



NMR FACILITY

This manual is intended to provide additional instructions for the liquid helium fill for the magnets at the California State University LA NMR Facility. For more comprehensive information about this procedure, please refer to the documentation that Bruker provides. Most of the information in this manual was taken from different Bruker manuals and modified and edited to fit our needs for this facility.

***Dr. Ali Jabalameli**
October 10, 2008*

Cryogen Fills

Superconducting magnets must to be filled periodically with liquid nitrogen (N_2) and liquid Helium (He). In our facility, the liquid nitrogen fill is once every week, on Wednesday during available time slots. The liquid helium fill is once every 90 days, on Thursday (entire day). The fill time will be reserved on FACES and on the weekly-posted NMR schedule for each fill. Cryogen fills must be performed correctly to avoid injury to personnel and damage to the magnet, which could lead to the magnet quench. For this reason, cryogen fill is one the most crucial operation in our facility. Before each fill one should thoroughly review and understand all the steps for each procedure, and all the steps must be followed step by step. At the end of each fill procedure, a checklist is provided that must be completed as procedure proceeds.

Instructions for the liquid helium fill for the NMR magnets



SAFETY



&



PROCEDURE



SAFETY



Helium is an asphyxiant.

Release of helium or liquid helium in an enclosed area with poor ventilation can lead to possible suffocation. The effect of oxygen deficiency resulting from a simple asphyxiant may include: rapid breathing, diminished mental alertness, impaired muscular coordination, faulty judgment, and depress on all sensation. As asphyxiation progresses, nausea, vomiting, prostration, loss of consciousness may result, eventually leading to convulsions, coma, and death.

Helium is very cold!!

The temperature of liquid helium is -452 degrees below zero Fahrenheit (-269 degrees C, 4.2 K) and will cause severe burns to the skin. Contact with liquid helium may cause frostbite or freeze burn in exposed tissues. Frostbite effects are a change in color of the skin to gray or white, possibly followed by blistering. If burns occur, treat the injury like a heat burn and seek medical help immediately.

Safety equipment required for helium fills.

All personnel working with liquid helium should wear the following safety equipment.

Safety glasses or face shield

Insulated gloves

Protective clothing (long sleeves, pants, and proper shoes)

PROCEDURE (LHe)

Since liquid helium fills are done so infrequently, it is highly recommended that the process be reviewed prior to each fill. Fill the liquid helium a day after the liquid nitrogen fill.

❖ *Equipment*

❖ *Preparation*

❖ *Fill-up*

❖ *Follow-up*

Equipment

The helium fill requires the followings items (Must check a day before the fill)

- 1- A liquid helium tank and a brass adaptor for the helium transfer line 



Warning: Make sure to use the right size adaptor and check it for broken O rings prior to use.



- 2- A special liquid helium transfer line and flutter tube or thumper 



Warning: The transfer line could lose its vacuum. Therefore, it should be checked.



- 3- A cylinder of pure compressed helium gas, equipped with a double chambers regulator, appropriate pressure gauge (reading between 1 to 3 psi), and a long hose. Make sure you have a backup cylinder.



- 4- A non- magnetic step ladder or ladder.



- 5- A plastic cap for the helium outlet (supplied by Bruker)



Preparation

1-Measure the liquid helium level in the magnet:

Using the BSMS keypad (or BSMS Control windows within TOSPPIN), measure the liquid helium level in the magnet. Record the data.



2- Prepare the liquid helium supply tank and determine the volume of liquid helium:

- A. Slowly bleed and depressurize the helium supply tank to atmospheric pressure by opening the “vent valve”. Do not allow a white flame to form, and plumbing of the liquid helium tank to frost excessively (liquid helium is being wasted).



- B. Open the top valve on the liquid helium tank and slowly insert the thumper tube until it touches the bottom of the tank, and then slide both blue plastic markers on the thumper tube to the top of the liquid helium port (this indicates the bottom of the liquid helium in the tank) .
- C. Slowly, continue raising the thumper tube, while feeling the vibration by closing off the top end of the tube with your thumb (very loosely so that you can feel the vibration.) As soon as you feel the vibrations intensify and the frequency changes, stop raising the thumper tube and slide the lower blue plastic marker to the top of the liquid helium port (this indicates the top of the liquid helium in the tank). The sudden change in the observed frequency is caused as the end of the thumper tube passes from the liquid helium zone to the gas zone above the liquid.



- D. Finally, use a ruler to measure the distance between the two plastic markers on the thumper tube; this translates to the volume of the liquid helium in the tank. In order to find the volume, compare your measurement with the chart on the liquid helium tank, and record the data. Screw the brass adapter to the liquid helium port on the tank.



3- Create a clear working space around the magnet:

Place the helium supply tank next to the magnet and calibrate your working space to ensure the liquid helium transfer line can easily reach the fill port on the top of the magnet.

