Cool Down Setup

The UVA Solid Polarized Targets

November 7, 2016

1 Preparation

Checklist before starting or even ordering the Helium

1.) Check Fridge, Separator, transfer lines, etc... for leaks ($10^{-}9$ mbar/s)
2.) Check Roots pumps are ready to run and had oil changed within 2 years
3.) Check target inserts are not leaking ($10^{-}7$ mbar/s)
4.) Check resister readouts on target insert and fridge
5.) Check microwaves and power supply are working and are set correctly
6.) Check for moisture in the microwave lines
7.) Check microwave power output
8.) Check and tune NMR system
9.) Check PDP and DAQ is working and ready and storing data
10.) Calibrate pressure readout

2 Vacuum of OVC and IVC

The vacuum space works as an insulator. Its important to separate the Helium layers from the atmosphere layers to preserver the Helium and to move the Helium with the least losses. Turn on the gray electric pump and let pump down to a few $10^{-}2$ (takes 6 hours). Turn on Pfeiffer Turbo (1000 Hz) and let pump down to a few $10^{-}5$, switch meter at $10^{-}3$ (see Fig. 6.) (takes 6 hours). This is usually started five or so day before the cool down to give the pump time to evacuate the space.

Pump out the three transfer lines using a turbo. Pump them out to $10^{-}9$ mbar/s. This includes the u-shaped jumper line, the L-shaped helium inflow line, and the main dewar transfer line. Pump each line out over night.

3 Flush out Fridge

Pump out the fridge with the separator pump and the white auxiliary mechanical pump. Pump down to -30 mmHg, then fill with He gas from the backfill to 0 mmHg

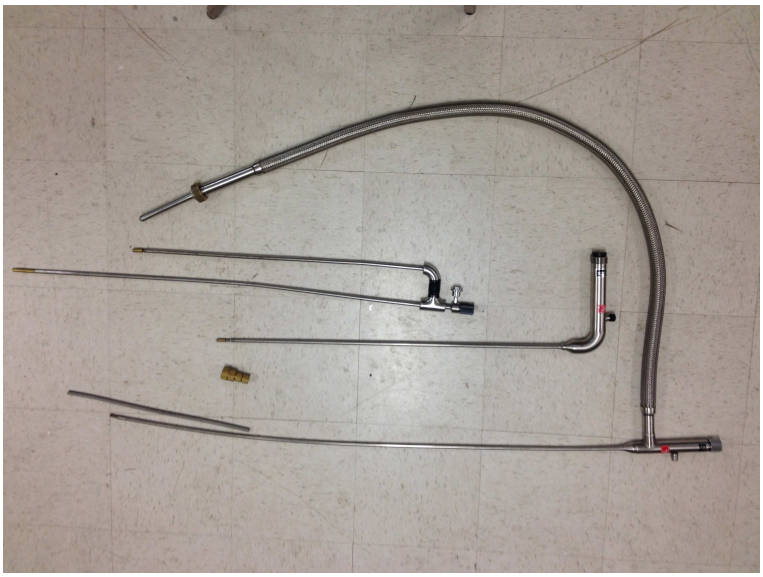

Figure 1: Transfer lines and goddard fitting. Show here is the u-shaped (jumper) the L-shaped transfer connector and the main LHe transfer line.

or more and pump out again with the separator and mechanical pump. Do this three or four times. In the final stage leave the fridge filled with He gas. With just a little pressure on fridge (over 780 mmHg) remove and clean bypass/run values and spray with Teflon lube.

4 Cool down the Magnet

Make sure the heat tape is on around the helium space O-ring area. Backfill to over 780 mmHg on fridge then proceed to fill the magnet. Make sure to fill the magnet space before the nitrogen shield. Fill the magnet space with Liquid Nitrogen (LN2). Open up the helium outlet (black pop-up value). Use the small copper transfer line to transfer LN2 to the magnet by connecting it from the dewar to the inner helium fill. Fill until orange connector (top of coil resistor) reads 141 Ohms, and green (bottom of coil resistor) reads 145 Ohms.

Fill the nitrogen shield with LN2. Use the outer elbow as the nitrogen fill with larger copper transfer line, open the nitrogen outlet. Fill until it overflows. Close gas fills (from backfill, etc...) and wait until the next day.

