

Variable Temperature



Sample Preparation

- Make sure the probe is rated for the ultimate/final temperature
- Fully degas the sample, particularly if going to low temperature
- Be aware of and do not exceed the boiling point/freezing point of your solvent:

Solvent	MP (°C)	BP (°C)
Chloroform	-63.4	61
Dimethyl Sulfoxide	18.4	189
Acetonitrile	-46	82
Acetone	-94.3	56
THF	-108.4	66

- Make sure your sample tube cap is secured with parafilm wax or use a J-young tube. When increasing temperatures, pressure in the tube may buildup and pop your cap off.



Select the Proper Spinner



Standard POM Spinner
0°C to +80°C



Kel-F Spinner
+80°C to +120°C



Ceramic Spinner
+120°C to +180°C and
0°C to -150°C

Sample T [°C]	-150 ... -80	-80 ... -40	-40 ... 0	0 ... 80	80 ... 120	120 ... 150
VT gas [l/h]	1800 ... 1100	1100 ... 800	800 ... 700	450 ... 350 (POM) 700 ... 500 (Kel-F)	500 ... 400	400 ... 300
Recom. VT gas [nl/h]	1200	1000	750	400 (POM) 600 (Kel-F)	450	350
Shim gas [l/min]	20	20	20	0	0 ... 20	20 ... 60
Flush gas [l/min]	5 ... 10	5	5	0	5	5
Spinner	Ceramic	Ceramic	Kel-F	POM / Kel-F	Kel-F	Ceramic



Before you begin...

- All probes in the NMR Core facility use probes are rated to handle temperatures between -150 to 180°C, however limits have been set.
- High temperatures are achieved using a heating element within the probe. This can be done on all probes.
- For low temperatures, external sources must be used for cooling the sample. There are three sources available:
 1. FTS Chiller (NCB400)
 2. BCU unit (REM400)
 3. liq-N₂ (REM300)
- Depending on the temperature range you will need to use nitrogen or air as the carrier gas.

Temperature Range	Cooling Device	Gas
+50 to +100 °C	None	Nitrogen
+20 to +50 °C	None	Dry Air
-150 to +20 °C	l-N ₂ or Chiller System	Nitrogen

Prior to booking time, ensure that you have nitrogen gas tank on-hand. All students are responsible for providing their own liq-N₂ and nitrogen gas when doing VT experiments.

- The N₂ gas cylinder should be secured to the wall.
- **Before changing to the VT setup, insert and acquire a room temperature ¹H spectrum.**



Chilling Units



BCU-I Chiller (REM400, UTL400)



FTS Chiller (NCB400)

- Air passes through the chilling unit and is cooled through the mechanical refrigeration system.
- Temperature regulation for BCU-I Chiller units are done through the Topspin interface. The FTS Chiller is done manually on the unit using keypad arrows.
- Always attached to the probe.



liq-N₂ Dewar Setup

- What you will need:



