DCIF Training Guide for Variable Temperature Experiments using the Bruker 501

(last update 12Mar2019)

The probe in the Bruker 501 is a Broadband Observe (BBFO) auto-tunable and auto-shimmable probe. The achievable temperature range is -100 °C to 100 °C. This document assumes that the user has been trained to manually acquire spectra using TopSpin on the Bruker 501.

All users must be trained by DCIF before changing the probe temperature!!!

!!! Make sure you use the correct spinner !!!

If you use the incorrect spinner, you risk damaging the probe. Ask a DCIF staff member for the ceramic spinner if you need to use it.

Before you begin:

At what temperature do you want to acquire data? _____

Does your NMR tube have any cracks or chips? _____

What is the boiling and freezing points of your solvent? _____

Stay 25 degrees away from the freezing or boiling point.

How much time you do you need to reserve? _____

See sample calculation below:

Calculate the time needed to change the temperature & return to room 25°C.	25 to 80 °C = 30 min (10°C every 5 minutes) 80º to 25ºC = 30 minutes
How many experiments? What is the acquisitions time?	2 expts @20 min each = 40 min
Equilibration Time per sample	2 samples, 5min = 10min
Total	110 min
Oops Factor (2x First Time)	220 min

Make sure you reserve enough time!

IN AN EMERGENCY:

****PLEASE** make sure that the gas flow to the probe is not turned off or interrupted for any reason.****

If the power fails and/or the gas flow is interrupted, you may notice a burning smell. That is the probe overheating which is a $50,000 piece of equipment. **This is very bad.**

IMMEDIATELY STOP ALL VT ACTIVITIES!

1. Immediately turn heater off (Turn VT off in the **edte** window).
2. Immediately remove the liquid nitrogen heat exchanger (if you are using this).
3. Immediately remove your sample.
4. Immediately contact the NMR Facility staff

Summary of Methodology

1. Calculate how much time you will need and reserve an appropriate amount of time.

2. Lock, tune, and shim.

3. Double click on the Temperature indicator in the status bar or type **edte** to open the Temperature Control Suite.

4. Step (in 10 degree increments) your way down or up in temperature to achieve your desired temperature. Monitor the temperature changes and watch for any problems.

5. Once you reach your target temperature, check the lock, and re-tune and re-shim before you collect your spectrum

6. Once finished, ramp back up or down to 25° C.

7. Clean up after yourself, and return all settings and equipment to their default values, settings and positions.

*** Make sure you are using the correct spinner. ***

Temperature Range	Spinner
0°C to -100°C	Ceramic
0°C to +50°C	Standard POM spinner (blue)
+50°C to 100 °C	Kel-f spinner

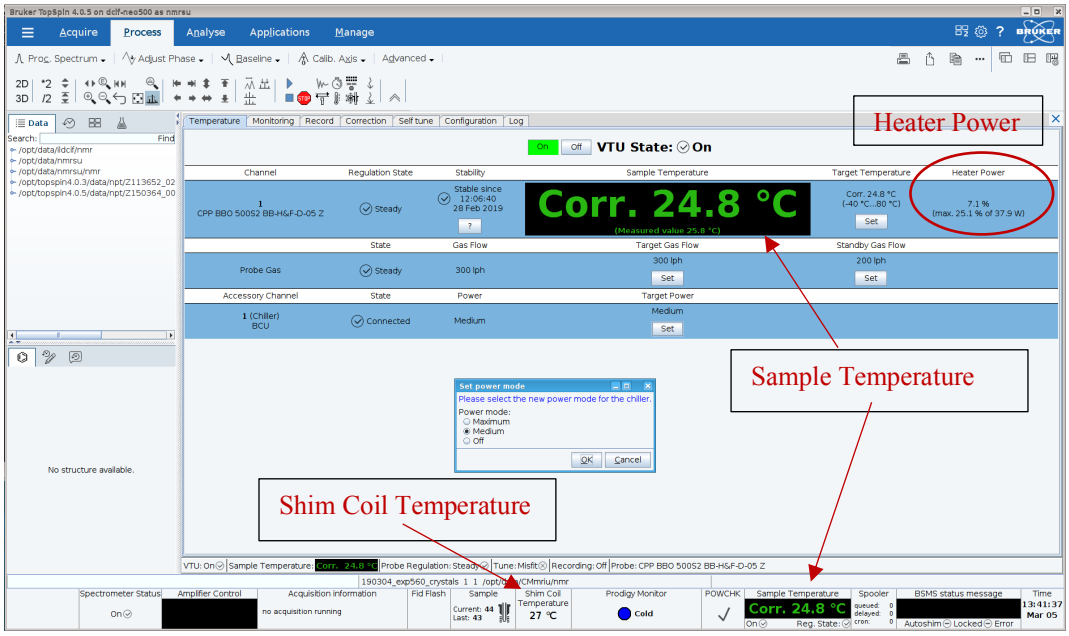
Ask a DCIF staff for the ceramic or kel-f spinner.

