University of Virginia

Duke University

HIγS Frozen Spin Target

HiFrost Manual

Author:
Ryan Duve

Supervisors:
Blaine Norum
Don Crabb
Pil-Neyo Seo

Updated May 10, 2014

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.

TODO: include photo of HiFrost

Contents

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

$$P = \frac{\uparrow - \downarrow}{\uparrow + \downarrow},$$

where ($\downarrow$, $\uparrow$) is the population of nuclei with m_z=(-$^1/_2$,$^1/_2$).

1.1.1 DNP

Dynamic Nuclear Polarization (DNP) is a process of using microwave radiation to pump electron-proton pairs to higher energy (polarized) 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 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.

1.2 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

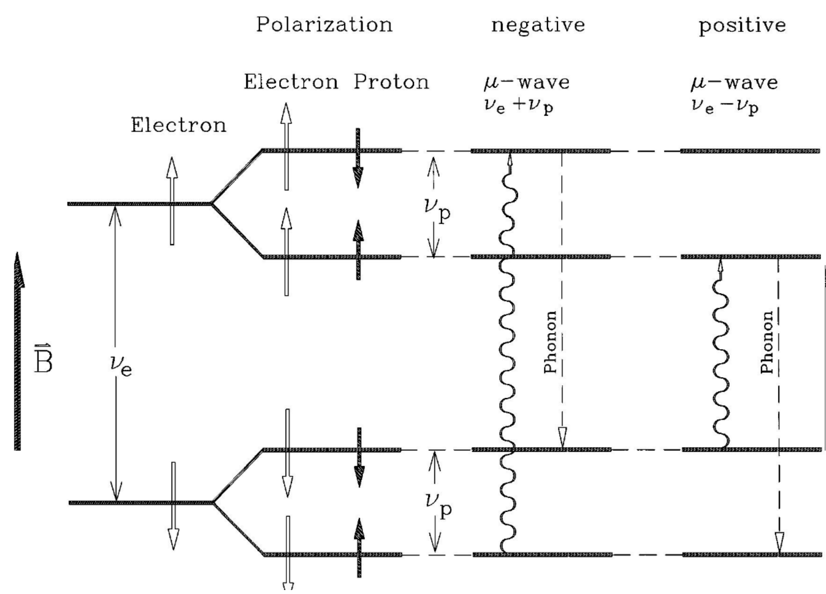

Figure 1.1: DNP diagram [CM97]. The white arrows are electron polarization and the black arrows are proton polarization. Microwaves at a frequency difference $\nu_e \pm \nu_p$ flip the spins of both particles, and the electron's superior lattice coupling ensures it will flip back before the proton.

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 Frozen Spin

The target is polarized in a 2.5 T field, called the polarizing field. The target will lose all polarization in an environment free of magnetic field. However, the detector cannot fit around the polarizing magnet, and the polarizing magnet would obstruct outcoming neutrons, anway). To maintain polarization while the target is in the detector, an internal 0.5 T field is turned on inside the fridge and the polarizing magnetic is wound down and removed. The 0.5 field, called the holding field, sufficiently maintains polarization of nucleons while the detector is moved aroud the target and data is taken.

1.4 Dilution 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.

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 1.2 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]