Remove the LN2 from the magnet space. Connect the nitrogen gas to the small helium outlet by the pop-up valve to pressurize. First remove pop-up valve and cap over and close other outlets. Connect helium inlet with small transfer line to a dewar to collect outgoing nitrogen, but first put baffle on the insert part of the transfer line. Use the larger of the two black helium space ports. You will need the small fitting to make the like fit the larger port. Turn on the nitrogen and push out LN2. You should see a steady stream of liquid, not a trickle (should expect to see more than 30

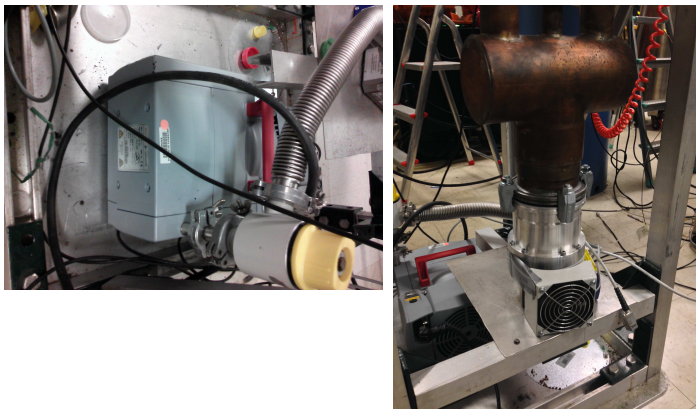

Figure 2: Electric pump (left) and The Pfeiffer Turbo pump (right) used to pump out the OVC/IVC.

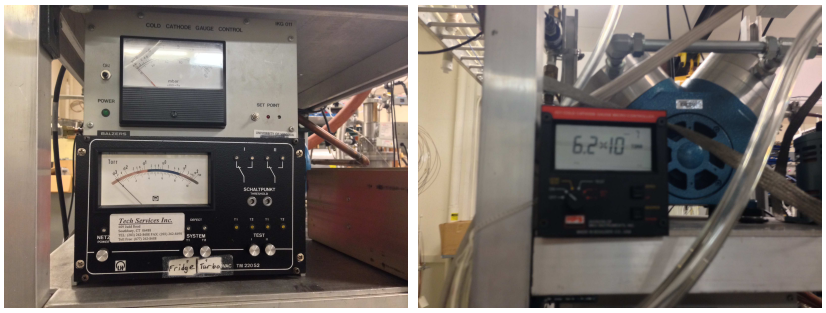

Figure 3: Meters used to readout pressure-rate in the vacuum space. The left is good down to $10^{(}-3)$ then you need the right.

litters).

After no more liquid switch to helium gas to push out LN2. Once no more LN2 vapor is coming out, disconnect the separator pump at the elbow and connect it to the helium inflow at the appropriate place by removing the top exit port. Fill with helium gas and pump it at least 4 times. The separator flow (Standard Liters per Minute) should go down close to zero when all LN2 and vapor are truly gone. Bring the separator flow down to about 25 inches of Hg each time. Then pressurize well over atmosphere with He gas. You can use the bottom coil resistor to check if liquid is gone. If the resistance does not increase when pumping then no liquid is present.

Fill the nitrogen shield again. Fill the magnet space with helium gas. Fill the separator line also with helium gas from the 'to separator pump' on the gas control valve board. (always pressurize lines before disconnecting to avoid air at all cost.) Backfill with He gas and then reconnect the separator pump to fridge.

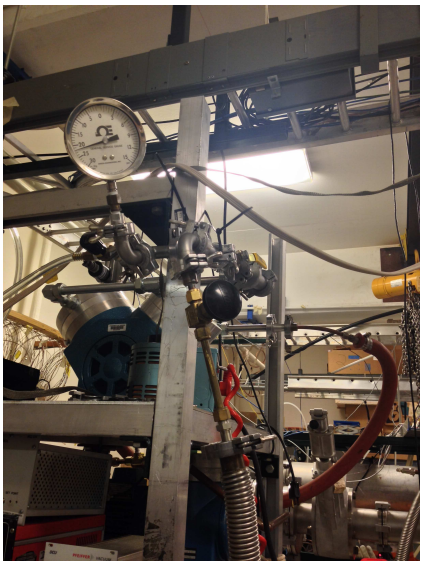

Figure 4: Separator value used to control the separator flow.