4- Prepare the high pressure helium gas cylinder:

First, determine a convenient place far away from the magnet and the magnetic field where you want to place the helium cylinder. Once you make sure that the transfer line for helium gas reaches the helium tank easily, calibrate the pressure to 1 psi and shut off the gas flow by turning off the needle valve.



5- Prepare the magnet for the fill:

- A. Slowly loosen and then quickly remove the helium check valve clamp, mounted on the back of the helium manifold and replace it with the plastic cap.



- B. Slowly loosen and quickly remove the black plug and cap assembly from the helium port and swiftly place the black plug back into the fill port funnel.



Warning: The magnet should never be left open to air for more than a few seconds.

- C. Plug the nitrogen ports on the magnet during filling of the helium dewar of the magnet. This step prevents cryopumping, a process whereby nitrogen, water, and oxygen are condensed out of the atmosphere into the nitrogen dewar due to the magnet stacks being cooled by the helium.

6- Prepare the liquid helium transfer line:

Place the black washer or the black screw cap assembly (similar to the one on the liquid helium fill port) on the short side of the liquid helium transfer line, which will be inserted into the magnet. Place a piece of black rubber hose on the long side of the transfer line, which will be inserted into the liquid helium tank.



Fill-up

1- Insertion procedure into the helium tank:

Close the safety valve and open both the vent valve and top valve on the liquid helium tank and slowly insert the helium transfer line (long side) into the tank (maintaining the tank pressure at 1 atmosphere) until the line is fully inserted. Should the line reach the bottom, pull the line back up about half an inch, tighten the brass adapter and place the black rubber hose to secure the transfer line position. Close the vent valve on the liquid helium tank. Purge the helium gas transfer line by opening the needle valve and then connect it to the helium gas supply. Be sure to maintain the 1 psi pressure that was adjusted on the pressure gauge on the helium gas cylinder.

2- Insertion procedure into the magnet:

Once the pressure builds up inside the tank, the helium gas will appear as a white vapor and then liquid helium will appear as a white flame along with helium gas. The flame should not exceed 6 inches. Frost may appear on the tip of transfer line. Remove the frost with a clean rag or glove and immediately remove the black plug from the helium fill port and insert the line into the magnet. If the line hits the bottom of helium stack, raise it up 2 inches and tighten the black screw. Immediately remove the plastic cap that was placed on the helium check valve port on the back of the manifold.



There should be a white vapor exhausting out of the port on the back of manifold. The white vapor should not exceed more than 10 inches in a horizontal plane from the manifold. Let the transfer continue until the exhausting white vapor intensifies. As soon as the white vapor intensifies, start monitoring the liquid helium level on the BSMS every 5 minutes.



Warning: If the white vapor exceeds more than 10 inches, reduce the pressure on the helium cylinder gas.

3- Fill Termination:

When the white flame becomes large and liquid air starts to drip off the manifold the helium can is full and the process should be stopped. The best way to determine if the magnet is full is to look for a change in the gas cloud coming out of the magnet vents. When the magnet is full, the cloud becomes very thick with a deep white center with a slight blue tint. Stop the fill by closing the vent valve on the liquid helium tank, disconnecting the gas connection to the liquid helium tank, and turning off the gas on the helium gas cylinder. Remove the transfer line from the helium fill port. The helium vents on the magnet should be closed promptly after the magnet is full; the fill port with the black plug and the port on the back of the manifold with the plastic cap. Remove the transfer line from the liquid helium tank. Close the top valve. Open the safety valve.



Warning: Monitor the helium level reading to confirm that the white flame and liquid air dripping are not due to the low liquid helium level in the tank or the high-pressure gas flow on the gas cylinder.



4- Fill Closure:

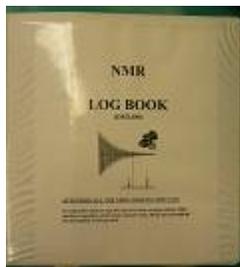
Swiftly replace the black plug and plastic cap with the original connectors and assemblies. Tighten all the valves and screws to ensure there is no leak. Measure the volume in the liquid helium tank as described earlier to determine the volume of the helium which was added during the fill. Measure the helium volume in the magnet from BSMS. Record the data in the book. Unplug the liquid nitrogen ports on the magnet.



Follow-up

1- Magnet follow-up:

After 30 minutes, you should double check all the valves again on the magnet to make sure everything is tight and there is no leak. Wipe off any water condensation formed on the magnet, ports, and manifold by the fill. Measure the helium level in the magnet again, record the measurement, and log the helium fill.



2-Liquid helium tank and helium gas cylinder follow-up:

Make sure all the valves are closed on the liquid helium tank (the safety valve must stay open) and the gas cylinder. Remove the brass adapter from the helium tank.



