

Low Temperature Instructions

ONLY 400 MHz in EL0083

Using the EVAPORATOR (lowest temp allowed: -90°C)

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 N₂ gas cylinder into the facility – do not bring the tank anywhere near the NMR magnets!

Fill the NMR VT liquid N₂ dewar with liquid N₂ that is supplied by your lab. Please do not remove this dewar from the NMR lab. It must be completely full to work properly. Place the evaporator apparatus into the dewar slowly (use goggles) – keeping the spray away from the NMR magnet.

You may spin your sample during acquisition, but realize this will require more N₂ 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, 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 should be OFF at 0% maximum power (flashing 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 ¹H NMR experiment (using **rpar** and **gpro**). If you have been trained to tune the probe type **wobb** for tuning before acquiring the ¹H spectrum.

Set up required N₂ gas supply

Heater should be turned OFF at 2% maximum power

Attach the N₂ gas cylinder (supplied by your lab) to the regulator from the line on the wall.

Switch the house air off, switch the N₂ gas on (at the wall), open N₂ cylinder valve so that this dry N₂ gas flows to the instrument. Make sure there is plenty of N₂ gas in your tank and that there is pressure flowing (no less than 60 psi).

Heater should be turned ON at 2% maximum power

Set up required liquid N₂ supply

Unhook the room temperature gas supply from the probe and attach the black liquid N₂ tubing to the probe. Make this switch quickly! The liquid N₂ is 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 should be ON at 8% maximum power
Gas flow should be at 400 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!** Increase the cooling power slowly (1% per 5 degrees) as you cool the probe as shown during training. You should only need 15-20% power for cooling. If the temperature doesn't drop, there is probably an issue with the seal at the LN2 evaporator.

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 cooling power as you heat the probe slowly.

Removing N2 apparatus

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

Temperature should be set to 20 °C
Heater should be ON at 2% maximum power
Cooler should be ON at 0% maximum power
Gas flow should be at 400 l/min

Go to the probe and quickly exchange the liquid N₂ supply with the room temperature gas supply (original line into the probe). Make the switch quickly! Watch the temperature now carefully in the **edte** window. The probe temperature may increase slightly once the room temperature supply is now going to the probe.

Once the temperature is stable again at 20 °C 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

Switch the N₂ gas (at the wall) off, switch on the house air, and close the valve on your N₂ gas cylinder. Remove the regulator carefully as there will be some pressure buildup in the line.

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.

IT SHOULD TAKE -ONE HOUR OR LONGER TO COOL TO -50 °C.