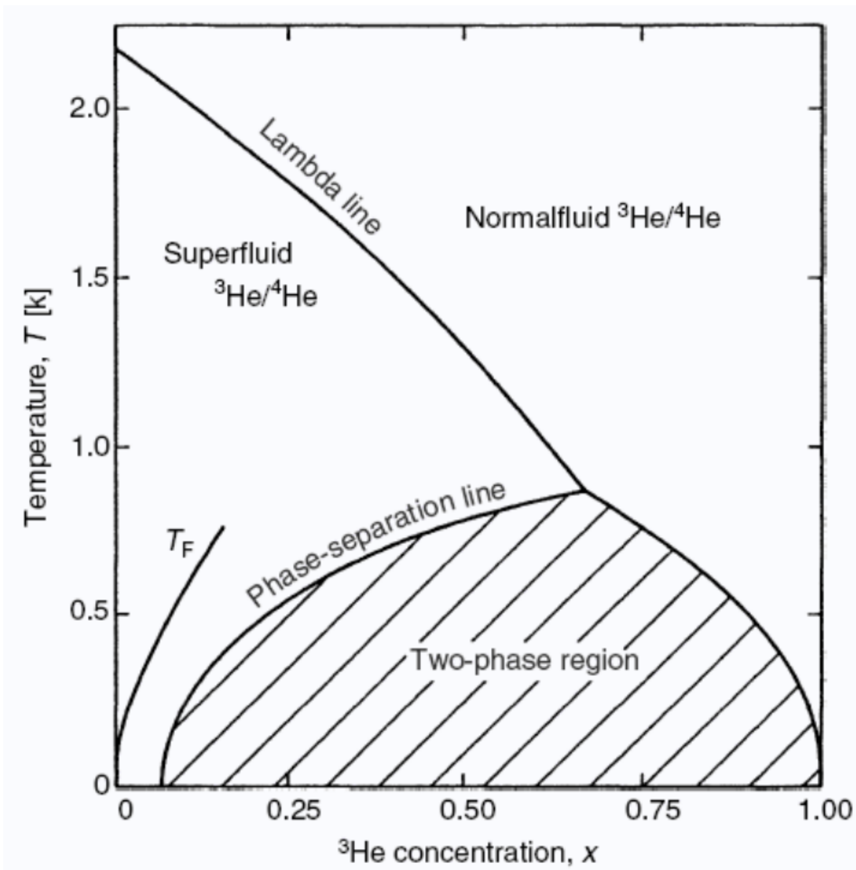

Figure 1.2: The famous diagram that shows the splitting of two distinct phases of ^{3}He-^{4}He mixture. [Bet89]

Chapter 2

Hardware

This chapter is an index and a catalog of all the equipment used in the project.

2.1 Fridge

2.1.1 Assembly Tools

OVC

- wire gripping pliers
- 5 mm allen key
- tee pilot pins

IVC

- solder station
- 2.5 mm allen key
- indium waste box
- wooden or hard plastic q-tips
- wire cutting pliers
- isopropyl alcohol

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

2.1.2 Cryogenic Instruments

^{4}He sensors

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

^{3}He sensors

- *Lakeshore*® DT-670 silicon diode
- *Omega*® KHLV-0502 Kapton heater
- *Lakeshore*® RX-202A-AA ruthenium oxide sensor
- *Lakeshore*® GR-200A-30 germanium diode
- 4-wire Matsushita resistor (model unknown)

2.1.3 Magnet Leads

- OFHC copper leads
- insulating mylar
- Kapton tape
- small flat head screwdriver
- minature flat head screws
- *Solid Sealing*® KT20678 copper conductor KF feedthrough

2.2 Target Material

- cryogenic storage LN_2 dewar
- 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*® 5/16″-18 nuts, bolts

2.4 Pump Station Area

- Alcatel or VIC leak checker
- KF-25 plastic blank off
- KF-25 flexible hose

2.4.1 ^{3}He Pumps

TODO: get pump oils listed next to pumps

- *Pfeiffer*® WKP4000 Roots pump
- *Alcatel*® MIV1000 Roots pump
- *Alcatel*® MIV350 Roots pump
- *Alcatel*® 2063-H mechanical pump

2.4.2 ^{4}He Pumps

- *Alcatel*® RSV1002 Roots pump
- *Alcatel*® RSV300 Roots pump
- *Alcatel*® 2063-H mechanical pump

2.4.3 LN_2 Trap

- LN_2 transfer dewar
- cryogenic gloves
- heat gun
- mechanical pump

2.4.4 Helium Gas Cylinders

- 1-1/8″ open wrench
- 1/2″ fNPT to hose barb fitting for dewar pressurization

2.5 Vault Pumps

2.5.1 OVC

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

2.5.2 IVC

- turbo pump
- *Pfeiffer*® PCR 280, ActivePirani/Capacitance gauge
- *Key*® 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

