

CSB NMR Facility

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N₂ Fill Tutorial for Vanderbilt Magnets

This page gives an overview of the cryogen filling procedures for our instruments at Vanderbilt.

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501, 502, 601, 602, 800

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INSTRUMENT	PERSON RESPONSIBLE
501 (Alka)	Young Jin Cho
502 (Milan)	Mark Ehrhardt
601 (Kite)	Brendan N. Borin
602 (Busard)	HakJun Kim
800 (Vulture)	Changlin Tian


- [Individual user](#)

1. Cryogen Tanks

General word about cryogen's:

- Boiling point of liquid Nitrogen: 77K or -196 °C, which is below that of oxygen
- Approximate expansion rate; volume of gas @ 15 °C, atmospheric pressure produced by a unit volume of liquid at normal boiling point: 700x
- Toxicity: minimal in well ventilated areas

It is important, that every container carrying liquid nitrogen has a permanent exhaust opening. Otherwise the dewar poses a real explosion hazard. Also, the boiling point raises from 77K @ 1 bar to 118K @ 20 bar.

	Liquid Nitrogen is extremely cold and can cause severe cold burns!
	Protective clothing: <ul style="list-style-type: none">◦ Wear gloves to prevent cold burns◦ Gloves must be loose fitting, so that they can be removed easily in case of a liquid spillage◦ Wear an appropriate eye protection

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Nitrogen

Supplier:

A-L Compressed Gas
875 Visco Drive
Nashville, TN, 37210
Phone: (615) 254 1457
Fax: (615) 254 1460

There are two kinds of dewars we usually have on site:

- 230/ high pressure liquid N₂ on wheels (~200 psi)

Used for the CD and UV spectrometer, sometimes on site for VT-air usage on the NMR instruments.

- 230/ low pressure liquid N₂ on wheels (max. 20 psi)

Used primarily to fill the magnets and sometimes for low temperature experiments on the spectrometer.

- There is standing N₂ order with A-L for both facilities.
- A-L typically delivers the dewars Monday mornings.
- It is good practice to have an empty dewar available by Monday mornings, so when a new dewar comes in, the empty one can be picked up. We have to pay extra for whatever dewar is sitting in our facility on the 1. of the month.
- Empty dewars can be called in if they are in excess of the one being picked up on Mondays anyway.

Normally the NMR staff will arrange for the nitrogen dewars to be on hand. If you need more, let them know. Should you have to call A-L, make sure you supply A-L with the proper P.O. We have a separate P.O. for the analytical facility and the structural biology one.

Delivery Slips:


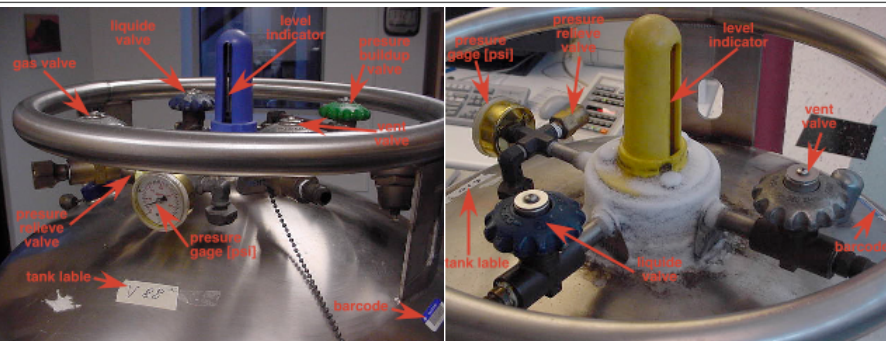
A-L most often leaves a yellow deliver slip on the tank. We collect them for book keeping reasons. Please deposit these slips at the following places:


NMR Facility: Shelf over the sink

Chemical Sciences NMR Facility: Leave delivery slips in the box @ Markus' office SC 5123