RAISING THE TEMPERATURE

Insert your sample. Load parameters. lock, tune, and shim.

DO NOT EXCEED THE -100 to +100 °C RANGE OR PERMANENT DAMAGE TO THE NMR PROBE MAY OCCUR

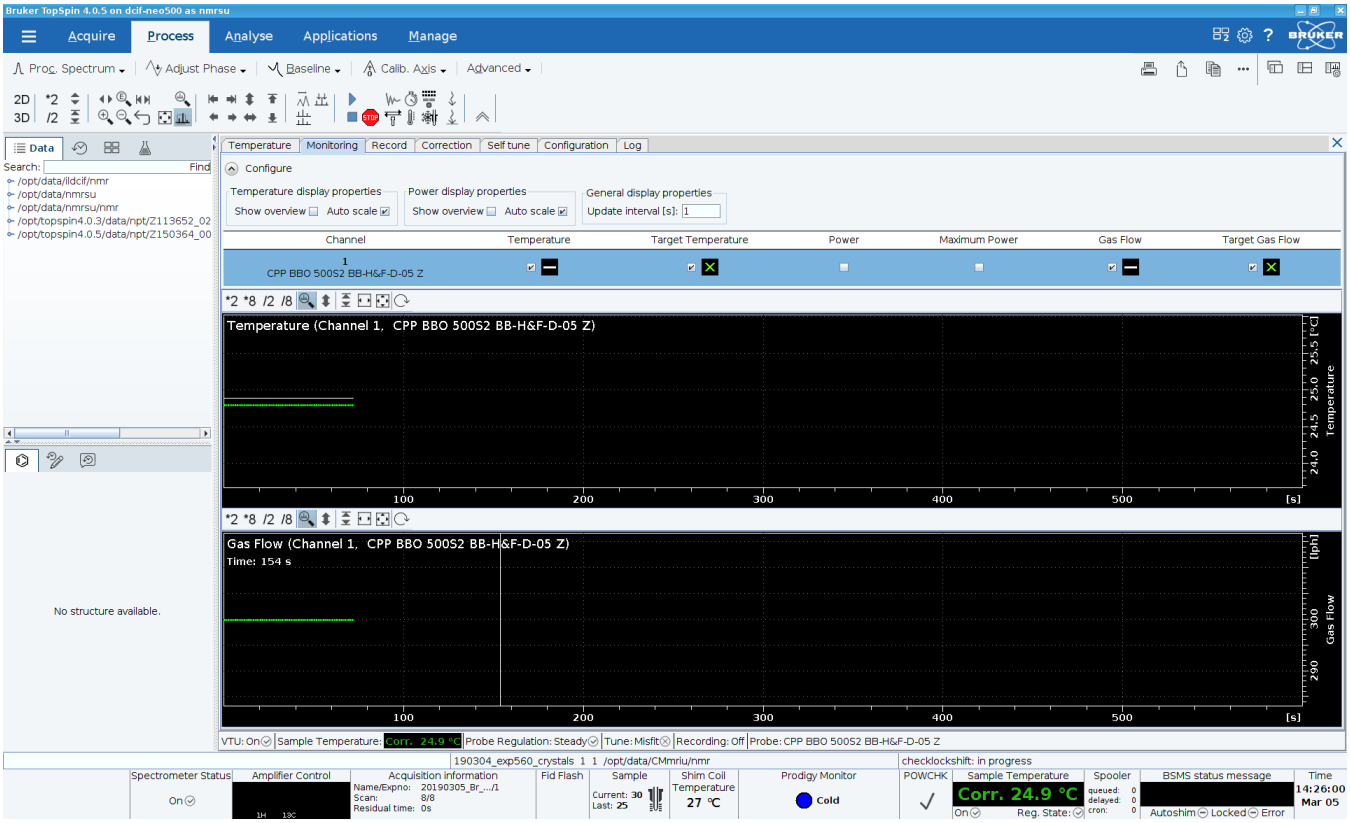
Type **edte** to open the Temperature Control Suite or double click on the sample temperature icon in the status bar. The 501 has an electronic chiller accessory known as the BCU. When heating a sample, click on the button labeled “target power” and set it to off.