TODO attach drawings of TLs to appendix

- WTL
- UTL

- MTL
- *Cryofab*® OPE1/2-KF25 SV-9 Operator KF25 outlet pumpout adapter

Cern LTL

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

2.7 Vault Electronics Rack

2.7.1 Modules

- 2x *Lakeshore*® 218S readout
- AVS47 resistance bridge
- *Cryomagnetics*® Model 1200 S/H level readout
- *Agilent*® 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
- TODO find size of crimp connection

2.8 NMR

1. computer with PDP installed
2. *NI*® BNC-2090 rack mounted terminal block
3. water cooled housing
4. water chiller TODO find model
5. *Omega*® SA1-TH-44006-40-T thermistor
6. *Omega*® DP25-TH thermistor readout
7. power supply
8. DIO panel
9. SMA wrenches
10. *Rhode and Schwartz*® SMA100A function generator
11. cable from R&S to SMA
12. *NI*® PCIe-GPIB 778930-01 computer card
13. *NI*®763061-02 cable
14. *NI*® PCIe-6321 - 781044-01 computer card
15. *NI*® 192061-02 cable
16. *NI*® PCIe-6509 - 779976-01 computer card
17. *NI*® 182762-02 cable
18. $\lambda/2$ cable
19. oscillator crystals
20. plastic screwdrivers
21. Q-Meters
22. Yale Cards
23. oscilloscope

2.9 Microwaves

- EIO
- Cober power supply
- *Phase Matrix*® 578 microwave counter
- power meter TODO find brand/model
- mixer TODO find brand/model
- various microwave guides and attenuators
- 5/64″ ball driver
- 3/32″ ball driver
- water chiller
- *Omega*® SA1-TH-44006-40-T thermistor
- *Omega*® DP25-TH thermistor readout
- *Gems Sensors*® 155421 flow sensor
- *Gems Sensors*® 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 emergency 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}Hegas 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. an emergency 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 emergency relief valve is already closed to maintain liquid flow to the 500LD (see TODO 500LD fill procedure). 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, 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 physically 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. TODO: contact Rita Oakes, 919-684-3136 (press 2), for what to do in event of a burn

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:

3.3 High Voltage Safety

3.4 Gravity Safety

3.5 Hot Surface Safety

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

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.

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

To polarize a target in the lab:

1. Prepare equipment

2. Cool fridge

3. Polarize

4. NMR

5. Frozen spin mode

4.1 Cooldown

4.1.1 Prep

Before LHe is ordered, the system must be verified to be in working order. The complete checklist for ordering helium is at the end of the document (see Appendix A).

4.1.2 Dewar Filling

4.1.3 Cool ^{4}He Section

4.1.4 Cool ^{3}He Section

4.1.5 Dilution

4.2 Polarization

4.3 NMR

4.4 Frozen Spin

Lorem ipsum dolor sit amet, consectetuer adipiscing elit. Etiam lobortis facilisis sem. Nullam nec mi et neque pharetra sollicitudin. Praesent imperdiet mi nec ante. Donec ullamcorper, felis non sodales commodo, lectus velit ultrices augue, a dignissim nibh lectus placerat pede. Vivamus nunc nunc, molestie ut, ultricies vel, semper in, velit. Ut porttitor. Praesent in sapien. Lorem ipsum dolor sit amet, consectetuer adipiscing elit. Duis fringilla tristique neque. Sed interdum libero ut metus. Pellentesque placerat. Nam rutrum augue a leo. Morbi sed elit sit amet ante lobortis sollicitudin. Praesent blandit blandit mauris. Praesent lectus tellus, aliquet aliquam, luctus a, egestas a, turpis. Mauris lacinia lorem sit amet ipsum. Nunc quis urna dictum turpis accumsan semper.

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 pumpout 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 pumpout until almost the entire vacuum jacket pumpout 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.

