, B) pressure relief valve, C) pressurization/exhaust port.

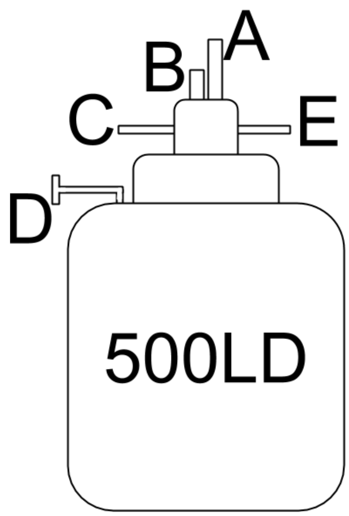

Figure 4.4: The 500LD ports.

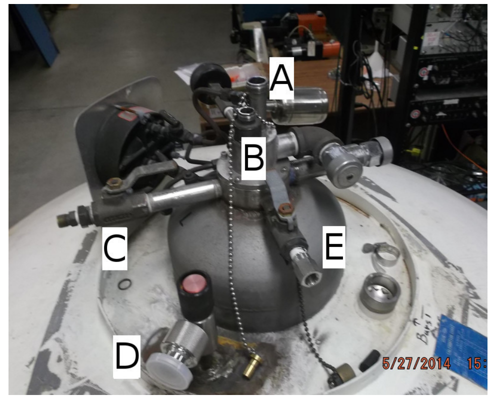

Figure 4.5: Photo of 500LD: A) LHe inlet port, B) LHe outlet port, C) pressurization port, D) vacuum pump out port, E) pressure relief valve.

4.2.2 500LD Fill

1. Weigh a full 250LD according to the procedure in Section 5.5 before breaking the seal on it.

2. Check 250LD level using the procedure in Section 5.12.

3. Place correct Goddard fittings on the 250LD and/or the UTL.

4. Blow out UTL with helium gas by fitting the universal hose over it and putting about 2 PSI on the helium cylinder.

5. [**initial 500LD fill only**] Purge the 500LD with helium gas by hooking up the pressurization line, closing the main flow valve, opening the pressure release valve and putting 5 pounds of pressure on the 500LD for about 10 minutes (Figure 4.7). Remove the pressurization line.

6. Open main flow valve on 500LD to vent pressure (if any), then open pressurization valve making sure the pressurization port is not pointed at anyone.

7. Release the 250LD pressure from the main flow valve and close the pressure relief valve. Slowly lower the correct end of the UTL down into the 250LD about 30 cm or until helium gas is blowing out the

500LD end, as shown in the left side of Figure 4.8. Then, lower the 500LD end in as far as it can go, while continuing to slowly lower the 250LD side at a rate of about 2 cm/s. A cold plume should be coming out of the 500LD. Check that no gas is escaping through the Goddard fittings on either dewar (if it is, replace fitting o-rings).

8. Hook up the pressurization line to the 250LD pressurization port using the procedure in Section 5.13 and open the pressurization valve as shown in the right side of Figure 4.8. There should now be 2 PSI on the 250LD.

9. Periodically measure the 250LD level using the procedure in Section 5.12. When it is empty, stop flow from the pressurization cylinder regulator and disconnect the pressurization line.

10. Simultaneously raise both sides of the UTL out of the dewars and hang it on the wall to warm up.

11. Bung the 500LD inlet port, close the 500LD pressurization valve, and leave open the pressure relief valve.

12. On the 250LD, open the pressure release valve and close the main flow and pressurization valves.

13. Recover the Goddard fittings from the 250LD and bring it back to the weigh area. Record the final weight on the log sheet.

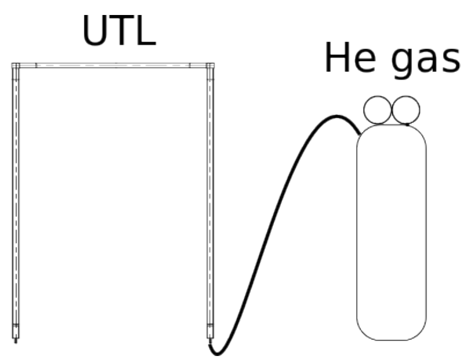

Figure 4.6: Blowing out the UTL with He gas (Step 4).

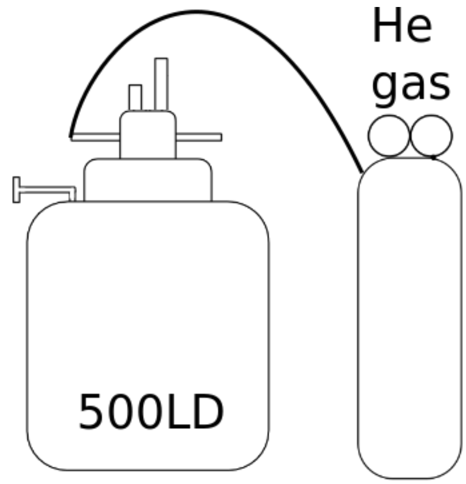

Figure 4.7: **Initial 500LD fill**, purging the 500LD with He gas (Step 5).

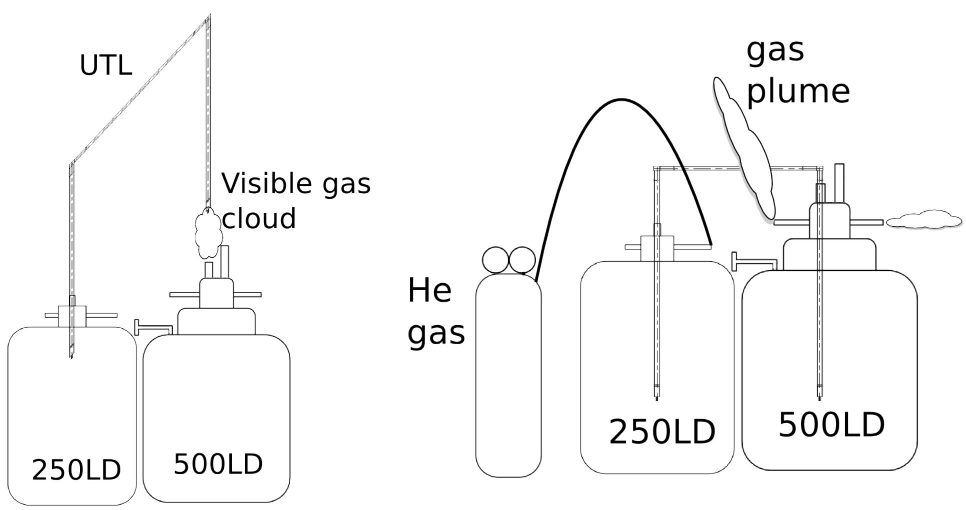

Figure 4.8: Inserting the UTL first in the 250LD, then the 500LD, as described in Steps 7 and 8. The pressurization line is attached to the 250LD to facilitate liquid transfer.

4.2.3 Initial 100LD Fill

The 100LD has an entrance port, a main flow port, an exhaust port and a pressure relief port. The exhaust and pressure relief ports have accompanying valves.

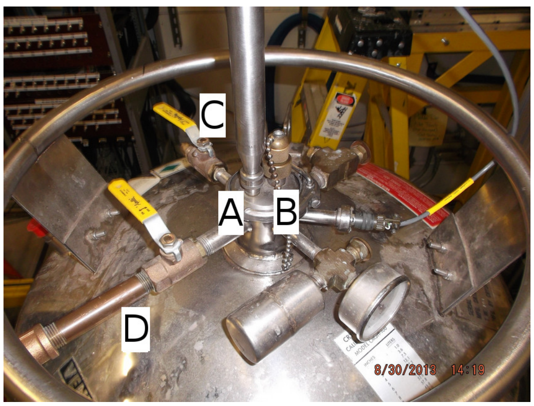

Figure 4.9: Photo of 100LD: A) LHe inlet port, B) LHe outlet port, C) pressure relief valve, D) pressurization/exhaust port.

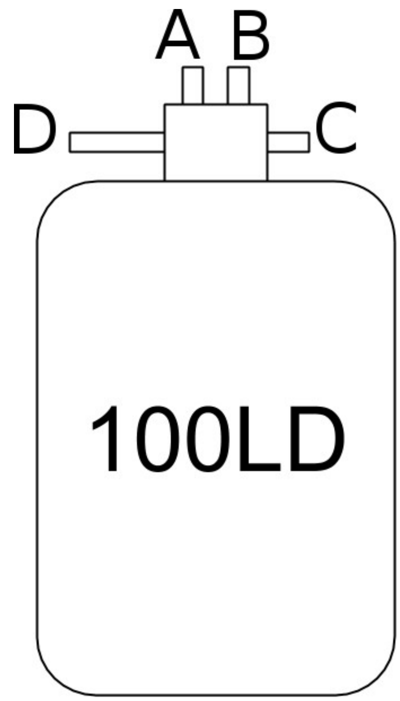

Figure 4.10: 100LD cartoon.

1. Purge the 500TL with helium gas (with the shutoff valve open).
2. Close the magnet LHe inlet valve on the WTL (blue valve).
3. Open the 100LD exhaust valve, close the pressure release valve and bung the main flow valve.
4. Uncap WTL by the pump station area.
5. Hook up the universal He gas line in the GV to the 100LD exhaust port and pressurize to 2 PSI.
6. Verify the positive pressure on the WTL by the pump station. Increase the helium cylinder pressure if necessary. Wait 10 minutes for the 100LD, 100TL and WTL to purge.

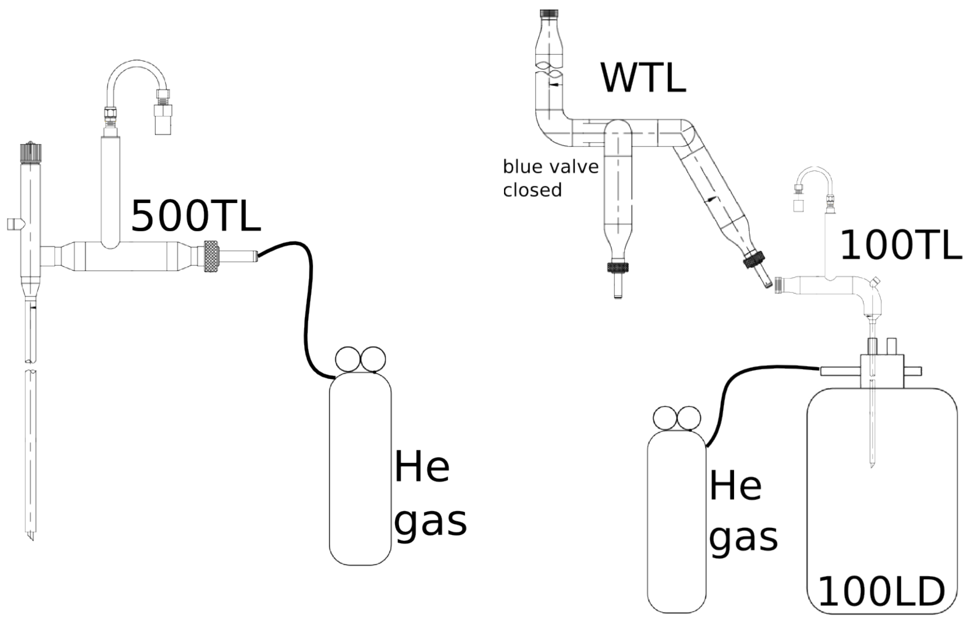

Figure 4.11: Blowing out the 500TL and WTL.