Liquid Helium Fill Check List

SAFETY

- Goggles Gloves Proper clothing

1- Equipment (Must check a day before the fill):

- Helium tank and brass adaptor
- Liquid helium transfer line and thumper
- Two helium gas cylinders with a pressure gauge and a hose
- Stepladder or ladder
- Plastic cap

2- Preparation:

- Measure the helium level in the magnet
- Prepare the liquid helium tank and measure the helium level in the tank
- Calibrate the workspace around the magnet by rehearsing the fill procedure
- Prepare the high-pressure helium cylinder for the fill
- Prepare the magnet for the fill
- Prepare the transfer line for the fill

3- Fill-up:

- Insertion procedure into the helium tank
- Insertion procedure into the magnet
- Fill termination
- Fill closure

4- Follow-up:

- Magnet follow-up
- Liquid helium tank and helium gas cylinder follow-up

Sign _____ head aid viewer
NMR _____ PS _____ Date _____

Instructions for the liquid nitrogen fill for the NMR magnets



SAFETY



&



PROCEDURE



SAFETY



Nitrogen is an asphyxiant.

Release of nitrogen or liquid nitrogen in an enclosed area with poor ventilation can lead to possible suffocation. The effect of oxygen deficiency resulting from a simple asphyxiant may include: rapid breathing, diminished mental alertness, impaired muscular coordination, faulty judgment, and depress on all sensation. As asphyxiation progresses, nausea, vomiting, prostration, loss of consciousness may result, eventually leading to convulsions, coma, and death.

Liquid Nitrogen is very cold!!

The temperature of liquid nitrogen is -320 degrees Fahrenheit (-196 degrees C, 77 K) and will cause severe burns to the skin. Contact with liquid nitrogen will cause severe burns to exposed tissues. If burns occur, seek medical help immediately.

Safety equipment required for Nitrogen fills.

All personnel working with liquid helium should wear the following safety equipment.

Safety glasses or face shield

Insulated gloves

Protective clothing (long sleeves, pants, and proper shoes)

PROCEDURE (LN₂)

❖ *Equipment*

❖ *Preparation*

❖ *Fill-up*

❖ *Follow-up*

Equipment

The nitrogen fill requires the following

- 1- A low pressure (<22 psi) non-magnetic stainless steel liquid nitrogen tank. 



Warning: Do not trust the pressure gauge; double check the release valve for the pressure rating. It is usually written on the valve.



- 2- One 7 ft rubber latex hose as a transfer line and two 3 ft rubber latex hoses for venting.



Preparation

1- Prepare the magnet for the fill.

Identify the fill port for the liquid nitrogen (it is usually marked). Gently remove two heaters from the two vent ports and replace them with two rubber hoses pointing outward, away from the magnet. These two rubber hoses are meant to direct the overflow liquid nitrogen away from the magnet seals, probe, and electronics.



2- Prepare the liquid nitrogen tank for the fill.

Connect the long rubber transfer line to the liquid port on the liquid nitrogen tank. Position the liquid nitrogen tank about three feet away from the magnet.



Fill-up

1- Purge the line.

Very slowly open the liquid valve on the nitrogen tank (<1 psi) to purge the transfer line for few seconds.



2- Connect the line and fill.

While the hose is venting, remove the vent cap from the magnet and attach the hose. Hold and keep the transfer line away from the magnet suspended in the air until the transfer line freezes.



Warning: The transfer line freezes quickly as the fill starts. Do not try to move the tank, or transfer line.

Warning: you must be sure not to exceed the recommended fill pressure and rate for your magnet.

3-Monitor the fill.

Visually check to insure that vapor is freely flowing out of both vent hoses. Look for the equal sized vapor trails on the both vent hoses



Warning: small or non-existent of vapor trail indicates that an ice block has formed inside the nitrogen can.

4-Terminate the fill.

Stop the fill as soon as you see excess liquid nitrogen flowing from the ends of the latex hoses. A crackling sound can be heard just before overflow.

Follow-up

1-Remove the line and replace with the cap.

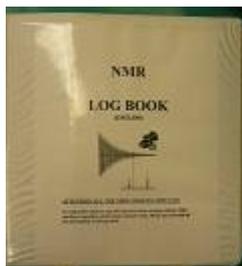
After the magnet is full, allow the transfer hose to thaw. Once the transfer hose is thawed, remove it and quickly place the vent cap back on the liquid nitrogen fill port on the magnet.



Warning: The magnet should never be left open to air for more than a few seconds.

2-Dry and log.

Wipe off any water condensation formed on the magnet and ports by the fill. Measure the helium level in the magnet, record the measurement, and log the nitrogen fill in the book.



Liquid Nitrogen Fill Check List

SAFETY

- Goggles
- Gloves
- Proper clothing

1- Equipment:

- A low pressure liquid nitrogen tank
- A 7 ft rubber latex hose as a transfer line.
- Two 3 ft rubber latex hose for the vent ports.

2- Preparation:

- Prepare the magnet for the fill
- Prepare the liquid nitrogen tank for the fill

3- Fill-up:

- Purge the line
- connect the line and fill
- Monitor the fill
- Terminate the fill

4- Follow-up:

- Remove the line and replace with the cap
- Dry and log.