DCIF Training Guide
Variable Temperature
Bruker "B400"

(last update 20100421)

As with all of the other DCIF tutorials, the conventions used in this guide are as follows:

- **Boldface** type indicates commands that are typed into the TopSpin input window or
- Italic ***[Boldface]*** type with square brackets indicates a button in the menu that is to be pushed.
- **[Boldface]> [Boldface]** type separated by a bracket indicate selection through a menu.
- **LMB** indicates the Left Mouse Button
- **MMB** indicates the Middle Mouse Button
- **RMB** indicates the Right Mouse Button
- Unless otherwise noted, all commands typed into the input window are followed by an <↵ Enter> keystroke.

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

!!! Use the "high-temp" spinner for temperature >60 °C !!!

Before you begin:

What temperature do you want to measure at?_____
What instrument/probe is appropriate?_____
Ask a DCIF staff member for recommendations.
Does your NMR tube have any cracks or chips?:_____
What is the boiling and freezing points of your solvent?:_____
Stay 5 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 20 °C.	20 to 80 °C = 60 min
Temperature Calibration Time	30 min per point= 30 min
How many experiments? What is the acquisitions time?	2 expts 20 min each= 40 min
Equilibration Time per sample & per standard	2 samples, 20 min = 40min
Total	170 min
Oops Factor (2x First Time)	340 min

IN AN EMERGENCY:

If the power fails, the gas supply is interrupted, you notice a burning smell, etc., IMMEDIATELY STOP ALL VT ACTIVITIES!

1. Immediately turn heater off
2. Immediately remove the liquid nitrogen dewar or FTS chiller.
3. Immediately remove your sample.
4. Immediately contact the NMR Facility staff.

Summary of Methodology

In other words, what you need to do.

1. Calculate how much time you will need and reserve an appropriate amount of time on the Bruker 400 NMR.
2. Lock, tune, and shim.
3. Select the correct carrier gas and set up the FTS, probe heater, and/or nitrogen evaporator as needed.
4. Type **edte** and set up your ramp.
5. Monitor the temperature changes and watch for any problems.
6. Once you reach your target temperature, measure the temperature with the calibrations standards (if needed).
7. Check the lock, re-tune, and re-shim before you collect your spectrum.
8. Once finished, ramp back up or down to 20° C.
9. Make sure the carrier gas is set back to air.
10. Clean up after yourself, and return all settings and equipment to their default.

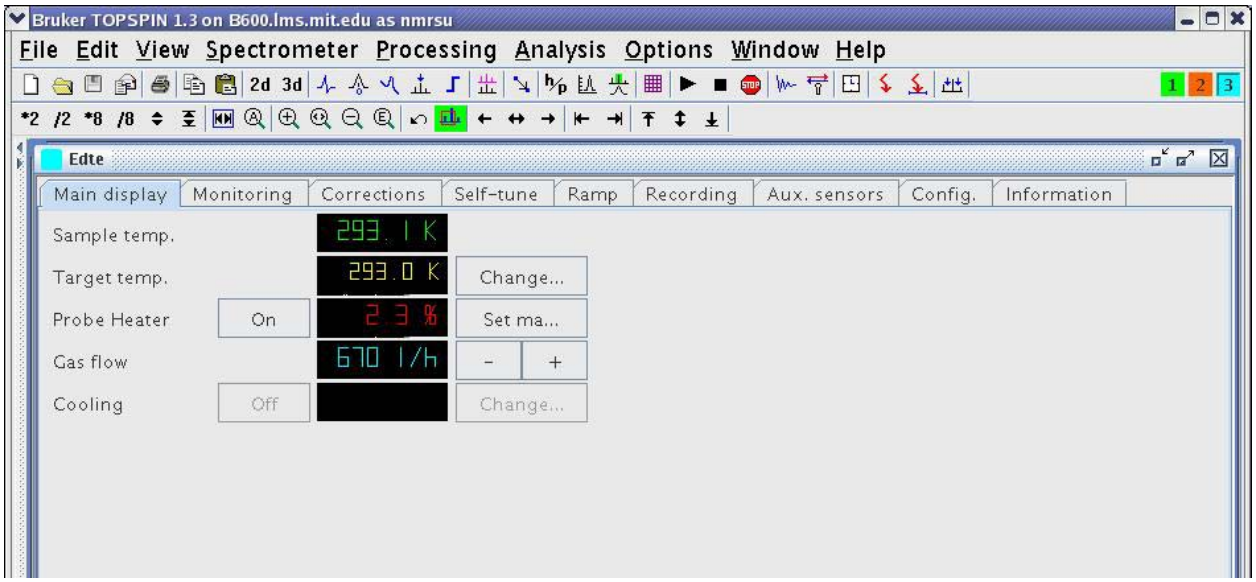
Getting Started:

1. Select the Carrier Gas based on the temperature range and cooling device.
   To select nitrogen:
   a. Turn off the compressed air (may keep 401 magnet legs on compressed air). The valve is closed when the handle is perpendicular to the pipe.
   b. Turn on the nitrogen.

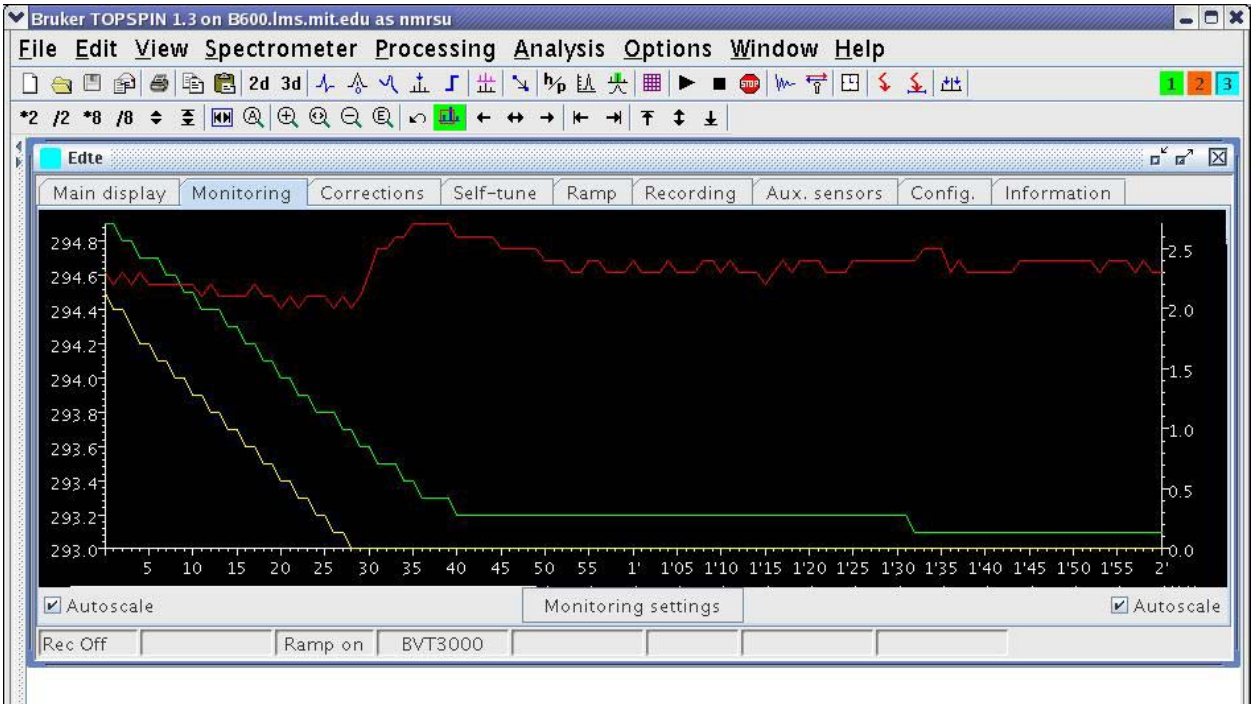
Temp Range	Cooling Device	Gas
50 to 100 °C	None	Nitrogen
20 to 50 °C	None	Air
-30 to 40 °C	FTS Preconditioner or Liquid Nitrogen Dewar	Air
-150 to 20 °C	Liquid Nitrogen Dewar	Nitrogen