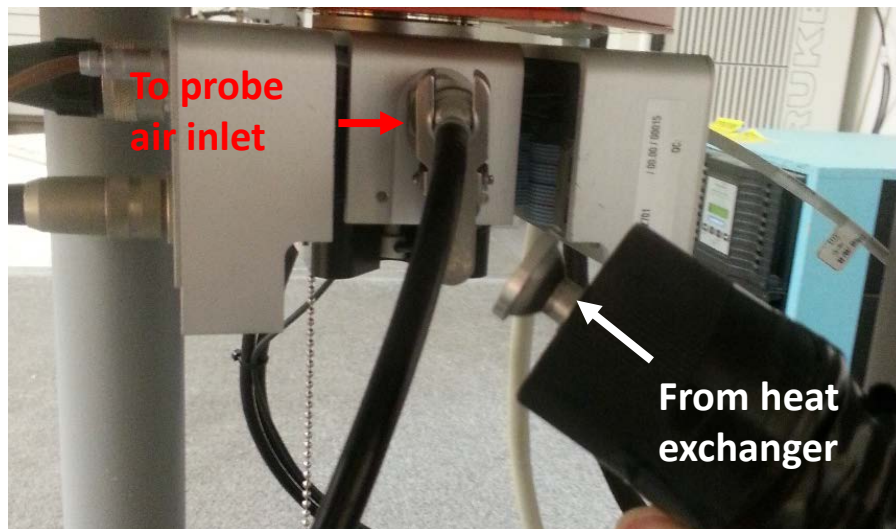
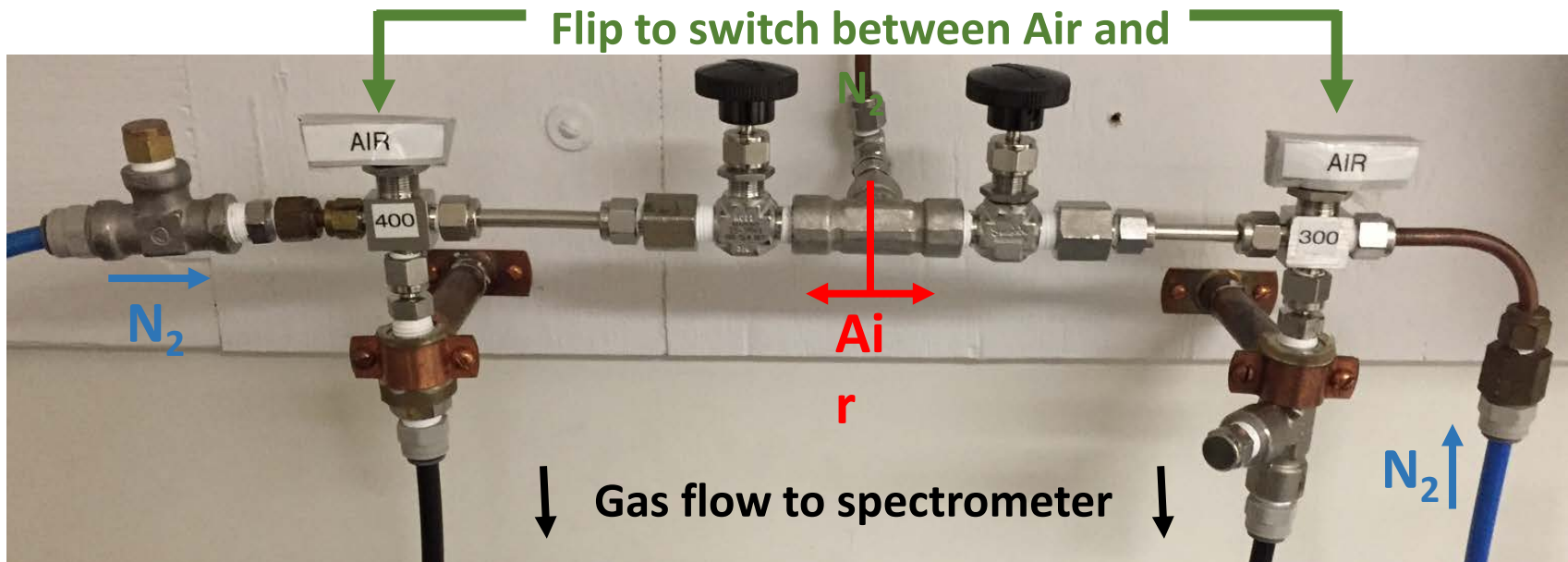
O-Ring and Clamp

- Assemble the VT unit by first placing the o-ring on the opening of the dewar.



- Insert the heat exchanger into the dewar
- Secure the heat exchanger onto the dewar using the clamp
- Using the digital scale make note of the total mass of the dewar assembly. This will be used to keep track of how much liquid nitrogen is remaining in the dewar. (NOTE: remember to tare the scale before measuring).
- Disassemble the unit and fill the dewar with liquid nitrogen.
- Replace the dewar on the scale, slowly insert the heat exchanger back into the dewar and secure using the clamp. Make note of the initial mass for a full tank.

N₂ Gas Flow



- Open the N₂ gas tank and set the pressure to 20 psi.
- On the wall behind the magnet there is a valve regulating air/N₂ gas flow for each magnet. Switch the valve to flow N₂ gas to the console.
- Remove the air tube on the probe by unfastening the metal clip. **CAUTION: THE CLIP IS MAGNETIC!** Connect the gas outlet of the heat exchanger to the gas inlet of the probe.

N₂ Gas Tank

- The main purpose of the N₂ gas tank is to purge any wet air from the system that could freeze when cooling and reduce any oxidation that could occur at elevated temperatures. **N₂ gas should always be flowing.**
- For non-spinning experiments the tank pressure should be set between 20 to 30 psi.
- When sample spinning is required increase the pressure to 40 to 50 psi. Higher pressure is required to ensure that the sample is slightly lifted (bearing gas) for frictionless spinning and to regulate spinning speeds.
- To eject samples while at low or high temperatures, first execute the “ej” command then increase the tank pressure to 50 – 60 psi. Higher pressures may be required if using the ceramic spinner or J-Young tubes. If switching samples, replace with your new sample and when it is floating, decrease the pressure back to 20 psi then execute “ij”.



Temperature Control - Topspin

1. To change the sample temperature (except for low temperatures on the NCB 400):

The image shows the Edte software interface with the 'Corrections' tab selected. The 'Sample temp.' is 298.7 K and the 'Target temp.' is 303.1 K. The 'Probe Heater' is off and the 'Cooling' is off. A red box highlights the 'Corrections' tab, and a red arrow points to the 'Activate' button for 'Linear correction [active]'. Another red box highlights the 'Load ...' button. A red arrow points from the 'Load ...' button to a 'File selection ...' dialog box. The dialog box lists several correction files, with '20161013-air-40-50.cor' selected. The 'OK' button is highlighted in the dialog box.