7. Close the 500TL shutoff valve and stop purging it.
8. Vent the 500LD through the pressurization port so as not to freeze the Goddard fittings.
9. Unbung the 500LD LHe outlet port.
10. Close the 500LD pressurization and pressure relief valves.
11. Slowly lower the 500TL about 30 cm.
12. Open the 500TL shutoff valve about half a turn.
13. While watching the gas coming out of the 500TL, slowly lower the 500TL in the 500LD until the bayonet can reach the WTL.
14. Open the 500TL shutoff valve the rest of the way. When the plume looks like a blowtorch, attach the 500TL to the WTL and tighten.
15. **Immediately remove the pressurization line from the 100LD exhaust port and open the 100LD pressure relief valve.** Verify gas is coming out the exhaust.

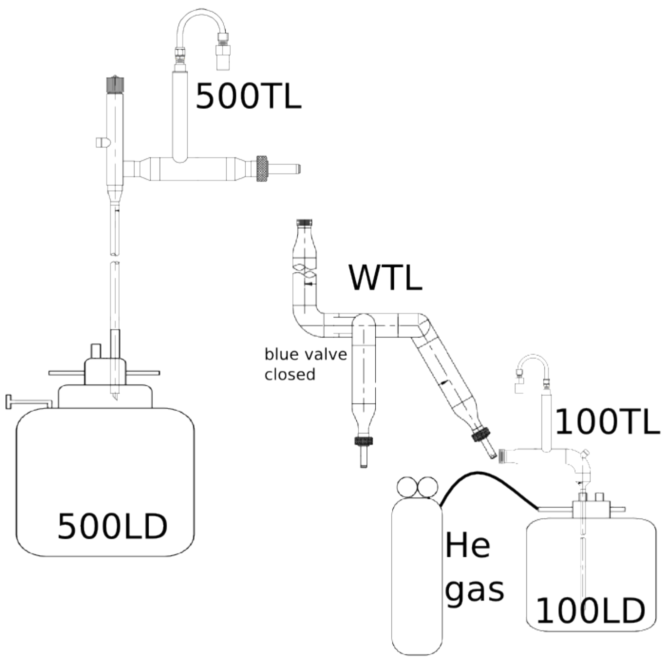

Figure 4.12: Lower the 500TL in the 500LD about 30 cm.

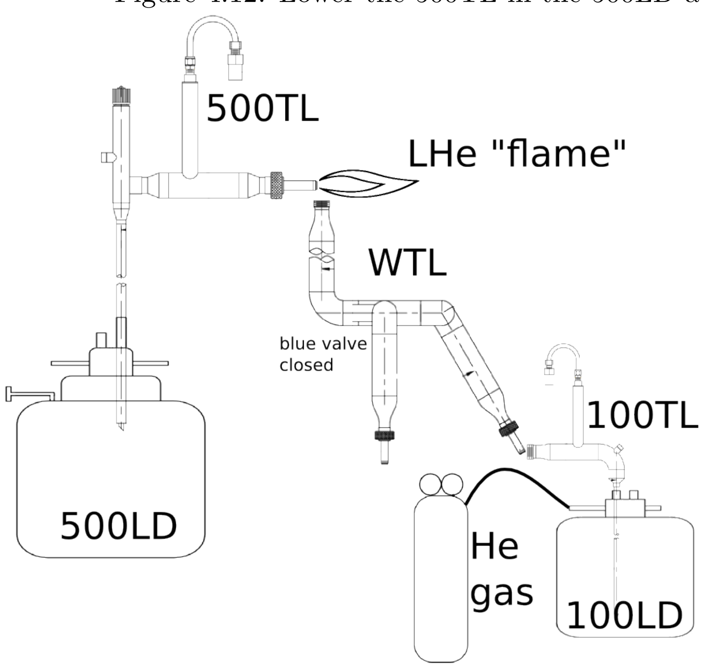

Figure 4.13: Lower the 500TL until the bayonet reaches the WTL.

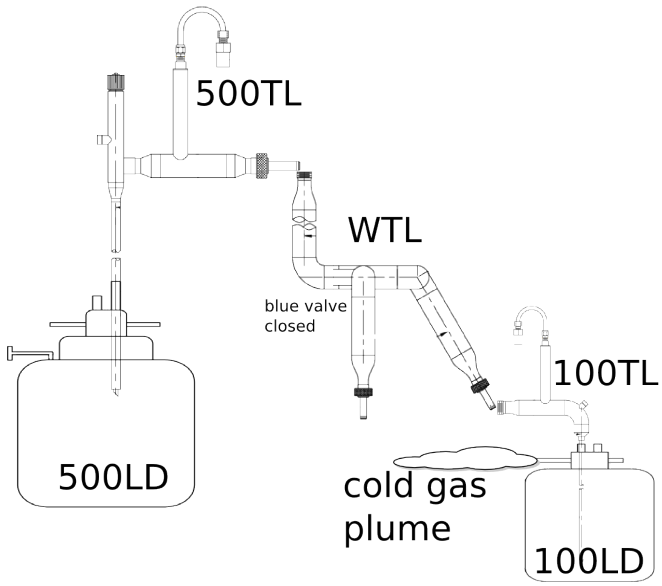

Figure 4.14: Insert the 500TL into the WTL and immediately remove pressure from the 100LD to exhaust the cold gas plume.

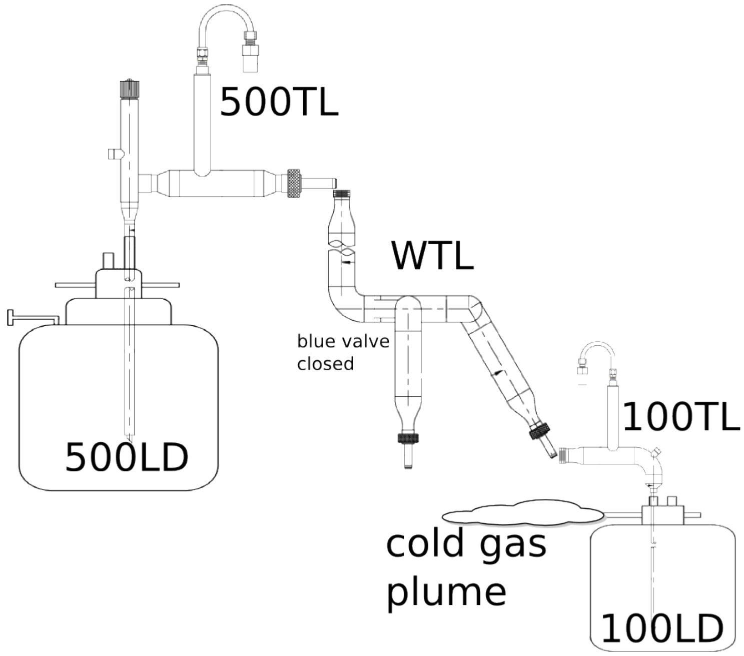

Figure 4.15: Finish lowering the 500TL into the 500LD.

16. Slowly lower the 500TL all the way down into the 500LD and tighten the Goddard fitting. Use the heat gun if the Goddard fitting freezes before the 500TL is completely in.

17. Hook up the pressurization line to the 500LD pressurization port using the procedure in Section 5.13 and open the 500LD pressurization valve.

18. Turn on the 100LD level probe and monitor the liquid level.

19. When the 100LD is full there will be a large white plume coming from the exhaust and the level probe will read around 24.9 (arbitrary units). Close the 500LD pressurization valve.

20. Open the 500LD pressure relief valve.

21. Close the 500TL shutoff valve.

22. Close the 100LD exhaust valve.

4.2.4 100LD Fill During Run

1. Verify the 500LD has enough LHe to top off the 100LD.

2. Set the regulator on the helium pressurization cylinder to 2 PSI.

3. Open the 100LD exhaust and pressure relief valves.

4. Hook up the pressurization line to the 500LD using the procedure in Section 5.13.

5. Open the shutoff valve on the 500TL

6. Close the 500LD pressure relief valve.

7. Open the 500LD pressurization valve.

8. Verify the 100LD Goddard Fittings are tight. If not, remove the pressurization from the 500LD and close the 500TL main flow valve immediately. Consult a high ranking lab official.

9. When the 100LD is full, close the 500LD pressurization valve.

10. Open the 500LD pressure relief valve.

11. Close the 500TL shutoff valve.

12. Close the 100LD exhaut valve.

4.2.5 Cool ^{4}He Section

When the 100LD is full, the fridge has been backfilled with helium gas and the rest of the system is ready for a cooldown, the LTL is inserted into the 100LD and fridge. The separator pumps on the fridge are started and the pressure difference is enough to pull helium from the 100LD (no dewar pressurization needed).

The LHe travels through the LTL into the separator, which collects liquid while rejecting gas. The gas is pulled out through two lines, the "separator" line which precools the ^{3}He inlet, and the shield line which provides a heat shink for the radiation shield.

When the separator is full, control valve 1 (CV1) allows helium to flow to the evaporator and control valve 2 (CV2) directs LHe to the holding magnet and IVC can. CV2 is also used to quickly cool the MC when target material is at risk of thermal damaged.

The evaporator can hold LHe at 4K, but evaporator pumps are run to lower the helium vapor pressure and lower the temperature down to a minimum of about 1K.

4.2.6 Insert LTL

1. See Section 4.1.1 to make sure the fridge is ready to take LHe.

2. Check the 100LD level probe and top off if necessary (see directions above).

3. Blow out LTL with helium gas.

4. Make sure 100LD can roll out from under the fridge stand area (so the LTL can be inserted), back under the fridge stand area, and then up to the fridge. The IVC pump cart may have to be shifted around.

5. Put 4 PSI on the fridge via the following steps: adjust the helium gas regulator to 4 PSI, open the valves between the regulator and the separator manifold, close all other separator manifold valves (so gas does not flow to separator pumps).

6. Remove the LHe inlet bung and KF clamp from the fridge and verify the positive pressure from the separator.

7. Stop purging LTL with gas and place a rubber in the fridge side.

8. Remove bung from 100LD main flow port and slowly insert the LTL until it lines up with the fridge.

9. Roll the 100LD in place under the fridge stand.

10. Remove the LTL bung and wait for the gas plume to look like a blue torch flame, then roll the 100LD towards the fridge until the LTL is fully inserted and tighten the KF clamp around the LTL-fridge inlet joint.

4.2.7 Cool Separator

1. Open CV1 and CV2 about 2 turns each.

2. Immediately turn off the pressurization from the helium cylinder and turn on the separator pumps. Open all valves between the separator pumps and the fridge, including bypass valves. Pump on shield at max flow for 30 minutes and separator for 15 minutes[Nii13].

3. Separator flow is only kept high when there is no liquid in the separator yet; thereafter control the shield flow keeping it below 20 mmol/s and reduce the separator flow to 5 mmol/s (see Appendex B for flow conversion). Normal shield flow is 5 mmol/s in equilibrium (a few hours after cooling).