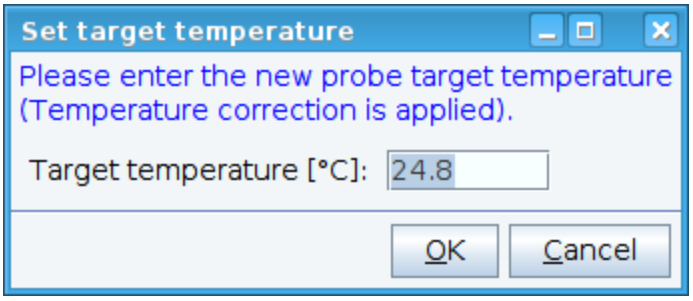
Monitor the **Sample Temperature** and the **Heater Power** near the top right. If the temperature value is **green**, you are at the target temperature (the default is 25ºC). If it is **blue**, the probe is below the target temperature and if it its **red**, it is above the target temperature. You can change the temperature units by right clicking on the Sample Temperature icon in the status bar, selecting Options, and then use the drop-down menu to select the desired temperature units. When heating the sample, the heater power will initially jump to close to maximum power, but then drop off. It should never jump to maximum and stay. If it does, you have tried to heat the sample in too great a step, and you should immediately set the temperature to back to its previous set point.

Monitor the **Shim Coil Temperature.** The shim coil is a package of epoxy and copper flex prints (X and Y shims) and conventional copper coils (Z shims). The epoxy can be damaged if the coil is heated to higher than +100 ºC. The system will shut down to protect itself if shim cooling is not activated (a warning message will be displayed in TopSpin. Notify the DCIF staff IMMEDIATELY if you see this warning message. We will make any necessary adjustments.). Shim Coil Temperature units can be changed in a manner analogous to the probe temperature.

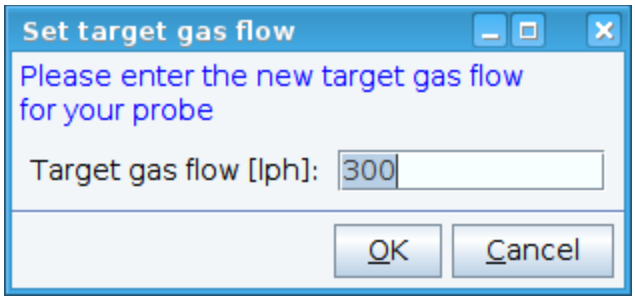
Click on the Monitoring tab. Make sure the Current Temperature, Target Temperature, Target Gas Flow, and Current Gas Flow boxes are all checked. This will allow a graphical representation of how steady the temperature and gas flow are as you heat your sample.



Click back on the Temperature tab in the Temperature Control Suite. To set a new Target Temperature, click on the set button. ***<u>You should only heat in 10 ºC increments.</u>*** Ramping the temperature up too fast can put too much power into the probe heating element which will damage the probe. This is true whether you are heating a cold probe back to room temperature, or heating a room temperature probe above 25 ºC.



Adjust the Target Gas Flow for the Probe Gas based on your target temperature (the default is 400l/h).



