**UNC-CH Chemistry NMR Facility**  
*Low Temperature Set-Up Procedure on the Bruker 500 using Topspin 3.5*

**Gas lines:**
- VT (variable temperature) gas line: black line, connects console and probe for room T and high T runs & connects the console and the transfer line for low T runs
- Secondary house nitrogen line: black line from the connection on the wall, used to flush the transfer line
- Transfer line: large, insulated metal line used with the heat exchanger

**Experiments need to be completed with ample time before your scheduled end time to allow enough time to reset everything back for room temperature use.**

1. Connect the LN2 VT Power box, located on the floor behind the magnet.
2. Connect the flush gas line to the transfer line.
3. Flush the transfer line for 5-10 minutes: open the N2 gas valve on the wall (turn yellow lever up a little bit to start the N2 gas flow).
4. While waiting, open the temperature control window: type “edte.” Under the temperature tab, set the standby gas flow rate as 400. Under the monitoring tab, check current temp, current power, gas flow.
5. Place a LN2 dewar filled with LN2 on the appropriate taped area to the right of the magnet (circles are drawn on the floor for precise placement)
6. Disconnect the flush line from the transfer line.
7. Disconnect the VT gas line from the probe and remove the connecting clamp at the end of the gas line. Connect the VT gas line to the connector on the heat exchanger.
8. Slowly place the heat exchanger in the dewar. Progress slowly so liquid nitrogen does not splash out of the dewar. Wear gloves and safety glasses.
9. Attach the heat exchanger to the probe. Be sure to adjust the location of the dewar so that the line is sitting snug and aligned with the VT gas connector on the probe. Clamp connection (snug, not tight).
10. Turn off the yellow N2 valve on the wall.
11. Use the appropriate variable temperature spinner (grayish beige in color). The regular blue ones cannot accommodate low or high temperature settings.
12. Open the temperature control window. Turn VT on. The temperature should be changed gradually in **no more than 10 degree increments** every 5-10 minutes, allowing the temperature to stabilize before shimming or acquisition. Ask NMR staff before you go below **-80 °C**. Monitor the temperature change and stability using the current temperature and set temperature graphs.
13. Shim at each step in temperature to maximize performance at low T. If topshim fails, type “topshim convcomp” to auto shim at lower temperatures.

14. If the instrument has a hard time getting to lower temperatures, adjust the target gas flow to 400-700. Do not go above 900 (you shouldn’t have to)!

15. Once finished with your experiments, slowly cool NMR back to room temperature (~10 ° every 5-10 minutes), allowing the temperature to stabilize at each step.

16. Once the temperature is stable at 15 °C, disconnect the transfer line from the probe and disconnect the VT gas line from the heat exchanger.

17. Reconnect the black VT gas line to the probe.

18. Disconnect the cable to the “LN2” power box, on the floor behind the magnet.

19. Set the standby gas flow to 300 lph (within the temperature control window of Topspin).

20. Check the temperature reading in the probe in the temperature control window.

21. Take the heat exchanger out of the liquid nitrogen dewar and carefully place the cold transfer line on the table. Wear gloves.

22. Flush the transfer line with the house nitrogen line until the line is nearly room temperature (all condensation is gone, may take up to 20 minutes) by opening the valve on the wall to turn on the gas flow.

23. After the transfer line has warmed up, turn off nitrogen valve on the wall.

24. Log off.