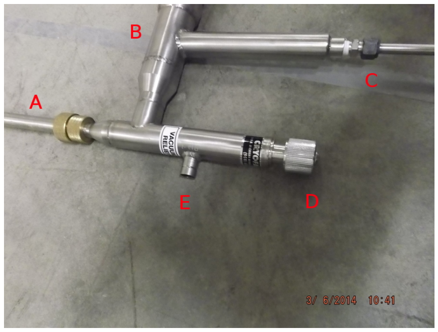

Figure 5.2: A: to TL stinger; B: to TL bayonet; C: emergency popoff valve; D: LHe flow valve; E: vacuum jacket pumpout.

Figure 5.3: Adapter on vacuum jacket pumpout.

Figure 5.4: Stem pushed in.

Figure 5.5: Stem pulled out.

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.

Figure 5.6: The LTL adapter.

Figure 5.7: The LTL.

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!
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.

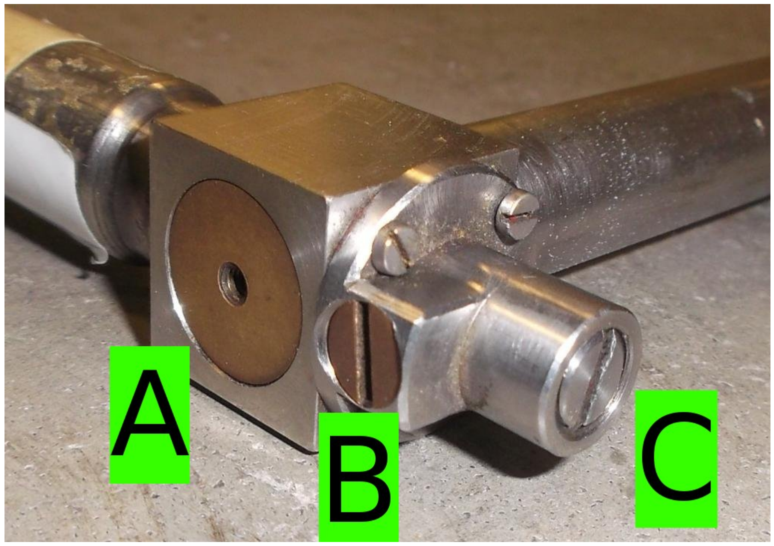

Figure 5.8: The three ports on the LTL: A) emergency relief popoff (this will shoot very high when the LTL freezes!), B) vacuum jacket pump port and brass placeholder screw, C) side screw valve.

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.

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

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

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***® 9″x 9″, 409, and kept for storage.

An *Enerpc*® P39 Porta Power **hydraulic hand pump** provides the force for extrusion.

5.2.3 Extrusion process

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

2. Remove steel rod from extrusion cylinder and insert blocks of indium. Place the steel rod back in the cylinder. *NB: after losses, 100 grams of indium provides approximately 10 feet of wire instead of the theoretical 50 feet.*

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

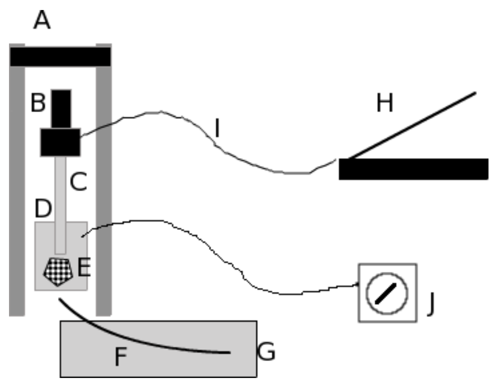

Figure 5.10: Indium extruder setup: A) extruder stand, B) hydraulic pump, C) steel rod, D) extrusion cylinder, E) indium scrap/ingot fragments, F) finished wire exiting die, G) sterile wipe sheet, H) hand jack, I) hydraulic line, J) thermocouple gauge

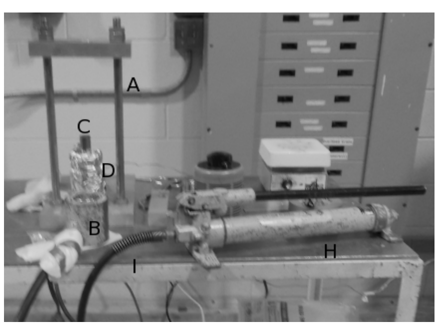

Figure 5.11: A photo of the setup.