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Details about the cylinders

Liquid N2 tank on wheels	Typical arrangement of valves on a N2 tank
	 <p data-bbox="651 1129 1542 1192">Typical dual pressure tank (high/low pressure) on the left, single pressure tank (high OR low) on the right.</p>
<p data-bbox="370 1266 646 1360">1. After the tank is delivered, it needs to be labeled at the pressure gage with a:</p> <ul data-bbox="428 1381 586 1591" style="list-style-type: none">• The V is designated for the Structural biology facility• Just a number is used at the analytical facility <p data-bbox="370 1619 646 1766">2. The last 5 digits of the barcode, the tank number and the date need to be noted in the list posted at the cryogen door</p> <p data-bbox="370 1772 646 1887">3. As the magnets are being filled, note on this label, how many minutes you have used on which magnet.</p>	<p data-bbox="651 1255 1036 1283">Typical tank for high and low pressure</p> <ol data-bbox="678 1304 1542 1913" style="list-style-type: none">1. Vanderbilt internal label2. Pressure gauge [psi] indicates the pressure built up in the magnet.<ol data-bbox="732 1367 1317 1394" style="list-style-type: none">1. NEVER use for magnet fills if this is more than 15 psi!!3. pressure relieve valve: there is a 22 psi and 230 psi relieve valve depending on the dewar (high / low pressure). As the dewar stands unused, the pressure will build up and once it reaches the max. allowed pressure, air might blow out of this relief valve. It never should blow out with a loud noise. This would indicate, that the pressure build up valve is open and more gas is produced than desired.4. Gas use (silver): typically used for N2 gas on high pressure dewars on the CD, UV, ..., has a female fitting.5. Vent use (silver): open to relieve pressure if it is too high. CAUTION: very loud!! (do not confuse with Gas use port)6. Pressure Build up (green): Open this valve only if you need high pressure on a tank.<ol data-bbox="732 1703 1243 1730" style="list-style-type: none">1. NEVER use it for tanks intended to fill magnets!7. Liquid use (blue): This is the port (male) to hook up the rubber hose for magnet fills.8. Level indicator: The white plunger inside this housing is supposed to show the N2 level inside the dewar. Quite often this does not work properly! That's why we recommend making a note on the label, how long and on which magnet this dewar has been used. Another indication of the fill status can be obtained by shaking the dewar. You might "feel" the wobble of the liquid inside the dewar this way, but this need some experience.

	It is possible for the valves to freeze up. This most likely happens during a fill, when the valve was wet at the beginning. To prevent this from happening, move the valve every so often. There are wrenches to close a frozen valve in an emergency. Be aware that they are magnetic and will be attracted by the magnet!
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2. N₂-fills on the individual Instruments:

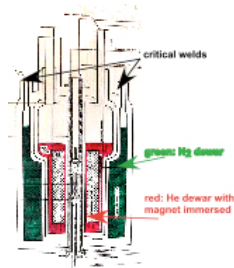
General:

The N₂-fill procedure is in general the same for all the magnets and so are the risks and safety precautions. These are some things to remember:

1. If you do not feel comfortable with what you are about to do, call for help!! Call on the person responsible for the system first, before you call Markus.

	<ol style="list-style-type: none">1. The most destructive thing during a fill is having liquid N₂ drop on to any O-ring, cable or other electronics like the preamplifier. <p>Therefore:</p> <ul style="list-style-type: none">▪ Make sure the hoses point away from the magnet and all cables / electronics▪ Never leave the magnet while filling it▪ Check the liquid valve periodically for freeze up <p>Check the pressure on the hose; a good flow is indicated by the hose slightly vibrating.</p>
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2. Always wear protective gear: Safety glasses and gloves
3. Never move the dewar during a fill, the rubber hose will break and cause liquid N₂ to spill, causing possible burn wounds or damage to the magnets.
4. The N₂ dewar is a cylindrical can wrapped around the He can inside the magnet dewar. Because of heat transfer, the can is free hanging on the three or four Nitrogen ports, only supported by some spacers on the bottom. Therefore care has to be given to the welds on top of the fill ports. Make sure they are not iced up, when you start a fill.