Recommended Target Gas Flow Rates

Sample T (in ºC)	**25 to +100 ºC**
Gas Flow Rate (l/h)	400 to 600

Keep in mind these are ranges. You may need to try other values in order to maintain your target temperature. If the temperature stabilizes above or below your setting, adjust the gas flow rate such that the temperature settles on the value you wish. i.e. if you have the temperature set for 50 °C but you are only getting to 45 °C you might need to cut the flow rate back by 50 l/h.

In the Monitoring tab, watch for any signs of trouble. If you see the Current Temperature or Current Power changing erratically, let a DCIF staff member know IMMEDIATELY. **Never leave the NMR unattended while conducting a VT experiment.**

Once the Target Temperature has been reached and the Sample Temperature in the bottom status bar has been **green** for a few minutes, the temperature is stabilized. Check your lock level, retune and reshim the instrument. **All of these adjustments will change with a change in temperature.** You may now acquire your data.

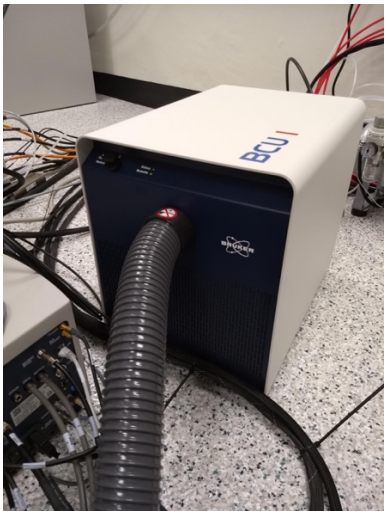
When you are finished, reopen the Temperature Control Suite, set the Target Temperature to 25 ºC, set the Target Gas Flow back to 400 l/h, and wait for the probe temperature to return to 25 ºC before closing the Temperature Control Suite, exiting TopSpin, and logging off the computer.

LOWERING THE TEMPERATURE

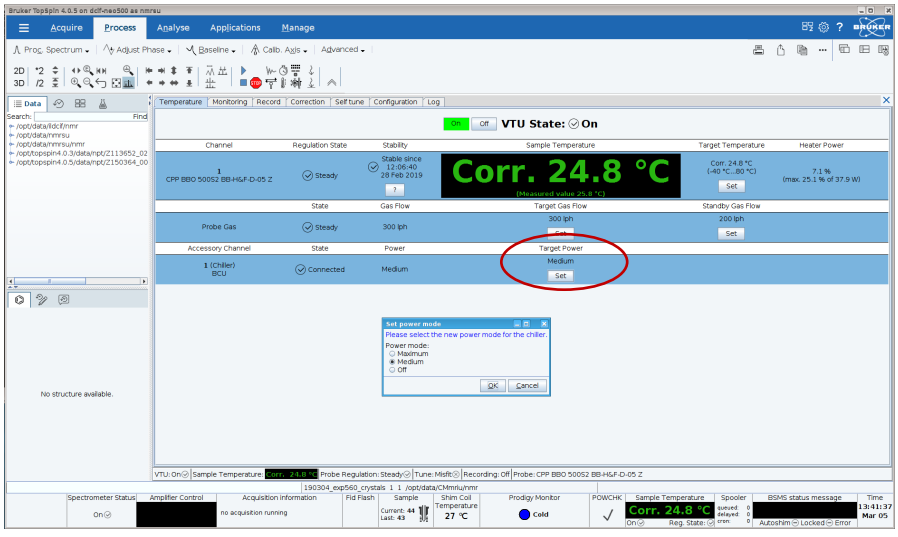
When cooling if your sample can start at room temperature, put it in the magnet, lock, tune, shim and acquire a quick 1D spectrum, so you have a starting point. The 501 has two chilling options. An electronic chiller is present that will cool samples to 0 ºC. If you are going below 0 ºC then the liquid nitrogen heat exchanger will need to be used. *Operation of the temperature control suite in the software is outline above, and is the same for cooling, so it won't be repeated here.*

Using the BCU to adjust the temperature between 0 °C and room temperature.

The BCU is shown below.



To use it click the target power button in the center of the screen and then select "maximum." You may then set the target temperature to any value between 0 and ambient temperature and the system should maintain that temperature. When finished return the system to 25 ºC.