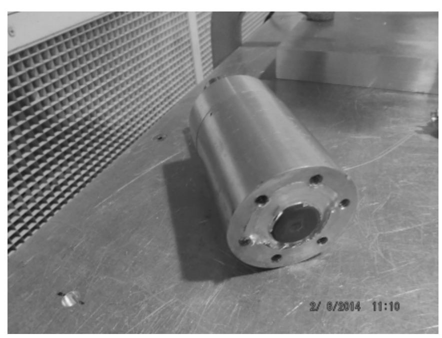

Figure 5.12: The extruder cylinder without the die. The steel rod is plunged all the way down and leftover indium can be seen on the surface.

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.

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 flanges the indium will seal together.

2. Measure the diameter of the indium to be used; it should be 1 mm

3. Cut a length equal to the circumference of the flange (TODO: find IVC/MC flange sizes) 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.

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 TODO photo.

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.

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, begin from the beginning. Hold in place with a chemistry clamp. TOOD show photo

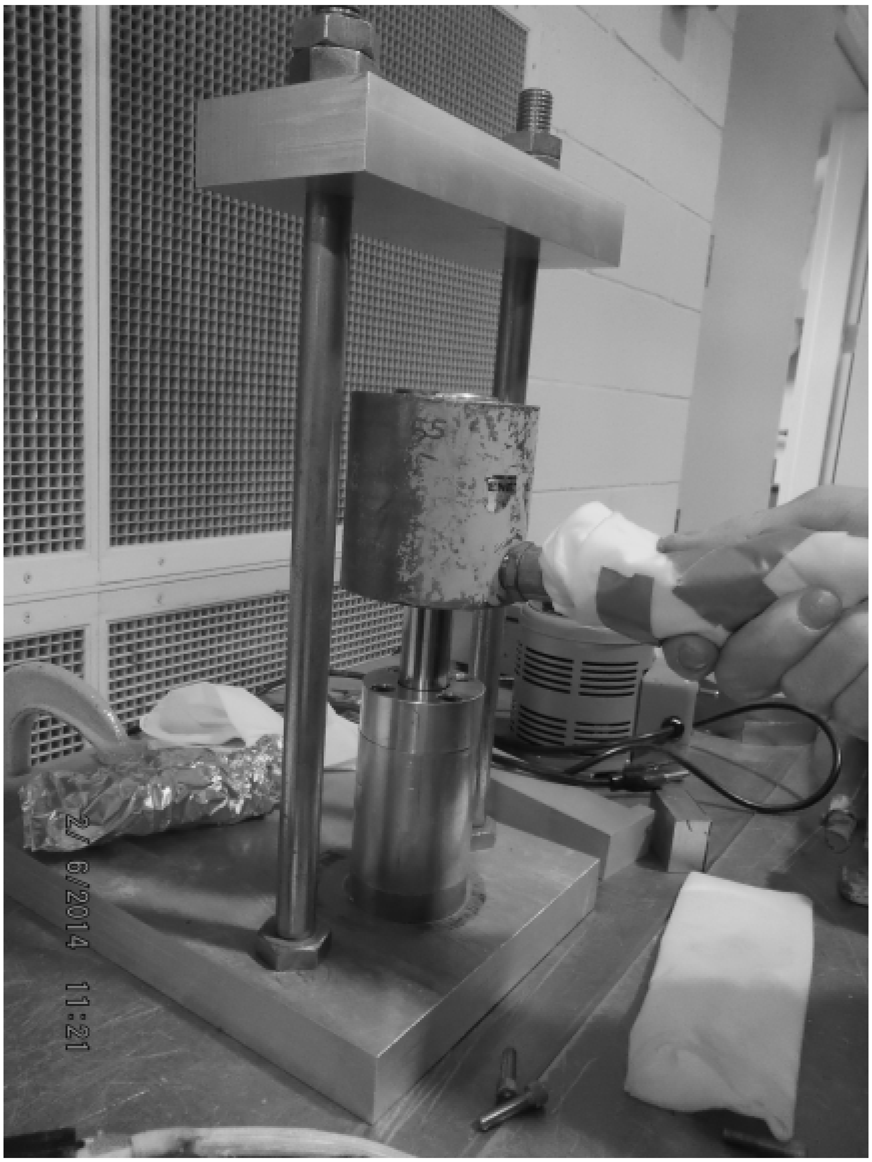

Figure 5.13: The hydraulic press sitting on top of the extrusion cylinder.

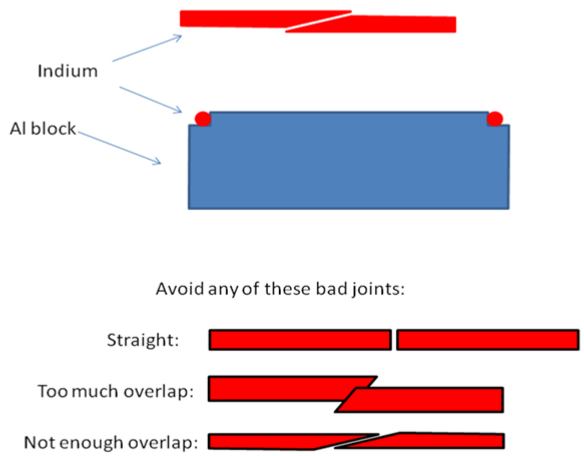

Figure 5.14: How to cut indium wire for fridge seals. [Zha13]

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 times each.
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.

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 Magnet Lead Installation

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 pumpout and ^{3}He pumpout 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 Changing Kenol Connectors

5.10 Installing Heating Tapes