5. **Never leave the N₂ ports open any longer than absolutely needed for the changes of tubes or heat exchangers.**
6. **Always make sure there is positive pressure on the N₂ ports, e.g. N₂ gas flowing out.**

If the fill is being done carefully, there is nothing really that can happen to the magnet.

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Main steps to be performed:

1. Release the air from the anti-vibration legs

- o **501:** No anti-vibration legs
- o **502:** Shut off valve on the wall ([air usage for anti-vibration legs](#)).

This takes quite a while (up to 5 min) to release all the air.



- o **601, 602:** Shut off valve on the rear magnet leg.



- o **800:** Anti-vibration legs not in use

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2. Close the magnet bore:





The magnet bore, where the sample is inserted needs to be closed with the black stopper. This prevents any moisture to drop into the magnet.

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3. Move dewar close to the magnet:

- Be careful not to move the dewar through the 5 G line of any other magnet, that might have an experiment running.
- Make sure the handle points away from the magnet. On some dewar it comes loose very easily and could fall onto the magnet.
- Get the dewar relatively close to the magnet, but don't hit the magnet. Also remember, should the valve freeze, you don't want to be too close, so you can bring the necessary tools.
- Be careful maneuvering the dewar through narrow door frames and along walls - take your time!

	When filling the 800: Hook up the hose in the wet-lab area. You will need a metallic wrench, that is highly attracted by the magnetic fields. Leave the wrench in the wet-lab area at all times!
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501:	502:	601 / 602:	800:
			
Leave the dewar in front of the wooden wall with the liquid port facing the magnet	Place the dewar in a spot, where it feels comfortable working with it.	Place the dewar in a spot, where it feels comfortable working with it.	Move the dewar close to the corner where the stairs meet the platform.

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4. Set up of magnets for N₂ transfer:

It is important, that the N₂-fill is done consistently, e.g. the fill tube from the dewar should always be hooked up on the same port of the magnet.

- A label indicates which port to hook up

- Material:

- **The rubber transfer hoses:**

NMR Facility: For the 502, 601 and 602 the transfer hose is at the sink in the wet lab of the facility. The 800, the transfer hose with the metal adapter attached on both sides is on the magnet supply box behind the magnet.

Chemical Sciences NMR Facility: The transfer hose for the 501 is at the sink next to the 400 magnet (SC 5132C).

- **Hoses for vent port:**

- 501, 502, 601 and 602: The short vent tubes are on the vibration legs or next to the magnets.

- 800: The metal flex tube and the yellow rope to fix the tube on the rail are behind the magnet supply box.

o **Heat gun and gloves:**

NMR Facility: They are in the drawer across the refrigerator in the wet lab.

Chemical Sciences NMR Facility: They are in the drawer underneath the refrigeration unit next to the 400 magnet (SC 5132C).


- Individual steps:

501, 502, 601, 602:

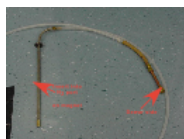
1. Hook up the rubber hose on the liquid outlet of the N₂ dewar, make sure it fits well over the male threads.
2. Remove heat exchanger on the fill port of the magnet. **Make sure there is no ice.**
3. Open the valve on the liquid port on the dewar and let it run a few seconds, then shut it off again. The purpose of this is:
 - Flush the tube with N₂
 - double check the pressure in the tank
 - Don't go too long or the hose might freeze or get too moist.
4. Hook the tube up to the fill port on the magnet. **Make sure it is tight, particularly on the 502!**
5. Remove the other vent ports on the magnet as necessary on each system (see pictures)
6. **Hook up the small tubes on these vent ports and direct them away into the open space.**
7. Slowly open the liquid valve on the dewar and start the fill. As the rubber hoses get cold, make sure they are properly positioned. This is particularly important for the vent hoses!!
8. Increase the N₂ flow slowly until a good transfer is achieved indicated by a slight vibration of the rubber hose.

