Instructions for Variable Temperature (VT) Operation

Variable temperature NMR is an excellent way to investigate dynamic behavior of molecules. Both qualitative and quantitative information about inversion, ring-flip, and other barriers can be extracted from a series of spectra at different temperatures.
VT operation is available on many of our spectrometers. We have set limits of 0 to 50 °C for most spectrometers. These temperatures were selected to protect the probes from damage due to either a solvent freezing and cracking the NMR tube (D_20 at 0 °C) or a solvent boiling and spilling in the probe ($CDCl_3$ at 62 °C). Both could cause significant probe damage. This has occurred, not because of the user that did the VT experiment, but because of the next user who was unaware of the probe temperature.

IMPORTANT: BE SURE THAT YOU DO NOT GO ABOVE THE BOILING POINT OR BELOW THE FREEZING POINT OF YOUR SOLVENT. IF YOU REQUIRE TEMPERATURES OUTSIDE THESE RANGES, PLEASE CONTACT US (386, 387, OR 792).
YOU MUST ALLOW AMPLE TIME FOR THE PROBE TO RETURN TO ROOM TEMPERATURE (UP TO 15 MINUTES) PRIOR TO THE NEXT USER'S TIME. FAILURE TO DO SO MAY LEAD TO PROBE DAMAGE.

Temperature Calibration

The temperature that is displayed on the instrument console and on the VNMR screen display is not necessarily the actual temperature. Therefore, it is important to calibrate the temperature. This calibration can be done a week in advance of the experiment or a week after, but it is best done on the day of the experiment. If you will be using a series of temperatures, you should obtain a calibration curve.

1. Lock and shim on your sample or on a standard.
2. Eject the sample and insert the ethylene glycol (for High temperature) or methanol (for Low temperature) sample. The sample is intentionally short in order to minimize diffusion effects; therefore, be sure to gauge the sample properly. Since there is no deuterated solvent in the sample, you will not be able to lock or shim. It doesn't matter. You will still be able to acquire the spectrum.
3. Type *temp*. A window will pop up. Drag the slider to the desired temperature.
4. Allow the probe and sample to equilibrate for at least 5 minutes.
5. Click **Setup** => **H1,CDCl3**.
6. Type *nt=1 ga*.
7. Type *aph*. Place the cursors on top of the 2 peaks.
8. Type *tempcal('e')* for ethylene glycol (high temp) or type *tempcal('m')* for methanol (low temp).
9. Repeat for the entire temperature range you require.
10. Plot the actual temperature vs. set temperature. The slope of this line will be the calibration factor.

Step-by-Step Instructions for VXR-500 (Anubis: B-7 subbasement)

- Choose a deuterated solvent that is a liquid in the temperature range you require.
- Eject the standard sample and insert your sample.

For high temperature operation:

1. Type *vttype=2 su*. After setup is complete, type *aa*.
2. Type *temp* and drag the slider in the pop-up window to the desired temperature.
3. Allow temperature to equilibrate for at least 5 minutes.
4. Shim and acquire spectrum as usual.
5. When completed, type *temp* and set the temperature to 25.

IMPORTANT: RESET THE TEMPERATURE AT LEAST 10 MINUTES PRIOR TO THE NEXT USER'S TIME SLOT.

For low temperature operation:

1. Type *temp*. Drag the slider in the pop-up window to the desired temperature.
2. Go to the chiller located against the wall behind the magnet (when looking from the computer. It is the west wall).
3. The setting should be 15.0. Using the down arrow key, set the temperature to 10 degrees below your desired temperature. For example, if I typed *temp=0 su*, I would set the chiller to -10.
4. The temperature should change rapidly. If there is little or no temperature change in 2 minutes, contact the NMR staff (792).
5. Allow temperature to equilibrate for at least 5 minutes.
6. Shim and acquire spectrum as usual.
7. When completed, type *temp* and set the temperature to 25.
8. Reset the chiller to 15.

IMPORTANT: RESET THE TEMPERATURE AT LEAST 10 MINUTES PRIOR TO THE NEXT USER'S TIME SLOT.

Step-by-Step Instructions for Other instruments

For high temperature operation:

1. Type *vttype=2 su*. After setup is complete, type *aa*.
2. Type *temp* and drag the slider in the pop-up window to the desired temperature.
3. Allow temperature to equilibrate for at least 5 minutes.
4. Shim and acquire spectrum as usual.
5. When completed, type *temp* and set the temperature to 25.

IMPORTANT: RESET THE TEMPERATURE AT LEAST 10 MINUTES PRIOR TO THE NEXT USER'S TIME SLOT.

For low temperature operation: WE HIGHLY RECOMMEND YOU CONTACT US (792) PRIOR TO DOING LOW TEMPERATURE WORK ON ANY INSTRUMENT OTHER THAN THE VXR-500.

1. Type *vttype=2 su*. After setup is complete, type *aa*.
2. Type *temp* and drag the slider in the pop-up window to the desired temperature.
3. Remove the Styrofoam bucket located on the leg of the magnet by twisting.
4. Fill the bucket with liquid nitrogen and CAREFULLY place back around VT cooling coil.
5. Allow temperature to equilibrate for at least 10 minutes.
6. Calibrate if desired.
7. Shim and acquire spectrum as usual.
8. When completed, remove the cooling bucket, type type *temp* and set the temperature to 25. Wait until the temperature has equilibrated to 25 °C.

IMPORTANT: RESET THE TEMPERATURE AT LEAST 10 MINUTES PRIOR TO THE NEXT USER'S TIME SLOT.