A. In the command line input *edte* to bring up the temperature control window

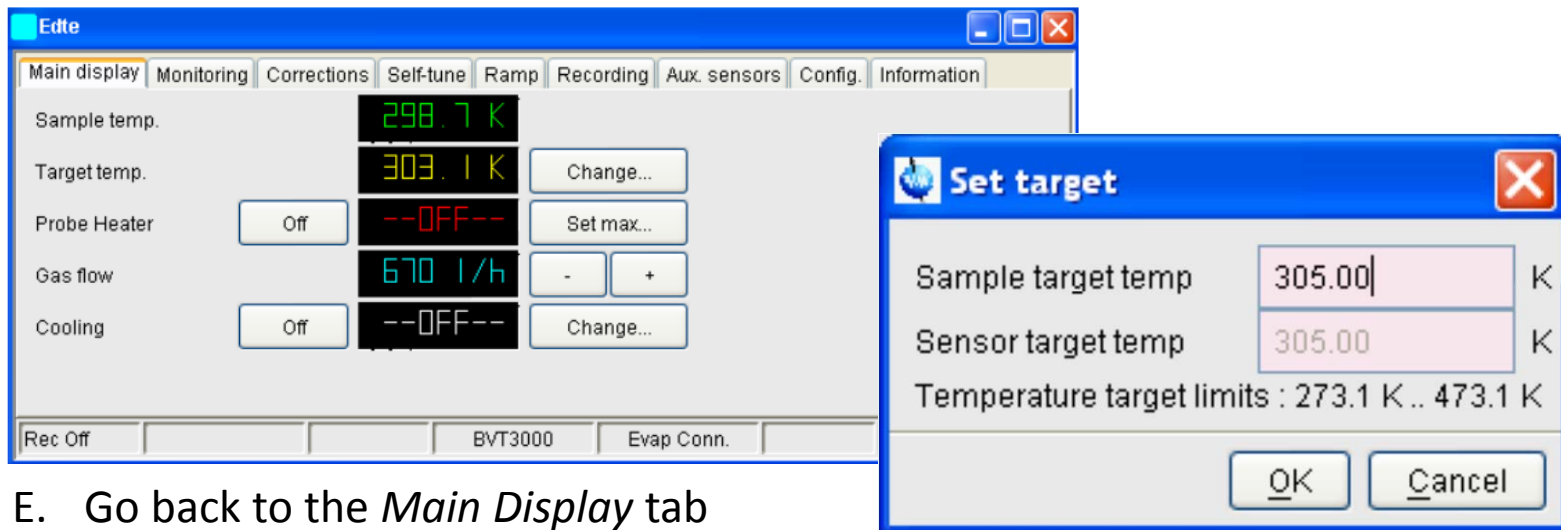
B. Go into the *Corrections* tab and activate Linear Corrections by clicking on the **Activate** button.

C. Click on the **Load** button, select the correct gas/temperature range correction file and press **OK**.

D. Click on the **Apply** button to set the correction file.



Temperature Control - Topspin



- E. Go back to the *Main Display* tab
 - F. Click the **Off** button to turn on the heater, even if you are going to low temperature. It will be used to regulate/stabilize the temperature
 - G. Increase the **Gas flow** to 400 to 500 l/h
 - H. If going to low temperatures, click the **Off** button to turn on the cooler. Click on **Change** and start at a low percentage (ie. 5%). Increase, if necessary, to regulate/stabilize lower temperatures
 - I. Click on the **Change...** button to enter the desired sample temperature in the Sample target temp field.
- * When changing temperatures increase/decrease in small intervals, no more than 25 degrees at a time.
- ** Allow some time for the temperature to stabilize and to heat/cool your sample. Once the target temperature is reached wait an additional 3 to 5 minutes.



Temperature Control – FTS Chiller

- The NCB 400 uses a third party chilling unit that is controlled externally from Topspin.
 - A. In the command line input *edte* to bring up the temperature control window
 - B. Go into the *Corrections* tab and activate Linear Corrections by clicking on the activate button.
 - C. Click on the load button, select the correct FTS correction file and press OK.
 - D. Click on the Apply button to set the correction file.
 - E. In the Main Display Window, increase the Gas flow to 400 to 500 l/h
 - F. On the FTS chilling unit, set the desired temperature using the ▲▼ arrow. There are some preset temperatures with the SP1, SP2, SP3, and SP4 buttons.
 - G. Press the **RUN** button to start cooling.

NOTE: **DO NOT** turn the probe heater on in topspin. The temperature is regulated using the heater with the FTS unit.