4. The ^{3}He system should be purged. Close any valves open to the circulation ring (thus making it hermetic) and turn on 2063H1 and open MV1a to pump on the fridge.

5. Open a large vessel of mash (or ^{4}He, for commissioning runs) and bring the condensation line up to 1 bar.

6. Close the large vessel and watch the pressure of the condensation line. The pressure should drop as the helium condenses.

7. When the separator is cold, open the bypass valve to cool the IVC. Monitor the pressure on the evaporator pumps inlet.

8. Backfill the IVC with about 40 mbar of helium gas.

9. Slowly open CV1, keeping the evaporator pump inlet at 1 mar or lower. Continue cooling the MC.

10. ^{3}He flow will be about 1 mbar/s. If the pressure goes up, warm up the fridge and flush with dry helium gas.

11. When the MC reaches 30 K, stop separator flows, close both needle valves and pump exchange gas out of the IVC. The IVC will be pumped on for the rest of the cooldown. Monitor the IVC pressure gauge for signs of a leak in the MC.

12. After 15 minutes of IVC pumping, restart the separator flows and slowly open the needle valves (there is no rush to reach 1 K). Monitor the pressure in the condensor line and add more gas if it gets below 500 mbar. A sudden drop of pressure at the ^{3}He pump inlet indicates the helium has condensed in the heat exchanger of the evaporator.

13. Condense the rest of the mash from the ^{3}He vessel. The MC will cool rapidly to 500 mK if there is 1% ^{3}He in the mixture.

14. Put about 30 L ^{3}He gas into the circulation loop; a reasonable starting ratio is 260:30 L of ^{4}He:^{3}He.

4.2.8 Cool ^{3}He Section

4.2.9 Dilution

4.3 Polarization

4.4 NMR

4.5 Frozen Spin

Chapter 5

Procedures

5.1 Evacuating Transfer lines

Helium transfer lines (TLs) are vacuum insulated tubes. One end is plunged directly into a helium dewar while the other delivers liquid helium to a vessel, with a pressure differential driving the helium through. If the vacuum jacket is compromised, the helium will cool the outer shell of the TL and frost up (helium will of course stop transferring). Once this happens, the only solution is to remove and warm up the transfer line and evacuate the vacuum space.

TLs are evacuated with a leak checker until the leak rate plateaus. It is recommended to pump overnight, but in a bind it can be done quicker. An evacuated TL will be fine for months, but in practice it is safer to pump down each TL a few days before beginning a cooldown in case the vacuum space was somehow compromised in storage/transport.

5.1.1 Cryofab TLs

Cryofab TLs have a proprietary vacuum pump out ports which require a special adapter to KF (Figure 5.1).

To pump out the vacuum space:

1. Warm up the LD.

2. Set the TL on the ground.

3. Loosen the brass fitting on the adapter (do not loosen all the way or the o-ring will fall out).

Figure 5.1: The Cryofab transfer line vacuum space to KF-25 adapter with stem pushed all the way in. The brass nut fits a 1-3/16″ wrench.

4. Pull the adapter stem out about an inch and fit the adapter on the TL pump out until almost the entire vacuum jacket pump out is covered (Figure 5.3).

5. Tighten the brass nut.

6. Support the weight of both ends of the TL on stable objects and attach the KF adapter to the leak checker (we use a chair and lab bench in Figure 5.4). Do not allow the TL to torque about the adapter or the TL and adapter may both be damaged.

7. Push the stem in.

8. Screw in the stem about 5 turns (count them if this is your first time) while gently pushing in so the threads grab. The threads are tightening when the stem is pulled in slightly each turn.

9. Gently pull the stem out half an inch. **If you pull too far you will break the adapter; see Figure 5.5.** If TL was under vacuum, you will hear a hiss indicating the vacuum jacket is vented.

10. Start the LD pump and wait for 2 minutes. This should bring the LR to a low enough value to detect an external leak. Spray both vacuum connections on the adapter with helium.

11. If the KF joint leaks, clean it and try again. If the TL-to-adapter joint leaks, either a) the adapter is not entirely over the TL pump out port or b) the brass nut is not tight enough. Try again.

12. Pump until the LR stops declining, preferably overnight.

13. Again, spray both joints with helium gas. Start over and remake the offending connection if necessary.

14. Flow helium gas through the TL (if there is a valve like Figure 5.2, open it first). If there is a leak between the inner cavity of the TL and the vacuum space the TL is broken and not suitable for a cooldown.

Athe end of leak checking, use the following procedure to disconnect the TL from the LD:

1. Push the stem straight in all way.

2. Vent the LD. If there is a long hiss, the stem was not pushed in all the way and the vacuum space is now filled with air. A short hiss indicates only the small volume in the KF adapter was vented (this is good).

3. Unscrew the stem. If you counted the number of turns in the above section, you know how many times to unscrew it. Otherwise, just turn a few dozen times to be sure. Not entirely unscrewing the stem will result in the TL being entirely vented in the next step.

4. Pull the stem back about an inch. **If you pull too far you will break the adapter; see Figure 5.5.** You should not hear a hiss.

5. Disconnect the KF connection from the LD and set the TL on the ground. This may require two people to do safely depending on the weight of the TL. Be careful not to bump the TL.

6. Loosen the brass nut about two turns and wiggle the adapter off the TL. Unscrewing the nut too much will make the connector fall apart; **unscrewing too little will break the adapter and/or TL if you try forcing it off**. Do not try forcing it.

5.1.2 LTL

The LTL, named for its shape (Figure 5.7), was designed at CERN. It did not come with a vacuum pump out adapter, so we made one by modeling the brass screw that plugs the pump port.

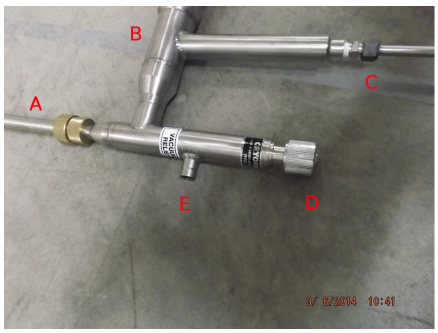

Figure 5.2: A: to TL stinger; B: to TL bayonet; C: emergency popoff valve; D: LHe flow valve; E: vacuum jacket pump out.

Figure 5.3: Adapter on vacuum jacket pump out.

Figure 5.4: Stem pushed in.

Figure 5.5: Stem pulled out.

Figure 5.6: The LTL adapter.

Figure 5.7: The LTL.

Gather the following:

- LTL
- KF adapter to LTL
- teflon tape
- flathead screwdriver
- leak detector (LD) with KF25 oring/clamp
- ziplock bag

1. Warm up the LD.
2. Wrap the KF adapter's threads with one or two layers of teflon tape. Do not block the tapered end with tape or it might ruin the seal.
3. Remove the top screw from the LTL and place it in the ziplock bag.
4. Open side screw valve 3 turns to break the vacuum. If there is a hiss, the LTL was holding a vacuum.
5. Hand tighten the adapter in the top hole while being careful not to let the emergency popoff valve fall out. It might be tricky to get the threads right with the tape between them, but do not strip the threads!

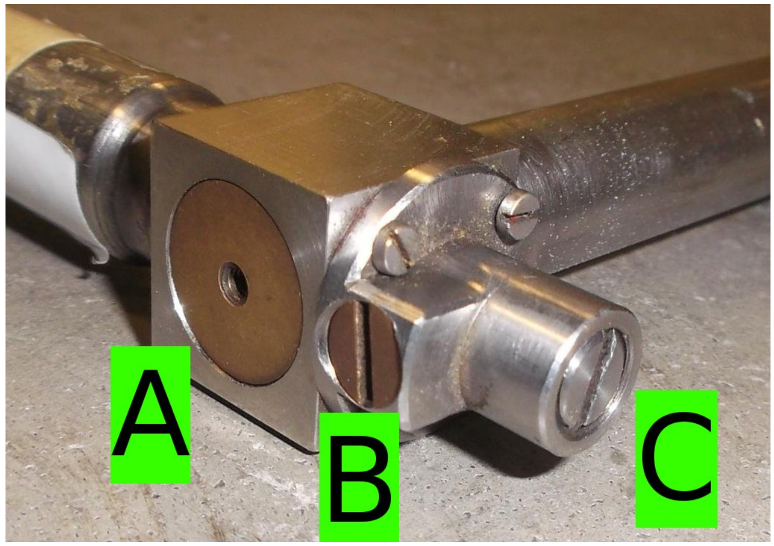

Figure 5.8: The three ports on the LTL: A) emergency relief popoff (this will shoot very high when the LTL freezes!), B) vacuum jacket pump port and brass placeholder screw, C) side screw valve.

6. Start pumping with the LD. The leak rate should should very quickly approach 10E-8.

7. After a few minutes of pumping, spray the KF and threaded joints with He gas to make sure the connection is leak tight. If the threaded joint is leaking, **carefully** rotate the LTL 1/8 of a turn, up to 1/4 turn max. If the joint is still leaking, the teflon tape will have to be removed and reapplied.

8. Pump overnight.

9. Close side screw valve and tighten.

10. Stop and vent LD and break the KF connection.

11. Unscrew the LTL adapter, making sure there is no teflon left over in the LTL port, and screw the brass top screw back in place.

12. Remove all teflon tape from the adapter.

Figure 5.9: Pumping down the LTL. The side screw valve is opened to allow the LD to pump the vacuum jacket.

5.2 Indium Extrusion

Hifrost requires indium wire precisely 1 mm in diameter for making MC and IVC seals. Indium seals must be scraped away after use, and the metal is always recovered (due to its scarcity). The Indium Association of America charges roughly $1000 for 13 feet, or about $20-30 per attempted seal, so it is worth creating our own wire from the so-called "scraps".

Indium extrusion is the name of the process that transforms bulk indium into nicely shaped wire.

5.2.1 Background

Indium scraps or ingots are put into a hollow cylinder with a small hole in the bottom, and the indium is softened with heat and pushed through to make a 1 mm wire. The melting point of indium is about 150° C, but approaching this temperature leaves the indium "runny" and impossible to shape. We have found heating the cylinder to 100° C with a heating tape yields the ideal consistency.

Our first indium die, the plate at the bottom of the hollow cylinder that has a small hole for shaping indium, had a 1 mm hole. For whatever reason, this produced indium 0.75 mm in diameter (too small for the HiFrost flanges). We increased the die to 41 mils (about 5% larger), and the indium wire came out at 1 mm.

When extruding the indium, a varying force on the hydraulic pump sometimes leads to uneven wire (something like sausage links). To prevent this, a continuous, non-varying force should be applied for an entire press.

5.2.2 Materials

The **extrusion cylinder** is an aluminum cylinder with six 1/4-20 bolt holes tapped in the bottom. The inside of the aluminum is lined with another hollow cylinder made of steel, which precisely matches a **steel rod** that slides in and out. If the rod and inner cylinder do not precisely fit, either the rod will not fit in the cylinder or the indium will leak out during the press. There is a small hole drilled in the side of the aluminum for a thermocouple.

