The probe in both the Bruker 400 and Bruker 401 is a BBO auto-tunable and auto-shimmable probe. The manufacturer’s specifications state that the probe’s temperature range is +150ºC to -150ºC. HOWEVER, extreme caution must be used when approaching these temperature limits. This document assumes that the user understands how to collect a basic 1D spectrum on the Bruker 400 and 401 NMRs in the DCIF.

*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 or Kel-F spinner if you need to use it.

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 do you need to reserve?: ________________________________
Make sure you reserve enough time!

The probe must be returned to 25°C by the end of your reserved time. NO EXCEPTIONS!

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. 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 evaporator (if you are using this).
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 NMR.
2. Lock, tune, and shim.
3. Select the correct carrier gas (see chart on next page).
4. Type `edte` to open the Temperature Control Suite.
5. Monitor the temperature changes (10° increments) and watch for any problems.
6. Once you reach your target temperature, check the lock, and re-tune and re-shim before you collect your spectrum.
7. Once finished, ramp back up or down to 25°C (10° increments).
8. Make sure the carrier gas is set back to air.
9. 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. ***

<table>
<thead>
<tr>
<th>Temperature Range</th>
<th>Spinner</th>
</tr>
</thead>
<tbody>
<tr>
<td>+120°C to +180°C</td>
<td>Ceramic</td>
</tr>
<tr>
<td>0°C to -150°C</td>
<td>Ceramic</td>
</tr>
<tr>
<td>0°C to +80°C</td>
<td>Standard POM spinner (blue)</td>
</tr>
<tr>
<td>+80°C to +120°C</td>
<td>Kel-F</td>
</tr>
</tbody>
</table>

Ask a DCIF staff for the Ceramic and/or Kel-F spinner.
RAISING THE TEMPERATURE

1. Insert your sample. Load parameters. Lock, tune, and shim.

2. Select the carrier gas based on the temperature range. For the 400, the gas valves are located on the wall directly above the spectrometer computer. For the 401, the gas values are located behind the 401 magnet and to the right of the Varian 500 computer. Consult the posted diagram for the correct valve configuration.

<table>
<thead>
<tr>
<th>Temperature Range</th>
<th>Cooling Device</th>
<th>Gas</th>
</tr>
</thead>
<tbody>
<tr>
<td>+50 to +100 °C</td>
<td>None</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>+20 to +50 °C</td>
<td>None</td>
<td>Air</td>
</tr>
</tbody>
</table>

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

3. Type edte to open the Temperature Control Suite.
4. You will want to keep a close watch on the **Probe Temperature** box located in the lower status bar of TopSpin. If the value is **green**, you are at the target temperature (the default is 25°C). If it is **blue**, the probe is below the target temperature and if it its **red**, it is above the target temperature. If you want this value to read Kelvin, Celsius or Fahrenheit, click on the **Configuration** tab in the Temperature Control Suite, and under General Configuration, select the Temperature unit you desire. You can also change the temperature units by right clicking on the Probe Temperature, selecting Options, and then using the drop down menu, selecting the desired temperature units.

5. You also want to watch the **Shim Coil Temperature** (located to the left of the Probe Temperature, in the lower status bar of TopSpin). 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 +80 °C. 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.).

If you want to change the units that Shim Coil Temperature is displayed in, right click on the Shim Coil Temperature, select set shim coil temperature units, and then using the drop down menu, select the desired temperature units.
7. Click back on the Temperature tab in the Temperature Control Suite. To set a new Target Temperature, click on the set button. Remember you are to increase your temperature in 10°C increments. Wait for the temperature to be stable (Probe Temperature green for 5 minutes) before going up the next 10°C increment.

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

<table>
<thead>
<tr>
<th>Sample T (in °C)</th>
<th>25 to +80 ºC</th>
<th>+80 to +120 ºC</th>
<th>+120 to +150 ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Flow Rate (l/h)</td>
<td>400 to 600</td>
<td>535 to 670</td>
<td>535 to 800</td>
</tr>
</tbody>
</table>

Keep in mind these are ranges. You may need to try more than one value in order to maintain your target temperature.

9. 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.**

10. Once the Target Temperature has been reached and the Probe Temperature in the bottom status bar has been green and stable for 10 minutes, close the Temperature Control Suite by clicking the X in the upper right corner of the Temperature Control Suite window.

11. Check your lock level. Retune and reshim the instrument. You may now collect a spectrum.

12. When you are finished, reopen the Temperature Control Suite, decrease your temperature in 10°C increments. The system should be left at 25°C and the Target Gas Flow set to 400 l/h for the next user. Close the Temperature Control Suite, exit TopSpin, and log off the computer.
LOWERING THE TEMPERATURE

1. PLEASE NOTE: Users are responsible for providing their own liquid nitrogen. Please make sure you fill the nitrogen Dewar BEFORE you begin your VT experiment. The Dewar should be full.

Liquid Nitrogen Dewar and Heat Exchanger
2. Connect the LN2 cable (runs from the heat exchanger) to the cable that runs from the back of the 400 and 401 consoles (Lower right side, VT Aux 1). This cable monitors the nitrogen level in the Dewar. It does NOT control temperature in any way. If it is already plugged in, leave it plugged in.