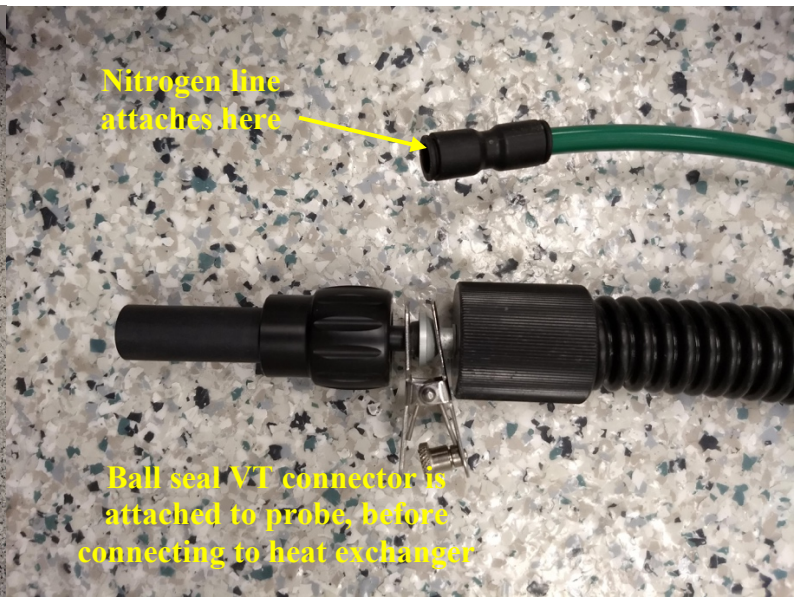
Using the liquid nitrogen heat exchanger

PLEASE NOTE: Users are responsible for providing their own liquid nitrogen. The dewar (26 liters) should be filled so this requires more than carrying down a 4-liter hand held dewar. There is a cart available for transporting the dewar, please ask.

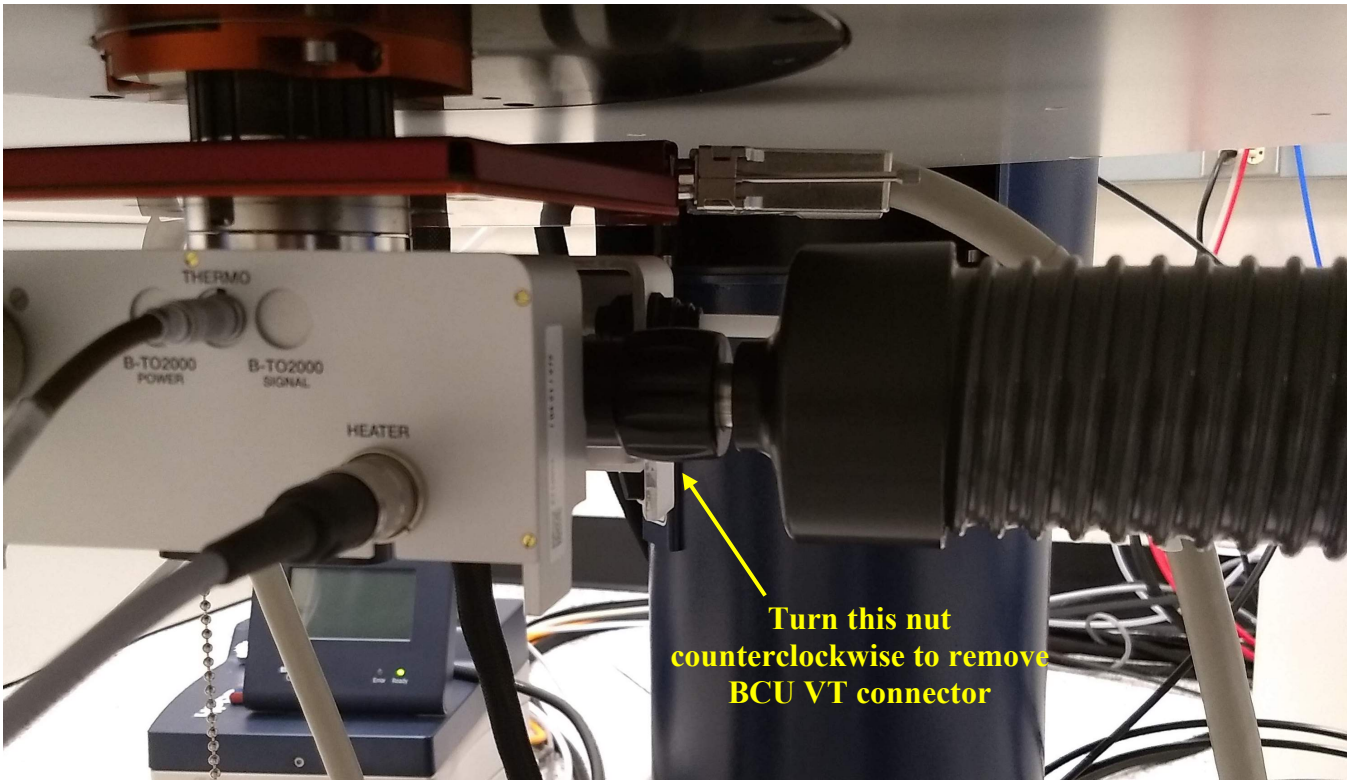
If you are going colder than 0 ºC, you will need to use the liquid nitrogen heat exchanger. This involves interruption of the gas flow to the probe in order to place the heat exchanger in-line and ***should be performed with the greatest of care.*** A quick overview is that the BCU will be disconnected from the probe, the $N_{2(g)}$ supply disconnected from the BCU, a separate VT connector added to the probe, the heat exchanger placed in liquid nitrogen, and then connected to the probe. ***All of these operations must be performed with the VTU state set to off in the Temperature Control Suite. Failure to do so can result in expensive probe damage!!***