Screwed into the bottom of the extrusion cylinder is the **indium die**, a plate with a hole through the middle to push indium through. The entrance hole in top of the die matches in the extrusion cylinder, and the exit hole in the bottom of the die shapes the indium wire. The die is a separate component from the extrusion cylinder for cleaning and modularity reasons.

Indium is generally recovered from scraps of old seals or ingots are bought from the Indium Association of America (currently a pound costs $300). Ingots should be cut with a knife into smaller blocks to fit inside the extrusion cylinder.

The **extrusion stand** is a mounted, horizontal bar for the hydraulic press to push against so pressure is applied to the extrusion cylinder. There must also be a hole in the base of the stand for the pressed indium wire to be collected.

A **thermocouple** sensor is used with an accompanying **thermocouple readout** to measure the temperature of the extrusion cylinder.

Heat tapes warm the indium so it may be pressed. The tapes should be long enough to wrap around the cylinder to warm it evenly.

A layer of **aluminum foil** is wrapped around the heating tapes to keep the heat from dissipating away.

Latex and clothe gloves are for personnel safety and keeping the indium sanitary.

A **variable alternating current** unit adjusts the temperature of the heating tape.

The indium is extuded onto sterile ***Absorbond Wiper Sheets, TX^{TM}*** 9″x 9″, 409, and kept for storage.

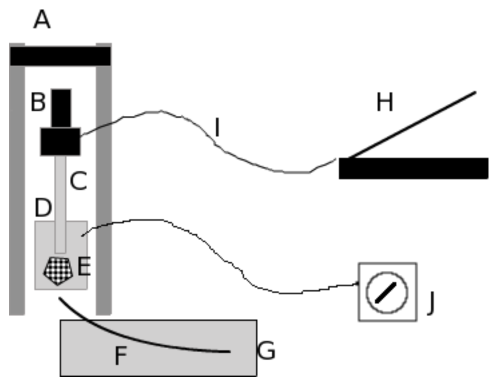

Figure 5.10: Indium extruder setup: A) extruder stand, B) hydraulic pump, C) steel rod, D) extrusion cylinder, E) indium scrap/ingot fragments, F) finished wire exiting die, G) sterile wipe sheet, H) hand jack, I) hydraulic line, J) thermocouple gauge

An *Enerpc*^{TM} P39 Porta Power **hydraulic hand pump** provides the force for extrusion.

5.2.3 Extrusion process

1. Prepare connections for heating tape and thermocouple gauge and readout.

2. Remove steel rod from extrusion cylinder and insert blocks of indium.[1] Place the steel rod back in the cylinder.

3. Wrap the cylinder with heating tape and aluminum foil.

4. Place the cylinder/rod under the extrusion stand.

5. Set the variable current source so the heating tape reaches about 70° C, which takes around 20 minutes. Increase the temperature in steps, allowing the thermocouple to equilibrate between steps, to 100° C. *NB: on our VariAC unit the 50 mark corresponds to* 75° *C and the 60 mark corresponds to* 100° *C*

6. Set the hydraulic jack on the extrusion cylinder and under the stand's horizontal bar.

[1]After losses, 100 grams of indium provides approximately 10 feet of wire instead of the theoretical 50 feet.

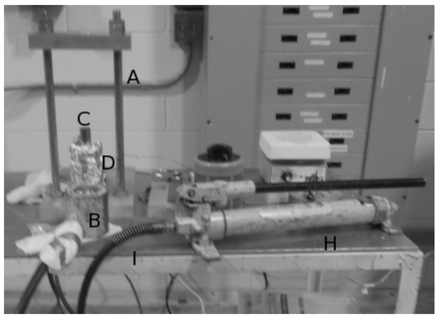

Figure 5.11: A photo of the setup.

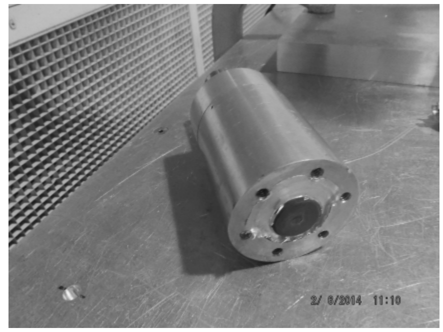

Figure 5.12: The extruder cylinder without the die. The steel rod is plunged all the way down and leftover indium can be seen on the surface.

7. Use the hand jack to slowly push the indium through the extrusion cylinder and out the die. The press takes about 5 minutes. A second person slowly winds the indium wire on the wiper sheets.

5.3 Making Indium Seals

1. First, clean both indium seal flanges.
2. Measure the diameter of the indium to be used; it should be 1 mm
3. Cut a length just greater than the circumference of the flange (MC: 158 mm; IVC: 271 mm) plus a centimeter or two. Do not allow the indium wire to touch any dirty surfaces.
4. Wrap the wire around the proper aluminum setting tool (Figure 5.15 for MC).
5. Using a razor blade, cut the wire at an angle as in Figure 5.14, firmly placed on the aluminum block.
6. Flip the mating can (IVC or MC bell housing) upside down and press firmly to the aluminum block (Figure 5.16).
7. Turn the parts so the aluminum block is on top, and carefully remove the block so the indium is transferred to the can. Check to make sure the wire was not shifted, especially where the two ends meets.

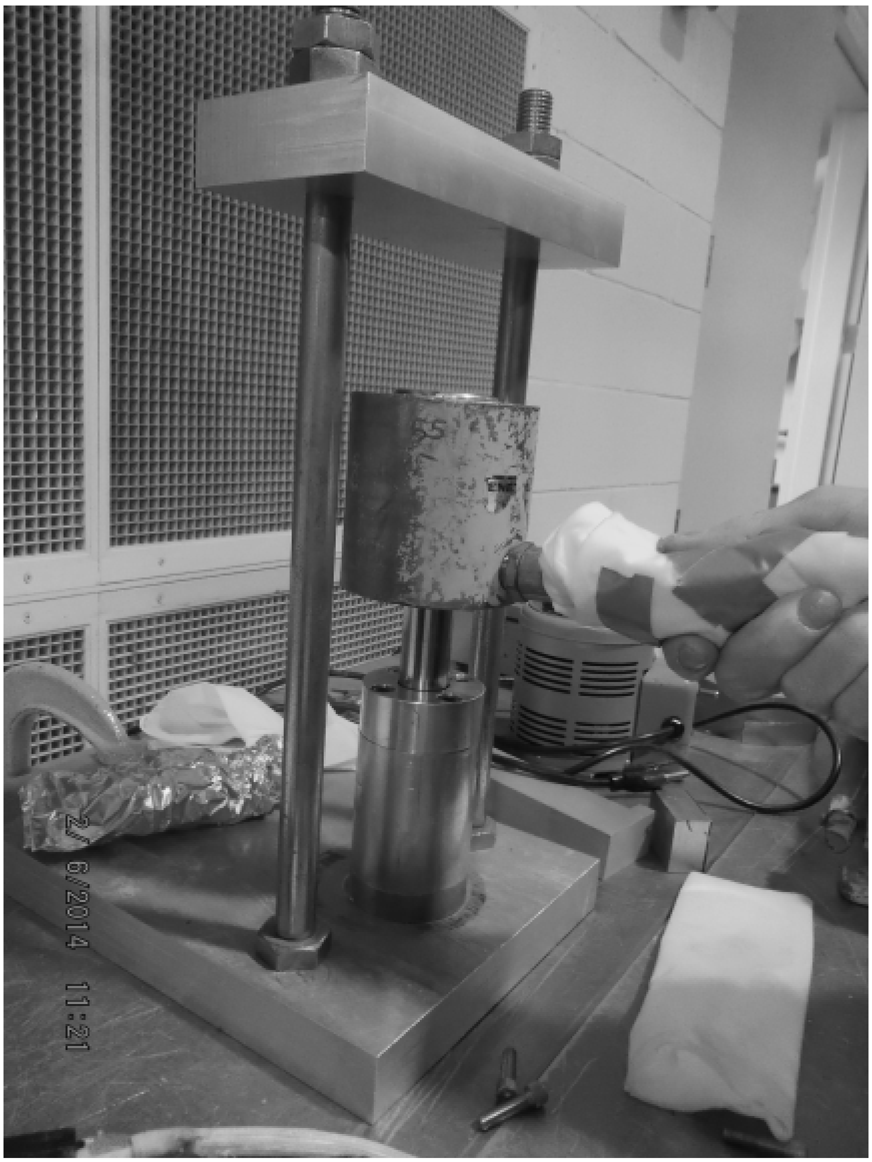

Figure 5.13: The hydraulic press sitting on top of the extrusion cylinder.

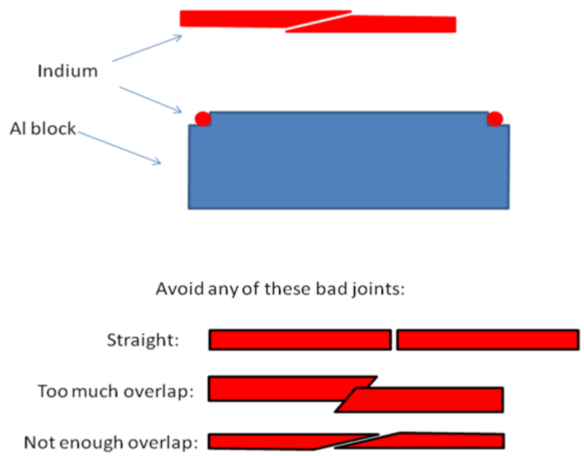

Figure 5.14: How to cut indium wire for fridge seals. [Zha13]

8. When it is time to make the seal on the fridge, carefully raise the can up to the fridge within 1 cm from touching, not allowing anything to touch the indium wire. If the wire is displaced, remake the seal. For the MC, hold in place with a chemistry clamp like in Figure 5.17, making sure to align the screw holes.

9. Insert two appropriate machine screws up through the can, and turn twice so they can support the weight of the can without the chemistry clamp. The indium should not touch the fridge flange.

10. Remove the clamp and turn the remaining four screws 2 turns each. For the MC, remove the chemistry clamp so the setup looks like Figure 5.18.

11. Alternate tightening the screws 1/4 turn each in a "star" pattern. That is, do not turn adjacent screws consecutively, and no single screw is tightened twice unless the other five screws have been tightened in between.

12. When the indium is compressed, make sure all the screws are evenly tightened, but do not strip the machine screws' hex heads.

13. If possible, leak check the indium seal.

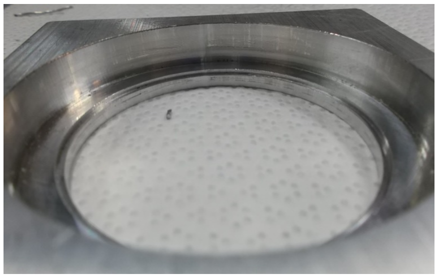

Figure 5.15: Aluminium block to press indium onto MC sealing surface (IVC block not shown).