4.1 LHe Fill of the Magnet

The magnet volume is approximately 45 L. With this pre-cooling with LN2 is should take roughly 100 L to fill the first time. Open the outflow of the Helium and prepare to fill the helium space with Liquid Helium (LHe). Insert the 'L' into the larger of the two black helium fill holes (once this has been inserted once it will stay). Blow out transfer line and 'stinger' extension the with He gas for 5 minutes or so. Insert the long skinny end of the transfer line into the dewar. Make sure goddard fittings are on and transfer line has been blown out with helium. Have the dewar pressure connected to the helium gas ($\sim$2.5 lbs), but not yet open to the dewar. Close the dewar pressure release value. Open the transfer line up top 3/4 of the way. Slowly lower the line into the dewar and wait for the plume to come out of the other end. Once a plume is clearly present quickly connect the 'L' and the transfer line. It must be threaded quickly and all the way in order to avoid atmosphere from getting in. When the helium level probe readout says 80% you will be as full as you can be. Below 20% the magnet should be filled again. Keep track of two resistors in magnet space as well as OVC pressure. Two resistors should get to 1K ohms and OVC gets down to $3 \times 10^{-}6$. The top resistor in the magnet space is only at about halfway filled. Once that is around 1k ohm need just look at the level probe in the magnet dewar. The helium will plume as soon as you start the fill but will die out then start to plume extensively again once its filled. When the helium level probe readout says 80% you will be as full as you can be. Below 20% the magnet should be filled again.

Turn heat tapes on to around 25%, 25%, and 80% (by separator).

Backfill the fridge and put in u-shaped transfer (jumper) line in. To start filling the fridge after the jumper has been inserted close the run and bypass valves, start to pump with the separator, and first set of mechanical pumps (system start). Start with separator flow around 60. You can now start to look at the resistors in fridge

(R1-R7). R3 is for separator. Will go from 1.04 to 1.2 K ohms. Fill the separator full of LHe and pump down the nose space with run and bypass closed. To do this turn on roots. Turn on the 600 first then the two big Roots pumps. The two big ones will only come on below 1 Torr. Keep pumping down the nose space to calibrate manometer to zero pressure.

5 Fill Fridge

Fill fridge by opening up run valve one turn after separator resistor indicates mostly full. Once fridge full, fill nose with bypass, then close and set run valve so main flow is around 20 and set flow around 20.

Figure 5: The flow meters showing the separator flow and the main flow.

6 Fill Fridge

Standby is with separator flow down to 10 and mechanical pumps on (system start) with Roots off, run and bypass closed, LHe level probe off or slow, and heat tapes turned down 15%.

7 Microwaves

The microwaves are provided by the EIO tube, which allows the frequency of microwaves to be changed within limits by adjusting a bellows on the oscillation cavity. Wave guides carry the microwaves from the tube to a horn which shine on the target cups. The picture to the right shows the gold horn above (to the right of, here) the two target cups on the new target inserts.

The microwave system consists of the CPI power supply, the EIO microwave tube, the frequency counter and the wave guilds. About 20 watts comes out of the tube directly. There is a 30 dbm split dropping the power by a factor of 1000 then a 10

dbm split dropping the power by a factor of 10 which puts about 2 mW into the mixer (which has about a 10 mW damage threshold). You end up with about 20 l/s increase in the main flow when the microwaves have been turn on and all is working (10 l/s for every Watt). When connecting the mixer always ground the coax cable by shorting out the inner lead to the outer. To setup the counter use band 3-8 with center frequency usually at 140 GHz.