3. Insert your sample. Load parameters. Lock, tune, and shim.

4. Select the carrier gas based on the temperature range. For the 400, the gas valves are located on the wall directly above the spectrometer computer. For the 401, the gas valves are located behind the 401 magnet and to the right of the Varian 500 computer. Consult the posted diagram for the correct valve configuration.

<table>
<thead>
<tr>
<th>Temperature Range</th>
<th>Cooling Device</th>
<th>Gas</th>
</tr>
</thead>
<tbody>
<tr>
<td>-150 to +25 °C</td>
<td>Liquid Nitrogen Dewar</td>
<td>Nitrogen</td>
</tr>
</tbody>
</table>


5. Type `edte` to open the Temperature Control Suite.

6. You will want to keep a close watch on the **Probe Temperature** box located in the lower status bar of TopSpin. If the 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 is **red**, it is above the target temperature.

If you want this value to read Kelvin, Celsius or Fahrenheit, click on the **Configuration** tab in the Temperature Control Suite, and under General Configuration, select the Temperature unit you desire. You can also change the temperature units by right clicking on the Probe Temperature, selecting Options, and then using the drop down menu, selecting the desired temperature units.
7. You also want to watch 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 °C. TopSpin warns and should remember to activate shim gas when the temperature drops below +5 °C. 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. The shim coil can safely get down to 193K (-80°C), but it not recommended.

If you want to change the units that Shim Coil Temperature is displayed in, right click on the Shim Coil Temperature, select set shim coil temperature units, and then using the drop down menu, select the desired temperature units.
9. Turn off the VT in edte. This will turn off the probe heater, and it’s now safe to unhook the air lines to the probe.

10. It helps to have two people for the next several steps, one working by the instrument, the other at the computer. Insert the heat exchanger into the liquid nitrogen Dewar. Connect the tube with the ball joint to the probe. Be careful, the clip is magnetic! Connect the black air line from the heat exchanger to the air line. Undo the "double female" connector and attach the heat exchanger's air line to the air line that runs to the back of the console. This connector is currently black plastic on the 400 and tan/gray plastic on the 401.
11. As soon as the heat exchanger is in line, turn the probe heater back on in edte. You do not want to have the cold air running into the probe without the probe heater on, as this will cause the probe temperature to plummet very fast. Set your **target temperature for 10 ºC and the gas flow for 200-250 l/h.** WATCH TO MAKE SURE THE PROBE TEMPERATURE DOES NOT DROP DANGEROUSLY FAST.

12. Set your Target Temperature (10ºC increments, with 5 minutes of stability in between each increment) and adjust the Target Gas Flow as necessary. You may have to increase the gas flow from 200 l/h to help the instrument get down to temperature. **DO NOT CRANK UP THE GAS FLOW TO MAKE THINGS GO FASTER.** Be patient!

### Recommended Target Gas Flow Rates

<table>
<thead>
<tr>
<th>Sample T (in ºC)</th>
<th>-150 to -80 ºC</th>
<th>-80 to 0 ºC</th>
<th>0 to +25 ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Flow Rate (l/h)</td>
<td>300 to 600</td>
<td>300 to 450</td>
<td>200 to 300</td>
</tr>
</tbody>
</table>

Keep in mind these are ranges. You may need to try more than one value in order to maintain your target temperature.

13. 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.**

14. Once the Target Temperature has been reached and the Probe Temperature in the bottom status bar has been **green** and stable for 10 minutes, close the Temperature Control Suite by clicking the X in the upper right corner of the Temperature Control Suite window.

15. Check your lock level. Retune and reshim the instrument. You may now collect a spectrum.

16. When you are finished, bring the probe back up to 0ºC (adjust gas flow rate as needed) in 10º increments. Once the sample temperature has reached 0ºC, pull the heat exchanger out of the Dewar and set it on the floor. **DO NOT TURN OFF THE GAS FLOW OR DISCONNECT THE AIR LINES FROM THE PROBE.** The lines from the heat exchanger are short, you may need to tip the Dewar a bit to help you pull the heat exchanger out. It helps to have two people do this. **Do not yank on or twist the gas lines!**
17. Set the target temperature to 10 °C and allow it to come up to about 15 °C.

18. Turn off the probe heater (turn off the VT in the edte) and return all air lines to their default position. Disconnect the heat exchanger from the probe. Select the correct carrier gas (valves on the wall) – that means air for room temperature.

19. Turn the probe heater back on (turn on the VT in the edte) and set the Target Temperature to 25ºC. Set the Target Gas Flow back to 400 l/h and wait for the probe temperature to return to 25ºC before closing the Temperature Control Suite, exiting TopSpin, and logging off the computer.

**NOTE:** If the VT needs to be reset, you can do this by closing edte and then TopSpin. On the floor by the magnet should be a small tan TC-T2 box. Disconnect the cable from the TC-T2 box, reconnect it, open TopSpin, and type `edte`. NEVER TURN OFF THE GAS FLOW TO THE PROBE.

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!!!*