800:

1. **When filling the 800:** Hook up the hose in the wet-lab area. You will need a metallic wrench, that is highly attracted by the magnetic fields. Leave the wrench in the wet-lab area at all times!
2. Move the dewar with the hose attached close to the 800 magnet.
3. 800: open the clamp on the fill port



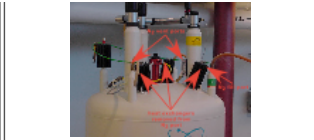
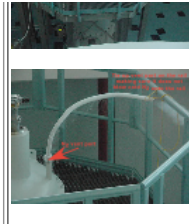
	Careful: <ul style="list-style-type: none">▪ N₂ dewar is under pressure!▪ Screws on clamp are slightly magnetic▪ Watch out for the O-ring!
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4. Open the valve on the liquid port on the dewar and let it run a few seconds, then shut it off again. The purpose of this is:
 - Flush the tube with N₂
 - Double check the pressure in the tank
 - Don't go too long or the hose might freeze or get too moist.
5. 800: Insert the metal rod into fill port and close it with the clamp.



6. Remove the other vent ports on the magnet as necessary on each system
7. 800: Open the clamp on the vent port (right side) and hook up the metal flex tube using the clamps. Tie up the flex tube on the rail, making sure the N₂ gas blows into the open space.
8. Slowly open the liquid valve on the dewar and start the fill. As the rubber hose gets cold, make sure it is properly positioned.
9. Increase the N₂ flow slowly until a good transfer is achieved indicated by a slight vibration of the rubber hose.

501:	502:	601 / 602:	800:
			

			
<ol style="list-style-type: none"> 1. Fill port is on the left 2. Vent ports in the rear and on the right 	<ol style="list-style-type: none"> 1. Remove heat exchanger, an O-ring holds it tight on the N₂ port 2. This port is the vent port 3. fill port is on the left side 4. Do NOT remove anything on the back port 	<ol style="list-style-type: none"> 1. Remove and place the heat exchangers safely around the N₂ ports 2. fill port is on the right, front 3. vent ports are on the right back and left front 4. Do NOT remove anything on the back left port 	<ol style="list-style-type: none"> 1. Fill port is on the left 2. Vent ports in the rear and on the right <p>Careful:</p> <ul style="list-style-type: none"> - N₂ dewar is under pressure! - screws on clamp are slightly magnetic - watch out for the O-ring!

Make sure all the vent tubes are directed away from the magnet into empty space!

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5. Fill N₂:

As stated above:

- o Make sure everything is hooked up properly
- o Work expeditiously
- o Start the fill by slowly open the liquid valve on the dewar and start the fill.
- o As the rubber hoses get cold, make sure they are properly positioned. This is particularly important for the vent hoses!!
- o Increase the N₂ flow slowly until a good transfer is achieved indicated by a slight vibration of the rubber hose.
- o Increase the flow by opening the liquid valve to maintain the slight vibration of the rubber hose
 - 800: One can see the liquid N₂ flow in the tube
 - Depending on the flow, a hum might be heard, which is normal
 - 502: One can check the N₂ level on the monitor
- o Check the liquid valve for operation, making sure it does not freeze up
- o Do not fill too fast, a fill after 7 days should last about:
 - 501: 25 min
 - 502: 30 min
 - 601, 602: 40 min
 - 800: 50 - 60 min


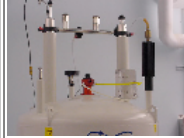


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6. When is the magnet full ? - closing it off

- Once liquid N₂ spills out on the open port, the magnet is full
- The liquid valve on the dewar needs to be closed immediately
- Use the heat gun to blow off the snow formed on the fill- and vent hoses
- Warm up the fill hose at the port, remove and close it:
 - 501: rubber hoses connecting to the flow meter
 - 502: pressure valve fitting
 - 601 and 602: heat exchanger
 - 800: cover flange with clamp
 - make sure it is closed tightly
 - position the screw properly since it is slightly magnetic (see picture)
- Immediately after, remove the vent tubes from the ports and lose them:
 - 501: rubber hoses connecting to the flow meter
 - 502: heat exchanger
 - Caution:
 - Make sure the port is completely dry, otherwise the O-ring inside the heat exchanger does not close properly
 - Make sure the connecting tube at the top is not kinked and the N₂ gas can flow out freely, the hose might have to be supported by the clamp screw of the He-port
 - 601 and 602: heat exchanger
 - 800: cover flange with clamp
 - make sure it is closed tightly
 - position the screw properly since it is slightly magnetic (see picture)