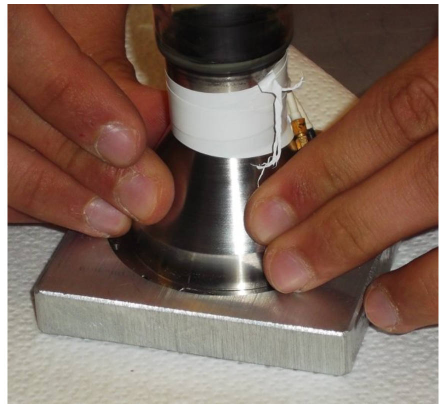

Figure 5.16: Pressing the MC onto the prepared indium ring.

Figure 5.17: The chemistry clamp holds the MC within 1 cm from the fridge, keeping the screw holes aligned, without letting the indium touch the fridge.

Figure 5.18: All six nuts and machine screws are supporting the weight of the hanging MC.

5.4 Breaking Indium Seals

1. Remove screws with 1.5 mm allen key and the modified MC wrench. Visually inspect each screw for indium or signs of stripping, and turn each nut back on the screw it came off of. If there is any torque resistance or sign of stripping, the screw was likely damaged when the indium seal was tightened and both the screw and nut should be discarded. If there is no damage, return the nuts/screws to the bag labeled "MC Screws and Nuts".

2. Use two MC screws and the 1.5 mm allen key as jacking screws the break the indium seal. Defining the top of the fridge (in the horizontal orientation) as 12:00, the jacking screw holes are at 1:00 and 5:00 look upstream. The jacking screw holes can be identified by threaded holes in the MC bell housing that have no matching through holes on the fridge MC flange. Alternate between screws making 1/4 turns until the MC is free.

3. Remove the MC from the fridge, careful not to damage the sensors on the copper dam.

4. Remove the jacking screws from the MC and place in the bag labeled "MC Screws and Nuts".

5. Scrape indium, if any, off the MC and place the MC in the safety zone.

6. Scrape indium off the fridge flange, recovering as much as possible in the indium scrap container; be very careful not to touch the stainless steel beam window.

5.5 Weighing LHe Dewars

Do not attempt this procedure until first being cleared by DFELL mechanical operations (see Joe Faircloth).

The Airgas LHe dewars are weighed as a de facto measurement of helium consumption. We weigh them before and after use.

1. Attach weighing rope/chain to the slots on top of the 250LD.

2. Power on the ceiling crane with the remote control and move it above the 250LD.

3. Lower the crane within a foot of the dewar.
4. Attach the dewar scale to the crane.
5. Secure the rope/chain on the 250LD to the bottom of the scale.
6. Raise the crane up until the dewar just leaves the ground.
7. Record the weight of the dewar on the Hifrost Dewar Record sheet.
8. Lower the crane, disconnect the scale and rope/chain, then return them to storage.
9. Raise the crane and return it to its original position.
10. Power off the remote control.

5.6 Mounting Fridge

5.7 Dismounting Fridge

5.8 Cleaning Viton/Buna O-rings and Grooves

HiFrost only uses isopropyl alcohol for cleaning vacuum parts. Methanol bottles are scattered around DFELL, but they are not to be used.

Most o-ring encountered are for KF joints. It is not practical to clean every o-ring every time it is touched, and this is unnecessary because our vacuum system is not UHV, so most KF joints can be visually inspected for dirt before use.

All ^{3}He system o-rings must be leak checked every time they are taken apart, regardless of whether they were cleaned or not.

To clean a KF o-ring:

1. remove o-ring from center piece
2. moisten a Kim Wipe with isopropyl alcohol
3. gently pinch the o-ring with the wipe, rotating it around to clean the whole surface
4. replace the oring over the centerpiece

5. wipe down the outside of the o-ring one more time

6. reassemble the KF joint, trying to touch only the centerpiece as much as possible (tip: gravity is your friend, here)

5.8.1 Fridge flanges

The triple flange, ^{3}He back flange, evaporator pump out and ^{3}He pump out all have rubber o-rings set in grooves. If any of these leak the fridge will not be able to acheive dilution (or ^{3}He will leak out), but they cannot be leak tested during a cold-target run because there is no time to pump down the system with a leak detector.

To minimize the change of them leaking, the following should be done each time one of these flanges are opened:

1. clean o-ring with isopropyl alcohol as described above

2. dampen qtip with alcohol and swap out o-ring groove (this will be very dirty from vacuum grease)

3. use a minimal amount of vacuum grease to coat the o-ring all the way around

4. replace the o-ring and seal the flange immediately

The vacuum grease feels unpleasant on hands, so latex gloves may be used. Otherwise, be sure to wash up afterward so vacuum grease doesn't contaminate other fridge components.

5.9 Installing Kenol Connectors

Kenol connectors are similar to VCR, except one sealing face is flat and the other has a raised ring to seal the gasket (see Figure 5.19). Two Kenol connectors are used for the separator and shield, and a smaller Kenol connector is used for the ^{3}He condensor line connection (see Figure 5.20).

- The fridge should be in the vertically mounted position when installing Kenol connectors.

- Drop the copper gasket in the Kenol port on the fridge.

- Vertically lower the mating connector down, pulling the nut up so the sealing face touches the copper gasket first.

- Keep the gasket centered by maintaining pressure on the male end while turning in the nut.
- When the nut is hand tight, use a wrench to tighten it further.
- Leak check the connection.

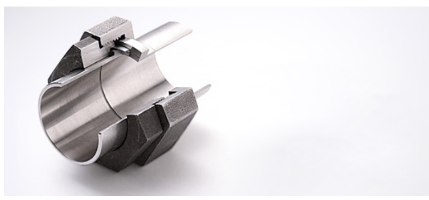

Figure 5.19: Kenol connector drawing [Gro].

Figure 5.20: The copper gasket of the separator Kenol connector is visible on the right; the ^{3}He Kenol connection is made on the left.

5.10 Installing Heating Tapes

5.11 Filling LN_2 Trap

5.12 Check 250LD/500LD LHe Level

The LHe level of helium dewars is checked upon delivery and after they are used to fill the 500LD. The 500LD LHe level is checked after it is filled, after initially filling the 100LD and periodically throughout the run. The 100LD has an internal level probe installed and does not use this procedure.

1. Remove zip ties and plastic wrap on full 250LD.
2. Make sure there is nothing sensitive directly above the dewar, then release pressure by opening main flow valve on top of the dewar. A large, cold plume will exhaust pressurized helium gas.

3. Use cryogenic gloves to slowly lower the level probe into the main flow valve. A smaller cold plume will exhaust upwards as the level probe cools down.

4. Plug the level probe into the level probe readout and record the reading. As a sanity check, lift the level probe a few inches to make sure the readout changes correspondingly.

5. Disconnect the level probe and remove it from the 250LD, storing it where it can safely warm up.

6. Close the main flow port.

5.13 Attach Pressurization Line to LHe Dewars

The 250LD and 500LD have LHe pushed out of them by pressurizing the top of their liquid contents with helium gas. The helium gas pushes down on the liquid surface, pushing liquid up a transfer line and out of the dewar.

The procedure below ensures air will not get in to the pressurization line and freeze the dewar port.

1. Check that the pressurization cylinder is not empty and a full helium cylinder is nearby. Make sure the regulator has single PSI increments discernable.

2. Open the pressurization cylinder and set the regulator to 2 PSI. Verify helium gas is flowing out.

3. Slowly open the dewar pressurization valve on the to-be-pressurized dewar until pressurized gas begins to flow out at about 5 PSI. If the pressurization frosts, close it and wait until it warms up, using a heat gun only if necessary.

4. With gas coming out of both the dewar pressurization port and the pressurization cylinder, mate the two connectors and close the dewar pressurization valve.

5.14 Purging Helium Lines

5.15 Vacuum Rise Testing

A leak checker is the proper way to determine if a vacuum volume is leak tight. However, it is not always practical and sometimes very difficult to leak check a large volume, so we use a vacuum rise test as a course characterization of the vacuum fidelity.

The concept is to pull a vacuum down in a volume, isolate it from the pump, then watch if the vacuum rises. Outgassing and a leak are generally the only two effects we will see, but outgassing usually stops the pressure rise far below 1 atm (e.g., water vapor pressurizes to about 24 mbar). If the volume is small, virtual leaks (finite, mostly trapped pockets of gas that take long times to equilibrate with the main volume) may also have a visible effect.

To set up, gather the following:

- pump
- associated KF vacuum fittings/hoses
- KF pressure gauge

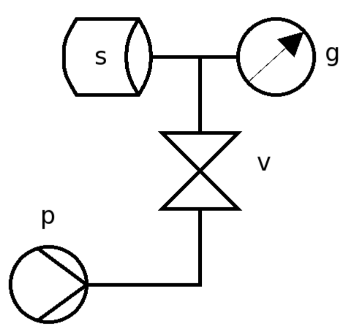

Figure 5.21: Vacuum rise schematic: *s* is the system to check; *g* is a pressure gauge; v is a leak-tight manual valve; *p* exhausts to atmosphere.

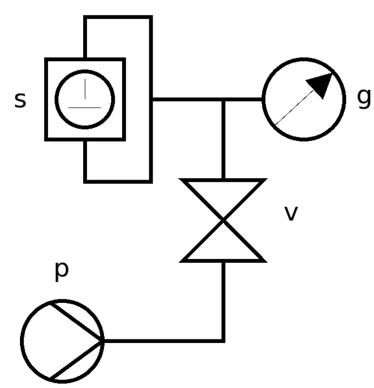

Figure 5.22: The same as Figure 5.21 except *s* has two ports, the inlet and exhaust of a pump, in this case.

The procedure is

1. Set up as shown in Figure 5.21 or Figure 5.22. If the system is powered (such as a pump), make sure it is unplugged.

2. Open v and pump down s until the pressure gauge g stops decreasing.

3. Close v and turn off p.

4. Monitor g until it stops rising. This can take from minutes to days.

If the system s is tight it will outgas to tens of mbar (depending on if it is contaminated with oil, water, etc) then stop. If it is necessary (and possible), s should then be checked with a leak detector to verify it is helium tight.

If the pressure gauge reaches an atmosphere, there is a leak in s. The time this takes determines how big the leak is. A quick way to verify s contains the leak is replacing it with a blank off and repeating the vacuum rise test. If g reaches an atmosphere again, there is a leak in the plumbing which should be repaired before trying the vacuum test again.

5.16 Disassembling Fridge

5.17 Assembling Fridge

When putting the fridge together, there are two primary concerns: forgetting to install something (e.g., superinsulation, microwave guide support) and leaky seals. This section does not include a cold target load and assumes the fridge is hanging vertically on the fridge stand in the Vault.

5.17.1 MC

Tools

- qtips/wire cutters/isopropyl alcohol
- indium scrap box
- 1.5 mm allen key
- MC open faced allen wrench (4.0)
- MC

Figure 5.23: The MC with a prepared indium seal behind held by a chemistry clamp.

- 1 mm indium wire
- chemistry clamp
- 6x M2x12 machine screws and nuts, 316SS

Read the section on making indium seals.

Hold the MC in place with the chemistry clamp so the bell housing is about 1 cm from touching the fridge flange. Place the bolts through the MC holes and gently tighten the nuts one or two turns so the bolts don't fall. Inspect the indium wire to make sure it is still in place, then tighten the nuts in a "star" pattern (never tightening adjacent screws consecutively). Hold the bolts with an allen key while turning the wrench so the heads do not strip. Use only the special MC wrench so the fridge does not scratch.