The BCU should be turned off, since you will disconnect the gas flow from the back of the BCU to route it through the heat exchanger. Please make sure you fill the nitrogen dewar ***BEFORE*** you begin your VT experiment. The dewar should be full. To route the house nitrogen through the liquid nitrogen heat exchanger, **turn off the VT in the edte window** and disconnect the nitrogen line from the back of the BCU. This is a push/pull connection (highlighted by the yellow arrow below) where you must push the outside of the connection, in while pulling on the tube. It should release without much force, so if you find you have to pull hard, you are not pushing the push connection inwards.

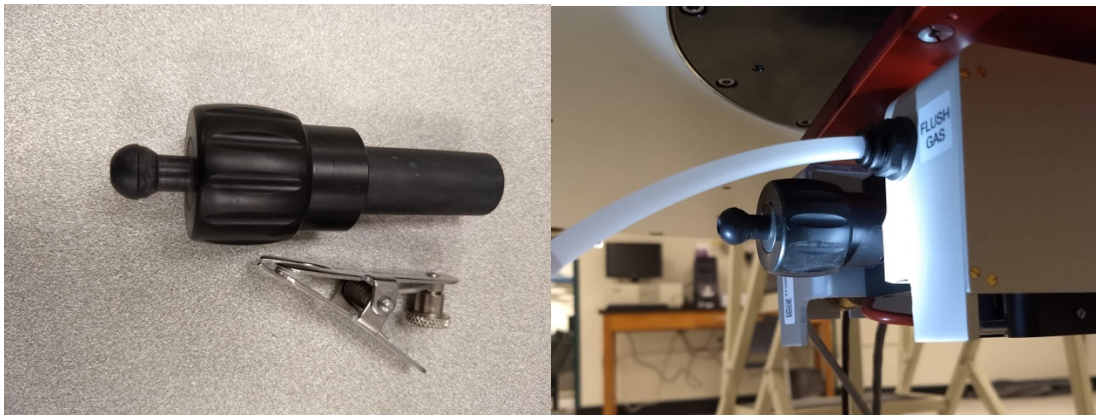
This nitrogen line will then be connected to the green line on the heat exchanger. Note that in the photo below, the ball seal VT connector is shown as attached, *but the attachment should not be made until the ball has been positioned in the probe, and the heat exchanger has been inserted in the liquid nitrogen tank.*



The next thing to do is remove the BCU VT connector from the probe. This is done by unscrewing the Teflon nut indicated in the photo below. Once this is done, the VT connector will slide straight back out of the probe. You can let it hang off to the side.