DO NOT EXCEED THE -150 to +100 °C RANGE WITHOUT TALKING TO THE DCIF STAFF

2. In TopSpin type **edte** to open the temperature controller
   a. Select: ***[Corrections]*** **"**No corrections [active]" should be displayed.
   b. Select: ***[Ramp]*** ramp rate of **2** degrees/min should be displayed.
   c. Normal Conditions:
      i. Gas: air
      ii. Sample Temp= 20 °C Thermocouple located below tube.
      iii. Target Temp= 20 °C
      iv. Heater= OFF (Set Max = 10%)
      v. Gas Flow= 270 L/h
      vi. Cooling= Empty



d. Within the edte window Open ***[Monitoring]*** to view Temperature and Output Power
    i. Use auto scale for both y-axis':
        Left: Temperature
        Right: Heater Power
    ii. At equilibrium, heater power fluxuations should be lower than 0.1 %
    iii. Use the graph to monitor temperature changes.
        Red line: probe heater
        Yellow line: target temperature
        Green line: sample temperature



3. Temperature Accuracy:
    a. The temperature inside the NMR tube will reach equilibrium time 5-20 min after the sample (probe) temperature equilibrates.
    b. Accurate temperature measurements should be made with calibration standards: Ethylene glycol for high temperature. Methanol for low temperature. Review the temperature ranges on the tables below.
    c. As the calibration standard equilibrates, measure a single 1H spectrum: Turn the sweep off **bsmsdisp >[Lock]>[Sweep]**. Load (**rpar**) proton parameters, **ns** = 1 **ds** = 0, **rga zg** and measure the distance between the two peaks to calculate the temperature inside the tube. See equations below.
    d. Repeat the measurement every 3-4 minutes to determine the equilibration rate. NMR samples of similar size should equilibrate at the same rate; if viscosity is high increase the equilibration time.

e. If temperature stability is critical, measure the temperature with a standard before and after your experiment.
f. When working over a large temperature range, create a calibration curve of Measured Temperature vs. Sample Temperature.

4. High Temperature: Ethylene Glycol(neat) (Accuracy & Range 273–416 K)
   Reference: Amman, Meier, Merbach, *J. Mag. Reson.* **1982**, 46, 319-321
   1. Equation: $T(K) = 466.5 - (\Delta \text{ (ppm)} \times 102.00)$

Temp deg C	Temp Kelvin	Δ ppm
92	365	1.0
81	354	1.1
71	344	1.2
61	334	1.3
51	324	1.4
41	314	1.5
30	303	1.6
20	293	1.7
10	283	1.8
0	273	1.9

5. Low Temperature Methanol (neat) (Accuracy & Range 178-330 K)
   Reference: Amman, Meier, Merbach, *J. Mag. Reson.* **1982**, 46, 319-321
   Equation: $T(K) = -23.832\ \Delta^2 - 29.46\ \Delta + 403.3$

Temp deg C	**Temp deg K**	**Δ ppm**
56	329	1.25
47	320	1.35
37	310	1.45
27	300	1.55
16	289	1.65
5	278	1.75
-6	267	1.85
-18	255	1.95
-31	242	2.05
-44	229	2.15
-57	216	2.25
-71	202	2.35
-85	188	2.45
-99	174	2.55

Raising the Temperature

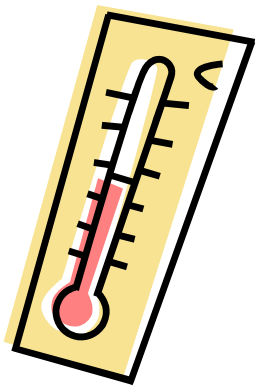
In the edte window,

1. Increase the gas flow to 1070 L/h ***[+] / [-]*** and turn the probe heater **[ON]**.
2. Select ***[Change]*** to modify the target temperature, enter the desired temperature. Select ***[OK]***. The software will increment the Target Temperature to the desired temperature according to the ramp rate.
3. View the target temperature, heater output, and sample temperature in the ***[Monitoring]*** window. At equilibrium, the Sample (probe) Temperature should be steady and the heater fluxuations should be < 0.1%. If the probe does not reach the desired temperature, increasing the gas flow will help cool the probe, increasing the heater power will boost the warming capacity. Adjust the desired sample temperature using the calibration standards.
4. Run ***[Self Tune]>[Start Self Tune]*** wait until self tune is finished. Rerun self tune if the temperature is changed by greater than 10 K.
5. After the NMR tube has equilibrated, shim the magnet and tune the probe

When Finished

1. Ramp back to 20° C.
2. Turn the heater off.
3. Set the gas flow to 270 L/h.
4. Return the gas supply to air, if necessary.