If loading conditions permit, leak check the MC before moving on.

5.17.2 Microwave Guide Support

Tools

- small flat head screwdriver
- squeeze-to-open tweezers

Three brass screws are usually stored in the threaded struts on the fridge where they fasten the waveguide support in place.

After unscrewing them, raise the microwave guide support over the MC making sure the waveguide is lined up with both the slot in the MC bell-housing and the circular waveguide connector on the fridge. One person holds the support while another tightens the screws. Hold the screws with tweezers to prevent dropping them down towards the MC (if this happens, you must start over, careful not to damage the fridge with the loose screw while taking off the support). Warning: the microwave guide support can still fall off when it is being held by one screw.

Make sure the waveguide (or the support) is not touching the MC. Normally this will not happen unless the support has been deformed.

5.17.3 IVC

Tools

- solder station
- 2.5 mm allen key
- indium scrap box
- qtips/wire cutters/isopropyl alcohol
- 6x M3x12 machine screws

Read the section on making indium seals.

Similar to installing the MC, first prepare the IVC indium seal and place it in the groove on the IVC flange. One person holds the IVC within about 1 cm from the fridge while another puts in the screws. Initially, only turn in 2 diametrically opposite screws 1 or 2 turns each so they support the IVC and it no longer needs to be held by another person. Turn in the other 4 screws 1 or 2 turns each. Inspect the indium seal to make sure it is still in place (it should not be touching the fridge flange yet) before turning in the screws in a "star pattern" similar to the MC. Be careful not to strip the screw heads.

Leak check if possible.

5.17.4 OVC

Tools

- pliers for pilot pins
- 5 mm allen key
- three OVC cans (inner, outer, nose cone)
- triple flange orings
- pilot pins
- 2x nut plates
- 8x M6x25 triple flange machine screws

Make sure the orings and grooves are clean (details at Section 5.8). Remove the OVC cans from the OVC protective crate and place them on the blue fridge lift. The black lines drawn on the outer flange surfaces should already be aligned by tee pins holding the two flanges together. Secure the OVC in place with the plastic nut and threaded rod that fits through the fridge lift platform. Hook up the OVC manifold to the OVC pump out port (making sure the hose is coming off at the appropriate angle for Blowfish clearance) and warm up the OVC turbo pump.

The nose cone is secured to the outer OVC can with yellow (non-residual) tape when it comes out of the storage crate. The tape prevents it from falling off while the fridge is vertical, but it can also prevent a proper o-ring seal during the initial pump down. The solution is to leave the tape on until right before OVC pumping begins, then remove it and keep a hand under the nose cone as the pumping begins. This ensures the nose cone will not fall at any point in the OVC prep. Once pumping begins, optionally leak check the nose cone and triple flange vacuum joints.

With the OVC still being pumped, remove it from the fridge lift and raise it over the fridge. This should not require appreciably more force than the weight of the OVC itself. Line up the black lines on the triple flange and push the pilot pins in by hand, using pliers if they need to be rotated or pulled out slightly.

Loosely turn in all the triple flange screws, careful not to strip the aluminum taps. Tighten in a star formation so the oring does not become unevenly compressed.

5.18 Removing/Replacing ^{3}He Baffles

Chapter 6

Subsystems

6.1 Thermistor Switch and Display

The thermistor system is set up to provide signals from all of the thermistors to the USB-1608G data acquisition (DAQ) unit (and so be visible and recorded outside the vault), and to maintain the ability to put any of the thermistor readings on a display while in the vault.

The EIO Body thermistor is wired to a dedicated single channel controller, which displays the temperature, has a normally-closed relay with a high-temperature trip point (set to 40 °C right now) in the Cober power supply interlock loop, and provides a DC voltage proportional to temperature to the DAQ (0 to 10 V corresponds to 0 to 100 °C).

Three other thermistors (EIO water flow out, Q-meter P and Q-meter D) are wired to the thermistor switch panels. These are wired in series, and a 100 μA power supply runs current through these, and DAQ channels are wired in parallel with each of the thermistor wires. The signal is proportional to thermistor resistance. These are 10 kΩ (at 25 °C) NTC thermistors, so the signal is 1 volt at 25 °C. A look-up table will need to be used to convert the readings to temperature.

The switch has 6 positions; Spare 1, EIO Flow, Q-meter P, Q-meter D, Spare 5 and Display Nothing. Set the switch to the thermistor which should be put on the display. When the switch is set to one of the first 5 positions, relays divert that thermistor signal to the display, and connect a short into the 100 μA PS loop. So, when you look at, say, EIO Flow, the DAQ signal for that channel will be zero volts, and that thermistor's temperature will show on the display. If the switch is set to Display Nothing, all 5 signals go the the DAQ. There is a 29.4 kΩ resistor for the display in the Display

Nothing position, which displays as 0 °C (or maybe 0.1).

The Spare 1 and Spare 5 positions also have 29.4 kΩ resistors, so the display will show zero on those positions as well, and the DAQ will get a pretty constant 2.94 V signal from them. These resistors can be replaced by thermistors in the future should the need for more temperature readings arise.

Referring to the schematic drawing, the switch is 4-Pole 6-Position (4P-6T). Two poles are used to energize the selected relay by sending 24 VDC and 24 Common to the selected coil.

In the not-energized state, each relay connects the thermistor for that channel to the 100 μA power supply. Each thermistor is also wired to a DAQ channel. The thermistors are wired in series, so the same 100 μA flows through all thermistors.

In the energized state, each relay connects the thermistor for that channel to the other two poles of the 4P-6T switch, which connects this thermistor to the thermistor display on the panel. Also, the thermistor is replaced by a short circuit so the 100 μA will continue to flow through the rest of the thermistors.

Also seen is a schematic, Figure 6.1, for the 100 μA power supply, which is based on a μA723 voltage regulator. Operating with a 24 VDC V_{cc} supply, the power supply output can provide 100 μA through a load of 0 to 100 kΩ.

The 6 photos, Figure 6.2, show the front and back of the three small panels which comprise the thermistor system Switch panel, Display panel, and Relay panel. The Switch panel and Relay panel are connected with a 25 wire DB-25 cable. The display panel also holds a flow switch display which is wired to the EIO cooling water flow switch.

6.2 IVC Manifold

The IVC manifold, see diagram on Figure 6.3, connects the turbo pump to the IVC connector on the fridge, allows flow of helium gas during MC cooling, and hosts the Pirani pressure gauge.

The pump cart the IVC sits on is powered by first plugging in the turbo pump fan, then plugging in the turbo pump controller, and finally, plugging in the mechanical pump, which automatically starts it up.

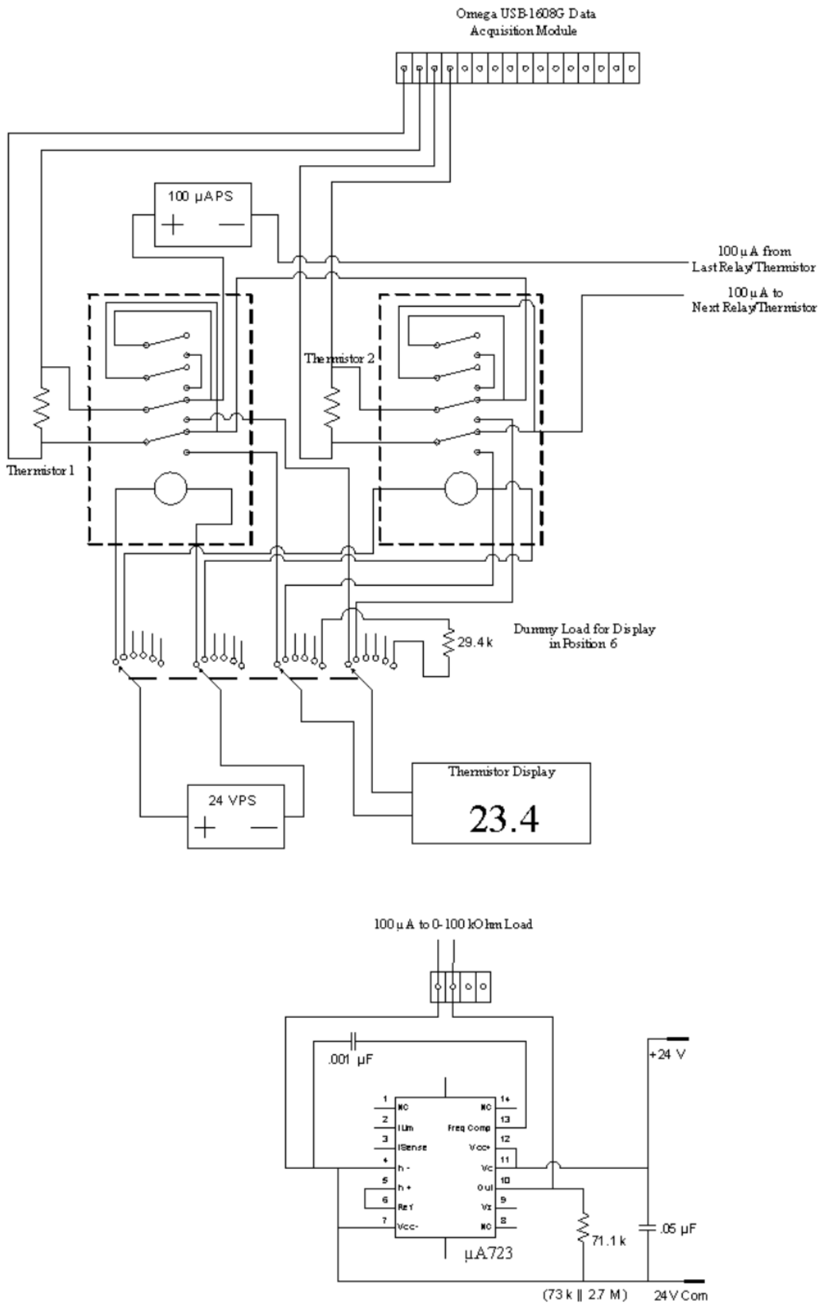

Figure 6.1: Schematic of thermistor system.

Figure 6.2: Photos of thermistor system.

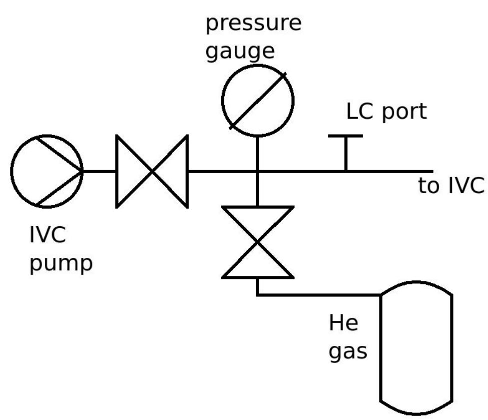

Figure 6.3: The IVC turbo and mechanical pumps evacuate the manifold and IVC line. The gas line is used to backfill the IVC to about 40 mbar, measured with the pressure gauge, of exchange gas during cooling.

6.3 OVC Manifold

The OVC manifold is simply a valve on top of a turbo pump that connects to the OVC pump out ports on the fridge. Between the fridge and the valve is a tee with a cold cathode pressure gauge. See Figure 6.4.