The ball seal VT connector shown on the left of the picture below will fit into the probe. Tighten the plastic knob gently being sure not to overtighten it.



When this is done, it is now time to immerse the heat exchanger into the liquid nitrogen dewar. There is not much reach on the cooling line, so be sure the dewar is close to the magnet. Also, if you hook the VT line to the ball seal connector on the probe before immersing the heat exchanger, there will not be room to get the heat exchanger into the dewar. ***Slowly and carefully immerse the heat exchanger into the liquid nitrogen. It will splash liquid N2 so be sure you are wearing safety glasses and are using cryo-gloves. Also stand back from the dewar so it does not splash you on the legs.*** Once the heat exchanger is in the dewar the ball seal connector can be attached to the probe. Use the small metal clamp to hold it in place. The direction of the line should be straight as was shown in the heat exchanger picture above. Once this is attached to the probe, return to the computer and turn the VT back on. You may now start going down in temperature.

Set the temperature for 10 ºC and then wait for the temperature to reach this set point. As you are going down in temperature you will want to monitor the **Shim Coil Temperature** (located to the left of the Probe Temperature, in the lower status bar of TopSpin). The Shim Coil Temperature should never go below -52 ºC. TopSpin warns and should remember to activate shim gas when the temperature drops below +5 ºC. If TopSpin does display this warning, please notify the DCIF staff. Shim gas is used to dry (prevent freezing) the whole shim system and to stabilize the falling temperature with the shim coil.

Set your Target Temperature and adjust the Target Gas Flow as necessary. As the temperature setting tends towards -80 ºC you will need to have a higher gas flow in order to

achieve that temperature. The problem is that if you turn the gas flow up too high, it will cause your sample to wobble slightly and eventually push upwards and out of the probe.

Recommended Target Gas Flow Rates

Sample T (in ºC)	**-**100 to 25
Gas Flow Rate (l/h)	400 to 600

Keep in mind these are ranges. You may need to try more than one value in order to maintain your target temperature. In the Monitoring tab, watch for any signs of trouble. If you see the Current Temperature or Current Power changing erratically, let a DCIF staff member know IMMEDIATELY. **Never leave the NMR unattended while conducting a VT experiment.**

Once the Target Temperature has been reached and the Probe Temperature in the bottom status bar has been **green** for 5 minutes, you may switch back to your data set. Check your lock level, retune and re-shim the instrument. You may now collect a spectrum.

When you are finished, step the target temperature back upwards. On the top right of the temperature tab is a section that shows the heater power. You want to watch this and not set the temperature so high that the system is sending a lot of power through the probe to warm it. ***Steps should not be more than 10 ºC at a time***, and if you notice that the power ramps up high, immediately turn the VT off, and adjust the standby gas flow to 400 l/hr. Once the sample temperature has reached 10 ºC, set the VTU state to off. Quickly disconnect the heat exchanger from the probe and remove the ball seal VT connector. Then connect the BCU VT connector back to the probe. Disconnect the $N_{2(g)}$ line from the heat exchanger and return it to the back of the BCU. Go back to the computer and check the probe temperature. If it took you a while to switch the connections, and the probe temperature has dropped again, ***make sure the temperature setting is not more than 20 ºC above the probe temperature***. If it is set the temperature to less than 20 ºC above the probe temperature and set the VTU state to on. Finally set the target temperature to 25 ºC. Set the Target Gas Flow back to 400 l/h and wait for the probe temperature to return to 25 ºC before closing the Temperature Control Suite, exiting TopSpin, and logging off the computer.

*****Please note that the temperatures reported are not the true temperatures. They will generally be within a few degrees of the actual temperature except at the extremes where they can be more than 10 degrees off. The actual temperature is measured by taking an NMR spectrum of a sample of MeOH (cold) or $HOCH_2CH_2OH$ (hot). Ask if you need to have the actual temperature. We have the samples and some calibration data.******

Any questions? Please contact the DCIF staff!

DO NOT attempt anything in this document until AFTER you have been trained by the DCIF staff!

NEVER turn off the gas flow to the probe while the VT is on!!!