

## Ultra Low Temperature Instructions - *Exchanger*

**ONLY DPX 400 MHz EL0083**  
**Lowest possible temp: -120°C**

Similar procedure for 600 MHz in CBEC 092 except the instrument already is using the house N<sub>2</sub>(g), but the exchanger will need to be connected in a different manner. Ask the NMR manager for specifics of setting up the exchanger with the 600 MHz. Temperature range on the 600 MHz using the exchanger: -20°C to -120°C

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### FOLLOW DIRECTIONS TO AVOID HARDWARE DAMAGE!!!

#### Preparation

USE Variable temperature spinner and make sure your solvent is appropriate for the temp range needed.

***Solvent should NOT freeze or boil +/- 10 degrees from range.***

Bring a full N<sub>2</sub> gas cylinder into the facility – do not bring the tank anywhere near the NMR magnets! This tank may only last 8-10 hours depending on the gas flow used. If your experiment lasts longer, you will need to contact the NMR manager.

Fill the NMR VT liquid N<sub>2</sub> dewar with liquid N<sub>2</sub> that is supplied by your lab. Please do not remove this dewar from the NMR lab. It must be completely full to work properly.

At the VT control unit, connect the **exchanger** cable to the N<sub>2</sub> position. Select the 'Exch' option. Go to the NMR magnet and make sure there is gas flow to the shim stack.

Do not spin your sample, because this will require more N<sub>2</sub> gas usage. At extreme temperatures the sample may not be able to spin due to the large flow of gas in the probe. If not spinning, you may want to acquire your room temperature example also without spinning for better comparison.

#### Check Default temperature settings

Type **edte** to open the temperature control window. Before attaching any of the cooling apparatus or N<sub>2</sub>(g), make sure the following settings are correct (default parameters at all times except during VT exp):

- Temperature should be set to 27 °C
- Heater should be ON at 2% maximum power
- Cooler will read EMPTY
- Gas flow should be at 400 l/min

#### Acquire a Proton Spectrum at Room Temp

Create a new dataset by typing **new** and naming your experiment as needed.

Lock, shim and run a normal <sup>1</sup>H NMR experiment (using **rpar** and **gpro**). If you have been trained to tune the probe type **wobb** for tuning before acquiring the <sup>1</sup>H spectrum.

#### Set up required N<sub>2</sub> gas supply

Heater should be turned OFF at 2% maximum power

Attach the N<sub>2</sub> gas cylinder (supplied by your lab) to the regulator from the line on the wall.

Switch the house air off, switch the N<sub>2</sub> gas on (at the wall), open N<sub>2</sub> cylinder valve so that this dry N<sub>2</sub> gas flows to the instrument. Make sure there is plenty of N<sub>2</sub> gas in your tank and that there is pressure flowing (no less than 60 psi).

Heater should be turned ON at 2% maximum power  
Gas flow reduced to 135 l/min

### Set up required liquid N<sub>2</sub> supply

Place the **exchanger** into the dewar slowly (use goggles) – keeping any N<sub>2</sub>(l) spray away from the NMR magnet and surrounding cables.

Unhook the room temperature gas supply from the probe. This green tubing line should be attached to the **exchanger**. Attach the black N<sub>2</sub>(g) transfer from the exchanger to the probe. The liquid N<sub>2</sub> will be evaporated through this apparatus to cool the probe and your sample.

### Reducing the temperature

In the **edte** window set the following parameters and wait.

Temperature should be set to 20 °C  
Heater should be ON at 2% maximum power  
Cooler will read FULL  
Gas flow should be at 270 l/min

Wait for the temperature to begin to decrease. Continue to reduce the temperature by 5-10 °C increments. **Do not change the temperature more than 10 degrees from actual reading at a time or you risk damaging the probe and potentially the NMR magnet!** The gas flow may need to be increased slightly during cooling and/or to retain the temperature desired. Please use the values in the table.

Temperature	Heater	Gas Flow to cool	Gas flow to stabilize	Exchanger Attached
27 (room temp)	2	---	400 l/min	no
20	2	270 l/min	135 l/min	yes
0	2	270 l/min	135 l/min	
-10	2	270 l/min	135 l/min	yes
-20	2	270 l/min	270 l/min	yes
-50	2	270 l/min	270 l/min	yes
-70	2 (3)	400 l/min	400 l/min	yes
Repeat, then return				
-20	2	135 l/min	---	yes
0	2	135 l/min	---	yes
20-27	2 (3)	135 l/min	---	yes

### Acquire NMR experiment

When you have reached a desired temperature, wobb, shim, and acquire the NMR experiments needed.

### Increasing the temperature to 20 °C

When the NMR experiments are complete, increase the temperature by 5-10 °C increments. **Do not change the temperature more than 10 degrees from actual reading at a time or you risk damaging the probe and potentially the NMR magnet!** Decrease the gas flow as you heat the probe slowly until you eventually reach 135 l/min.

**Never reduce the gas flow to 0 l/min while the exchanger is in the liquid nitrogen and attached to probe. The heater will automatically turn OFF causing the probe to cool quickly to liquid nitrogen temperature!**

### Removing N<sub>2</sub> apparatus

Stop increasing the temperature at 20-27 °C set the following parameters

Temperature should be set to 20 °C  
Gas flow should be set to 135 l/min

Quickly exchange the liquid N<sub>2</sub> supply (at the probe) with the room temperature gas supply (green line on liquid N<sub>2</sub> supply) back. Make the switch quickly!

Gas flow should be set to 400 l/min  
Heater should be ON at 2% maximum power

Watch the temperature in the **edte** window. The probe temperature may *decrease* slightly once the room temperature supply is now going to the probe.

Set the **edte** parameters back to the default settings making sure the probe returns to room temperature.

Temperature should be set to 27 °C  
Heater should be ON at 2% maximum power  
Cooler should be OFF at 0% maximum power (flashing EMPTY)  
Gas flow should be at 400 l/min

### Switching the N<sub>2</sub>(g) to House Air:

When the temperature reaches 27 °C:

Heater should be turned OFF at 2% maximum power

Close the valve on your N<sub>2</sub> gas cylinder and let the air bleed out. Switch the N<sub>2</sub> gas (at the wall) off, switch on the house air. Remove the regulator from the gas cylinder carefully as there may be some pressure buildup in the line.

At the VT control unit, connect the **evaporator** cable to the N<sub>2</sub> position. Select the 'Evap' option.

Remove sample.

**DO NOT LEAVE THE INSTRUMENT FOR THE NEXT PERSON IF ROOM TEMPERATURE HAS NOT BEEN ESTABLISHED!**

**IT WILL TAKE at least 30 MINUTES FOR THE INSTRUMENT TO RETURN TO ROOM TEMPERATURE FROM -50 °C.**