The green button on the front of the HiCube powers on the controller, and then the power button the front panel starts the pump. Make sure the ballast valve in the back is closed before pumping. If the turbo stalls before reaching 1500 Hz, slowly wind down the pump, blank it off, and try again. Power cycle as a last resort.

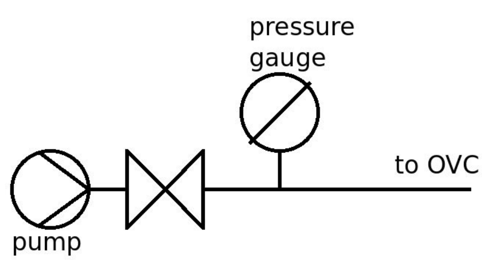

Figure 6.4: The OVC pump is a Pfeiffer HiCube station, and the pressure gauge is a Pfeiffer IKR 251 cold cathode gauge.

Appendix A

LHe Order Requirement Checklist

Date: ____________________ Name: ____________________

LHe transfer

- ☐ {☐ 500TL, ☐ LTL} pumped down
- ☐ delivery dewar Goddard fittings assembled and ready
- ☐ two helium cylinders by gas manifold (one is full)
- ☐ dewar pressurization line is ready
- ☐ number of additional He cylinders on hand: ______
- ☐ dewar scale and weight record form is ready
- ☐ LHe thumper or probe/readout are ready
- ☐ heat gun is plugged in and reaches dewar
- ☐ cryo safety equipment is ready for LHe transfer
- ☐ walkie talkies charged
- ☐ 100TL installed in 100LD; bayonet attached to WTL

Fridge

- ☐ target material installed: ______
- ☐ MC spacer/dam installed
- ☐ MC Si room temp: ______
- ☐ AVS-47 room temp: Ch 0: ______, Ch 1: ______, Ch 2: ______
- ☐ leak check MC indium seal ______mbar l/s
- ☐ ^{3}He circ loop VRT: ______mbar/day
- ☐ leak check IVC indium seal: ______mbar l/s
- ☐ leak check OVC: ______mbar l/s
- ☐ align fridge with beamline
- ☐ flow impedance through fridge observed
- ☐ ^{4}He Lakeshore sensors read to computer

Pump station area - ^{3}He

☐ leak detector available
☐ LN_2 trap regenerated
☐ LN_2 trap dewar filled
☐ mash cleaned
☐ T_1: ______mbar, T_2: ______mbar, T_3: ______mbar
☐ flow meter on ^{3}He gas rack powered on and recording
☐ mash cleaning system is set up for runtime cleaning
☐ all pump oil view ports half full

Pump station area - ^{4}He

☐ {☐ sep, ☐ shield, ☐ evap} flowmeters working
☐ sep/shield purged ______ times; backfilled with He gas
☐ evap purged ______ times; backfilled with He gas
☐ all pump oil view ports half full
☐ reading on 2000L LN_2 tank: ______%

Gamma Vault

☐ 100LD Goddard fittings assembled and installed on 100LD
☐ install exhaust tubing on 100LD and run over wall
☐ 100LD LHe level probe readout reading to computer
☐ OVC pressure: ______mbar
☐ IVC pressure: ______mbar
☐ fridge heating tapes installed
☐ heat gun is plugged in and reaches 100LD Goddards

Polarizing magnet

☐ Goddard fittings inspected/installed
☐ LN_2 and LHe readouts working
☐ power suppy HP 6061A ready
☐ control programmer AMI 420 ready
☐ heat gun with extension cord set up on magnet
☐ cryo safety equipment placed on magnet stand
☐ magnet/detector carriage installed

Holding magnet

☐ resistance across holding magnet: ______Ω
☐ power supply AMI PS 12100 ready
☐ control programmer AMI 420 ready
☐ energy absorber AMI 601 ready
☐ cable plugged into fridge

Microwaves

- ☐ EIO water is filled and flowing
- ☐ laptop webcam working
- ☐ microwaves tested with power meter: ______mW
- ☐ microwave frequency tested: ______GHz
- ☐ microwave guide connected to fridge
- ☐ thermistors and water flow readouts work and read to computer

NMR

- ☐ NMR water is running
- ☐ PDP running
- ☐ NMR is tuned
- ☐ $\lambda/2$ cable reconnected after swing

Appendix B

SLPM Conversion

Some HiFrost documents from CERN refer to flow rates of ^{4}He and ^{3}He in millimoles per second. The formula to convert this flow rate to SLPM is

$$[\text{SLPM}] = \left(\frac{60\ \text{s}}{\text{min}}\right)\left(\frac{x\ \text{mol}}{\text{s}}\right)\left(\frac{4\ \text{g}}{\text{mol}}\right)\left(\frac{1\ \text{L}}{0.1785\ \text{g}}\right) \tag{B.1}$$

for ^{4}He and

$$[\text{SLPM}] = \left(\frac{60\ \text{s}}{\text{min}}\right)\left(\frac{x\ \text{mol}}{\text{s}}\right)\left(\frac{3.01\ \text{g}}{\text{mol}}\right)\left(\frac{1\ \text{L}}{0.135\ \text{g}}\right) \tag{B.2}$$

for ^{3}He[Lin], where [SLPM] is the standard flow volume, x is the flow rate in mol/s, k is the Boltzmann constant and the definitions for STP are a temperature of 273.15 K and a pressure of 100 kPa.

Figure B.1 shows some values of SLPM to mmol/s flow.

SLPM	mmol/s (^{3}He)	mmol/s (^{4}He)
5	3.74	3.71
10	7.48	7.44
15	11.21	11.16
20	14.95	14.86
25	18.69	18.60
30	22.42	22.31
40	29.90	29.75
50	37.38	37.19
60	44.85	44.63
80	59.80	59.50
100	74.75	74.38

Figure B.1: Flow rate conversion between mmol/s to SLPM for helium.

Appendix C

Transfer Line Drawings

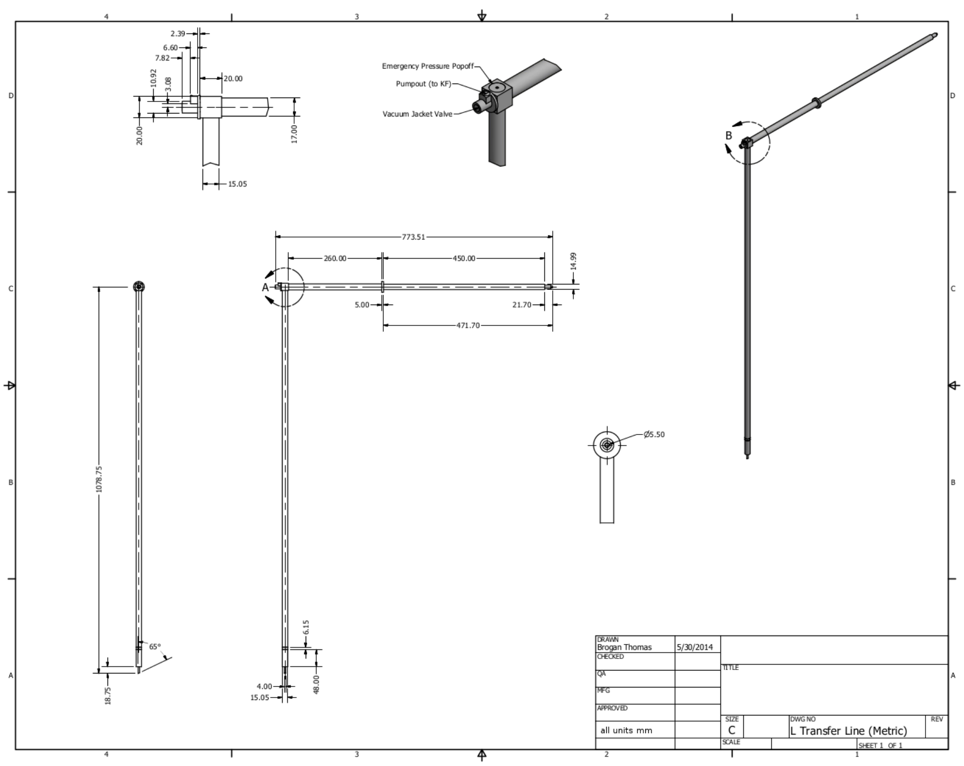

Figure C.1: Drawing of the LTL.

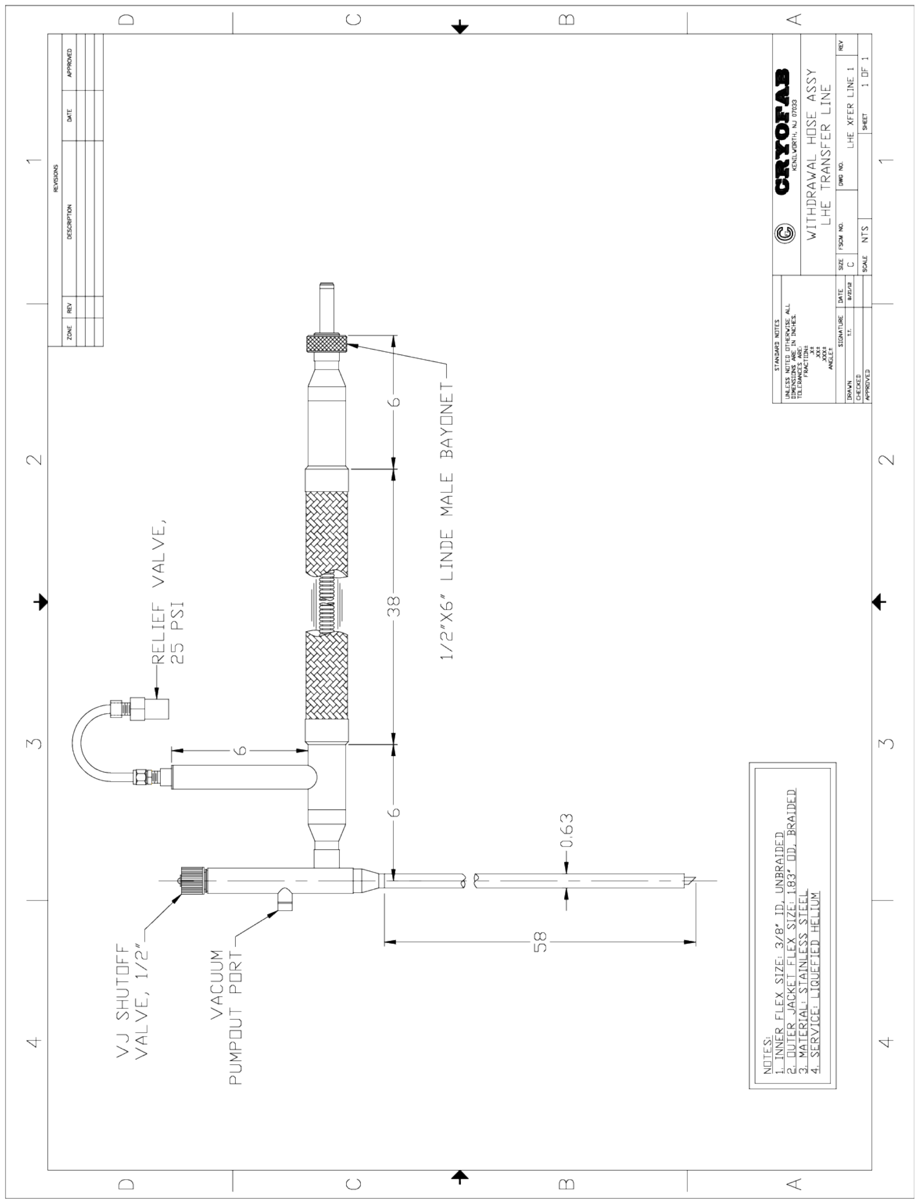

Figure C.2: Drawing of the 500TL.