Properly closed off Nitrogen ports:

501:	502:	601 / 602:	800:
			
All 3 ports are connected back to the rubber hoses combining and connecting to the N ₂ flow meter. The white towels soak up any condensing water and need to be tight around the N ₂ stacks. Change them when dirty or unclean!	Make sure the hose after the heat exchanger is not kinked!!	The heat exchangers need to be seated properly on the pots again.	The clamps on either side need to be oriented properly and closed well. Inspect the O-ring for cracks before putting it back on.

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7. How do I recognize an empty dewar during a fill?

The following signs indicate a dewar running empty during a fill:

- The most obvious sign is given by the transfer hose stopping to vibrate. One might recognize this in two ways:
 - visually
 - touch the transfer hose and it will be calm
- 800: A visual inspection of the transfer hose does not show liquid anymore
- 502: The N₂ level on the monitor does not increase

If you suspect an empty dewar stop the fill and close the ports off properly. Continuing to blow N₂ gas into the magnet dewar depletes it from liquid N₂ to the point where it might run empty!

Therefore it is very helpful if the label on the N₂ dewar has a proper list of which magnet has been filled for how many minutes with this dewar. (See note in the introduction of the dewars)

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8. Finishing and clean-up

- Turn on the air for the vibration legs on the 502, 601 and 602
- Remove transfer hose from the dewar and put it back to its place:
 - For the 502, 601 and 602 is at the sink in the wet lab of the facility, the one for the 501 is at the sink next to the 400 magnet.
 - For the 800, the transfer hose with the metal adapter attached on both sides is on the magnet supply box behind the magnet.
- Store hoses for vent port:
 - The short vent tubes are on the vibration legs of the 502, 601 and 602, and in the 501 room
 - The metal flex tube and the yellow rope to fix the tube on the rail are behind the magnet supply box
 - In the wet lab of the facility, in the drawer across the refrigerator
 - In the drawer underneath the refrigeration unit next to the 400 magnet
- On the label of the N₂ dewars note, which magnet you have been using this dewar on and for how long. Then store the dewar:
 - In the cryogen room at the facility
 - behind the service elevator in SC 5113
- If the dewar is empty, take a new label and clearly mark the dewar as EMPTY.

Double check, whether you see the N₂ flow meter pick up some flow and the place is cleaned up.

Make the appropriate entry on the web page:

[Nitrogen Fill Log Page](#)

1. select spectrometer and click go
2. add your username (the one you use for NMR time reservations)
3. add the date
4. make any comments like partial fills
5. click add to post the N₂ fill on the schedule

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3. Organization

Responsibilities:

- **People in charge of a magnet system:**

The people in charge of a system have the following responsibilities:

INSTRUMENT	PERSON RESPONSIBLE
501 (Alka)	Young Jin Cho
502 (Milan)	Mark Ehrhardt
601 (Kite)	Brendan N. Borin
602 (Busard)	HakJun Kim
800 (Vulture)	Changlin Tian

- 1. First and foremost, the person responsible for any magnet system has to make sure that the magnets are being filled with liquid N₂ every week. A magnet should be filled within 7-10 days of a previous fill. Exceptions might be discussed with Markus.**
- 2. If this person is out of, the NMR staff has to be informed about the substitution plan.**
- 3. If nobody is signed up on a spectrometer, this person is responsible to organize the fill for that day/week.**
4. These persons are the first contact if a user has a question.
5. Help new users filling the magnets.
6. Initially, they might be present and assist during each fill

- **Individual users:**

Each individual user has the following responsibilities:

- 1. Fill the magnet with liquid N₂ on the system he is using, whenever the schedule indicates a fill needed.**
2. Make sure in advance there is enough liquid N₂ on site, otherwise inform the NMR staff
3. If you can not do the fill or need some help, make the appropriate arrangements

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