Any questions? Please contact the DCIF staff!



Lowering the Temperature

FTS unit

Normal Conditions (no VT):

Preconditoner set to 20°C.
Probe heater OFF.
Preconditioner range -30°C to 40°C.
High temperature alarm at 50°C.
Low temperature alarm at -40°C.

1. Connect the rubber air line, the black controller cable and the two blue connectors to the back of the AIR-JET unit. The blue connectors are labeled **1** and **2** in marker, so be sure to plug them into the correct sockets.
2. Open the door to the air dryer and turn it on. Adjust the upper gauge to 45 psi and the lower meter to 2 psi.
3. Turn on the AIR-JET unit and the FTS SYSTEMS (preconditoner) box.
4. Press the **SP2** button on the FTS SYSTEMS. This should set the temperature to 20°C.
5. In the **edte** interface, set the gas flow at 1070 L/h and turn **[ON]** the probe heater.
6. Disconnect the green gas supply line from the probe; be careful, the clip is attracted to the magnet.
7. Connect the large black hose that runs from the AIR JET to the probe.
8. Set the FTS SYSTEMS (preconditoner) to 10° below the target temperature. **DO NOT EXCEED THE RANGE!**
9. In the edte window, select ***[Change]*** Target Temperature, enter the desired temperature. Select ***[OK]***. The software will increment the Target Temperature to the desired temperature according to the ramp rate. At equilibrium, the Sample (probe temperature) should be steady and the heater fluxuations should be < 0.1%. If the probe does not reach the desired temperature, increasing the gas flow will help cool the probe, increasing the heater power will boost the warming capacity. Adjust the desired sample temperature using the calibration standards.
10. Run [Self Tune]>[Start Self Tune] wait until self tune is finished. Rerun self tune if the temperature is changed by greater than 10 K.
11. After the NMR tube has equilibrated, shim the magnet and tune the probe.

When Finished

1. Ramp back to 20° C, let the probe equilibrate for 10 min.
2. Turn the heater off.
3. Disconnect the large black air line from the probe.
4. Reconnect the ball joint to the gas supply line.
5. Set the gas flow to 270 L/h.

6. Return the gas supply to air, if necessary.
7. Turn off the air dryer, the AIR-JET unit, and the FTS SYSTEMS (preconditoner).
8. Disconnect the rubber air line, the black controller cable and the two blue connectors from the back of the AIR-JET unit.

Nitrogen Evaporator

1. Set the gas flow at 1070 L/h.
2. Turn the heater off.
3. Disconnect the green gas supply line from the probe; be careful, the clip is attracted to the magnet.
4. Disconnect the ball joint on the supply line at the black union.
5. Connect the gas supply line to the nitrogen cold finger. Check for flow and listen for any water trapped in the cold finger. If water is detected, increase the gas flow to 2000 L/h and dry out the cold finger.
6. Fill the nitrogen Dewar half full of liquid nitrogen
   {Researchers are responsible for providing their own cryogens}
7. Slowly insert the cold finger, into the dewar and check for gas flow. If no flow is detected, remove the cold finger and thaw the ice blockage.
8. Turn the gas flow to 0/missing.
9. Connect the black cold finger tube to the probe.
10. Turn the gas flow to 1070 L/h.
11. Turn the heater on.
12. In the edte window, select ***[Change]*** Target Temperature, enter the desired temperature. Select ***[OK]***. The software will increment the Target Temperature to the desired temperature according to the ramp rate. At equilibrium, the Sample (probe temperature) should be steady and the heater fluxuations should be < 0.1%. If the probe does not reach the desired temperature, increasing the gas flow will help cool the probe, increasing the heater power will boost the warming capacity. Adjust the desired sample temperature using the calibration standards.
13. Run [Self Tune]>[Start Self Tune] wait until self tune is finished. Rerun self tune if the temperature is changed by greater than 10 K.
14. After the NMR tube has equilibrated, shim the magnet and tune the probe.

When Finished

1. Ramp back to 20° C, let the probe equilibrate for 10 min
2. Turn the heater off.
3. Turn the gas flow to 0/missing.
4. Disconnect the cold finger from the probe
5. Reconnect the ball joint to the gas supply line.
6. Set the gas flow to 270 L/h.
7. Set the probe gas to air.

Any questions? Please contact the DCIF staff!

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