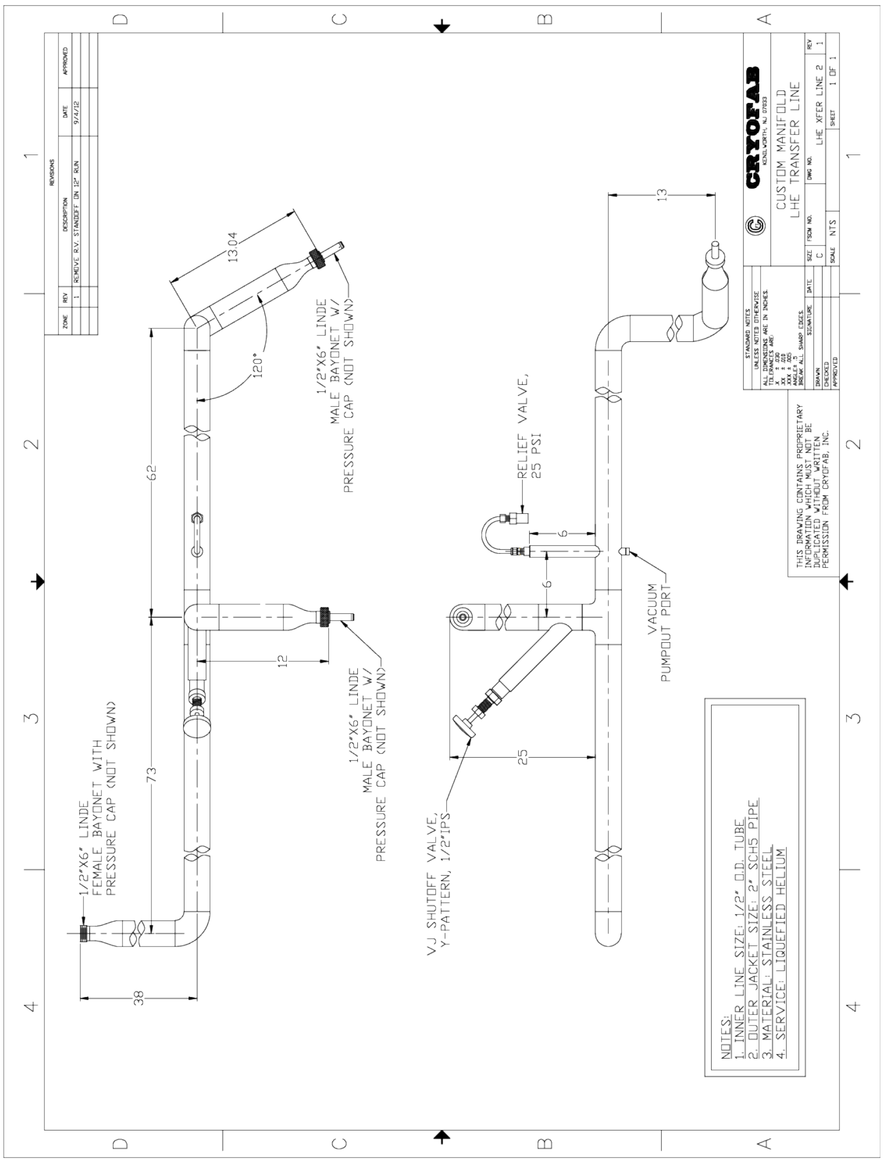

Figure C.3: Drawing of the WTL.

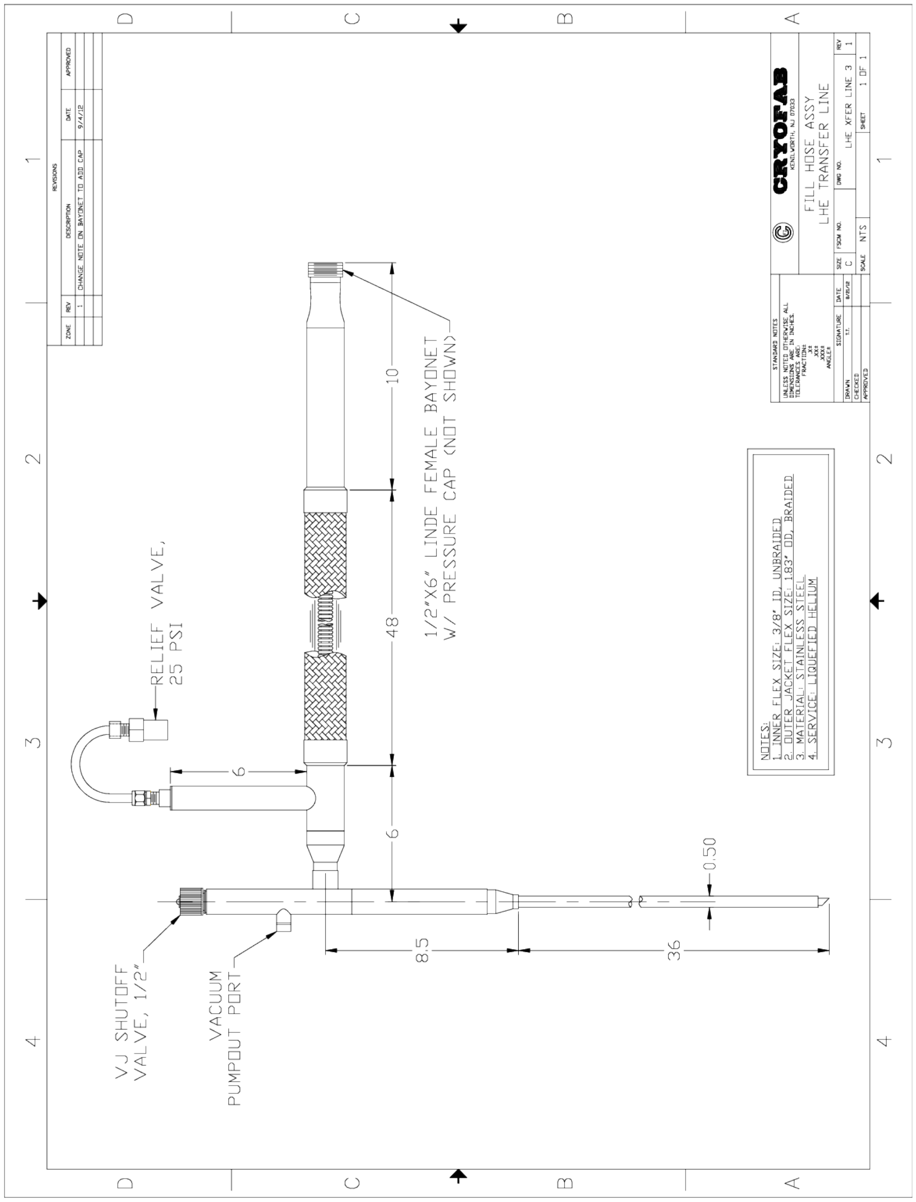

Figure C.4: Drawing of the MTL.

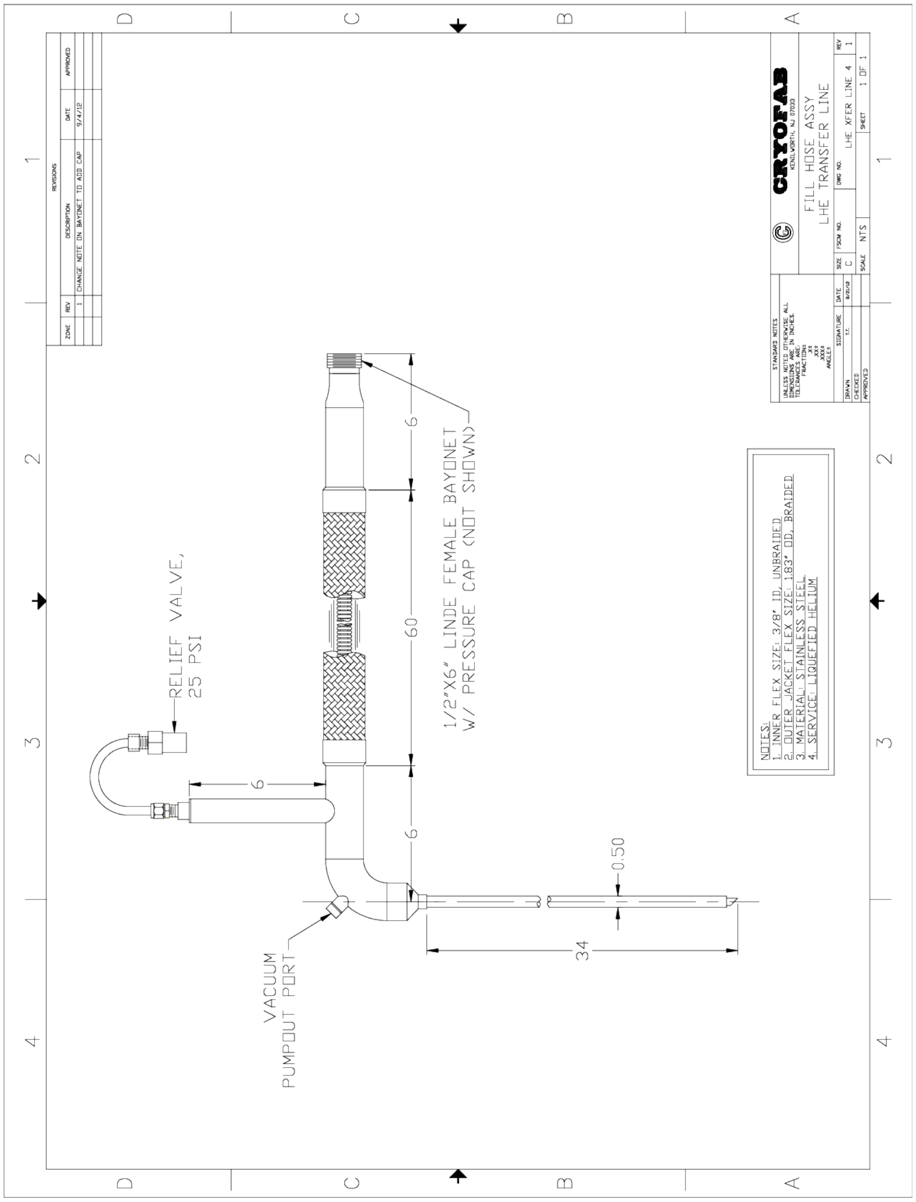

Figure C.5: Drawing of the 100TL.

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