8 Microwaves for DNP

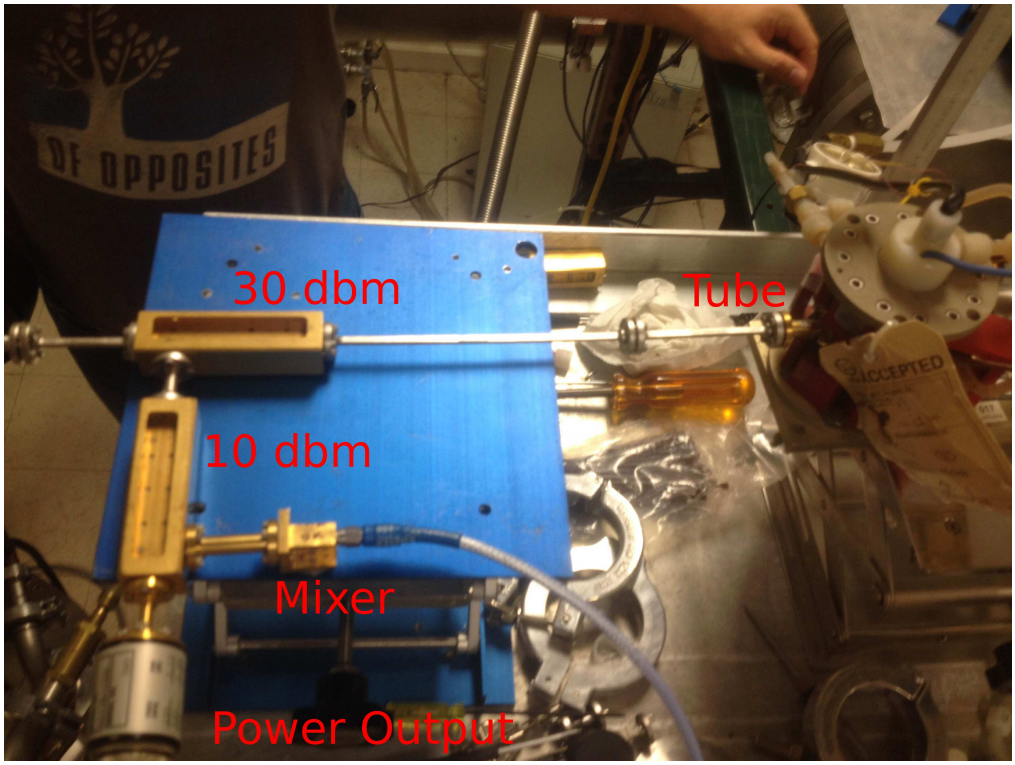

Figure 6: Microwave Attenuation

9 Taking Data

9.1 Baseline

To accurately measure the area of the NMR signal from polarization, we must carefully exclude any systematic changes in the NMR Q-curve background which are not due to polarization. To do this, we take a baseline measurement of the circuit's response without the polarization signal. This can be achieved by shifting the Larmor frequency of the proton out of range of our signal by changing the magnetic field. The baseline NMR signal is very sensitive to minute changes in the NMR circuit,

and it it important to make frequent baseline measurements to ensure an accurate polarization measurement. A baseline should be taken at least every every time there was a need to change the NMR tune. In addition to subtracting the baseline signal, a polynomial fit is performed to wings the NMR signal. This polynomial fit subtraction should remove any residual baseline signal and leave only the signal due to the target polarization. Follow this procedure to take a baseline.

1. Shift the field by changing the set-point on the magnet current by 0.5 amps
2. Press the "Pause" button to stop the NMR data collection
3. Press the "Baseline" button. A new dialogue box will pop up
4. Press the "Create New Baseline" button. The dialogue box will disappear
5. Change the number of sweeps to 2000, and make sure you press enter
6. Press "One Point". Wait for the countdown to finish
7. Select the baseline you made by pressing the "Baseline" button again
8. Return the holding field to its original position

9.2 Thermal Equilibrium Measurements

To calibrate our polarization we must measure the calibration constant, which relates area under the NMR curve to the proper polarization. To do this, we take advantage of the known polarization when the sample is at thermal equilibrium. After forming the calibration constant using this static, known polarization and the measured NMR area, we can apply this constant when the target is being dynamically polarized with microwaves. A thermal equilibrium measurement (or TE) requires removing the beam and the microwaves, setting the pressure and temperature in the nose to be as constant as possible, and waiting for the NMR area to stabilize. The relaxation time of the polarization depends on the temperature, so the temperature is raised above 1K to decrease the time spent waiting. Even so, this will likely take as much as an hour for NH_3 and possible much longer for other materials. The number and quality of the thermal equilibrium measurements directly affects the error on the target polarization measurement, so the TE should not be rushed. Afterwards, you can copy the events file to the Twist computer and important to the website to calculate the TE.

1. Make sure you have a STEADY Nose level (Roots off)
2. Have the run valve cracked (watch the nose temps.)
3. Set sweeps to 2000, if necessary
4. Hit "Take Data" button
5. Wait for timer to count down
6. Make entry in the logbook over the TE time
7. Write: Time, NMR Area, 4He Press, Temp
8. Watch data until it flattens out
9. Using at least 15 data points calcuate TE

http://twist.phys.virginia.edu/tools/te_calcnew.php
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