5.11 Filling LN_2 Trap

5.12 Purging Helium Lines

5.13 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.

To set up, gather the following:

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

The procedure is

1. Set up as shown in Figure 5.15 or Figure 5.16. 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

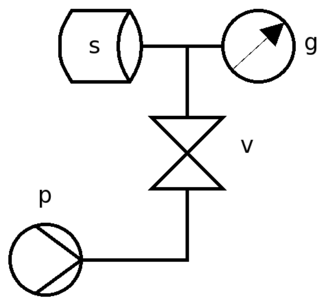

Figure 5.15: Vacuum rise schematic: s is the system to check; g is a pressure gauge; v is a leak-tight manual valve; p pumps to atmosphere.

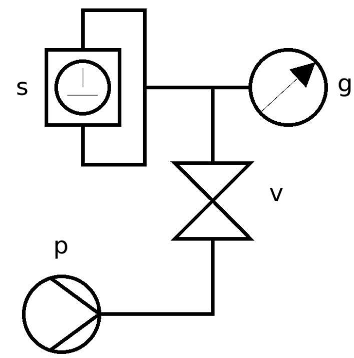

Figure 5.16: The same as Figure 5.15 except s has two ports, the inlet and exhaust of a pump, in this case.

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.

5.14 Stripping Fridge

5.15 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.

5.15.1 MC

Tools

- qtips/wire cutters/isopropyl alcohol
- indium scrap box
- 1.5 mm allen key

Figure 5.17: The MC with a prepared indium seal behind held by a chemistry clamp.

- MC open faced allen wrench (4.0)
- MC
- 1 mm indium wire
- chemistry clamp
- TODO sizes for MC nuts and bolts

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.

5.15.2 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.

5.15.3 IVC

Tools

- solder station
- 2.5 mm allen key
- indium scrap box
- qtips/wire cutters/isopropyl alcohol
- TODO find out IVC screw sizes

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.

5.15.4 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
- triple flange screws TODO find size

Make sure the orings and grooves are clean (see cleaning orings section). Place the outer OVC can on the blue fridge lift in the Vault, and lower the inner OVC can inside of it, making sure the black lines on the flanges line up. Place the pilot pins up through the pilot holes and secure the cans in place with nuts and threaded rod that fit through the fridge lift platform. Hook up the OVC manifold to the pumpout (making sure the hose is coming off at the appropriate angle for Blowfish clearance) and warm up the OVC turbo pump. Raise the nose cone in place, being sure to line up the large dents, and begin pumping. Leak check the OVC.

With the OVC still being pumped, remove it from the fridge lift and raise it over the fridge. 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.

5.16 Removing/Replacing ^{3}He Baffles

Appendix A

Checklist

Checklist Before Ordering LHe

Date: ______________________________ Name: ______________________________

Pump station area - He-4

- ❏ flow meters on and working
- ❏ laptop webcam working
- ❏ {□ LTL, □ WTL, □ UTL, □ MTL, □ 100TL} pumped down
- ❏ {□ 250LD, □ 500LD} Goddard fittings assembled and ready
- ❏ 500LD level probe/monitor plugged in and powered on
- ❏ helium gas cylinder hooked up to manifold
- ❏ pressurization helium cylinder hooked up to 500 LD
- ❏ at least one full backup helium cylinder nearby
- ❏ dewar scale is ready
- ❏ system is purged and backfilled with helium gas

Pump station area - He-3

- ❏ LN2 trap filled
- ❏ Mark-III dewar filled
- ❏ He-3 lines vacuum tested
- ❏ LN2 trap regenerated
- ❏ record He-3 tank pressures in log book (T1, T2, T3)
- ❏ flow meter on He-3 gas rack on and reading
- ❏ system is purged and backfilled with helium gas

Gamma vault

- ❏ 100LD Goddard fittings assembled and installed on 100LD
- ❏ 100LD level sensor plugged in and reading
- ❏ magnet LN2 sensor plugged in and reading
- ❏ magnet LHe sensor plugged in and reading
- ❏ fill LN2 in magnet space
- ❏ OVC is assembled and pumped down to ~1E-5 mbar
- ❏ IVC manifold is assembled and hooked up to fridge
- ❏ Lakeshore sensors are being read to computer
- ❏ AVS-47 bridge is warmed up and all sensors are working
- ❏ walkie talkies charged

Microwaves

- ❏ EIO water is running
- ❏ micowaves tested with power meter
- ❏ wave guide is hooked up to fridge

NMR

- ❏ signal achieved with oscillator crystal in mixing chamber coil
- ❏ NMR water is running
- ❏ PDP running

Bibliography

[Air] Airgas, *Isopropanol material safety data sheet*, `http://www.airgas.com/documents/pdf/001105.pdf`.

[Bet89] DS Betts, *An introduction to millikelvin technology*, Cambridge University Press, 1989.

[CG80] G Court and DW Gifford, *Liverpool nmr module, q-meter fundamentals, and circuit testing*, Ultra Physics LTD, November 1980.

[CM97] D Crabb and W Meyer, *Solid polarized targets for nuclear and particle physics experiments*, Annual Reviews Nuclear and Particle Physics **47** (1997), 67–109.

[EPA13] United States Environmental Protection Agency EPA, *Learn about lead*, `http://www2.epa.gov/lead/learn-about-lead#exposed`, July 2013.

[Hoc] Kai Hock, *Cooling techniques*, `hep.ph.liv.ac.uk/~hock/Teaching/2011-2012/4-cooling-techniques.pdf`.

[Mat06] Brent Mattox, *Investigative report on chemistry 301a cylinder explosion*, Tech. report, Texas AM University, January 2006.

[Sta98] Kevin C. Staudinger, *Occupational lead poisoning*, `http://www.aafp.org/afp/1998/0215/p719.html`, February 1998.

[TJNL] Industrial Hygiene Group Thomas Jefferson National Laboratory, *Oxygen deficiency hazard safety booklet*, `jlab.org/accel/safetylb/ODH-book.pdf`.

[Zha13] Jinkui Zhao, *On making indium seals*, private email correspondence, June 2013.