Step By Step Instructions for VT Experiments on the Bruker 300 MHz Spectrometer

(1) Use the drop down menu to create a new experiment/dataset for acquiring the room temperature 1D spectrum. The name of this data set should be used for the rest of the VT experiments on the same sample for a specific nucleus. Be sure to set the experiment number to 1 and clearly indicate in the title that this spectrum was acquired at room temperature (22°C).

(2) Go through the normal steps for acquiring the 1D spectrum as outlined in the 1D training manual.

(3) Ensure that the shims at room temperature are sufficient for the resolution you would like in your VT spectra. If you are unhappy with the shims you can either manually shim or go through another auto-shim process. NOTE: You will need to check the shim at each temperature and shim when required. A good starting room temperature shim ensures that this process is much easier. SAVE YOUR ROOM TEMP SHIMS: Once you are happy with your shims you must save your room temperature shims. This can be done by typing "wsh RoomTemp-VT" into the command line and hitting enter. You will use this file to reload the initial room temperature shims in step 11 below.

(4) Reference your spectra and then write down the value for the SR parameter.

Referencing can be done by clicking on the calibrate spectrum option in the NMR step by step drop down menu. The SR parameter can be found in the ProcPars tab or by simply typing SR in the command line and pressing enter. You will need this number later when referencing your VT spectra at various temperatures. NOTE: this number should be recorded for your ROOM TEMPERATURE spectrum, as the shift of the reference line is also temperature dependant and will change at each temperature.

(5) Create a new data set for each temperature you will be working with. These new data sets should use the same experiment name and the current parameters but a different experiment number for each temperature. Clearly indicate the temperature in the title of each experiment.

(6) Open the VT interface by typing edte in the command line and hitting enter. Please be patient and wait while the VT interface opens. The default settings should be the following:

Target temp = 22ºC

Probe heater = ON

Max power = 5%

Gas flow = 535 L/h

Cooling = empty and off

Be sure that when you finish your VT experiments these parameters are set back to the default values shown above. If you do not return the parameters to these values, you could lose your VT privileges. If these values are not at the default setting when you open the VT interface, please inform the facility manager immediately.

(7) You are now ready to setup the VT hardware for your experiment. **YOU MUST** carefully follow the policies and procedures for VT use of the instrument **AT ALL TIMES**. The following three sections provide instructions for setting up the instrument to work properly inside the three VT ranges of the spectrometer:

(A) Normal and High Temperature Operational Range: +20 ºC to +150 ºC

(a) For this VT range no setup is required and your experiments can be run with the console in its normal operation mode. Be sure to check both the air regulator on the wall and the console regulator to ensure that they read 90psi and >5bar, respectively. If they do not read these values STOP and contact the facility manager immediately.

(b) Make sure to know the boiling point of your solvent. **DO NOT** exceed the boiling point of your solvent. Doing so can cause severe damage to the probe head!

(B) Low Temperature Range: 20 ºC to -55 ºC

(a) You must check both the air regulator on the wall and the console regulator to ensure that they read 90psi and >5bar, respectively. If they do not read these values STOP and contact the facility manager immediately.

(b) When using the low temperature range you should make sure to know the melting point of your solvent. **DO NOT** go lower than 10 degrees from the melting point of your solvent as doing so can cause severe damage to the probe head!

(c) The mass-multiplier ring should be used for all low temperature experiments.

(d) Go to the back of the BCU II unit and change the dial setting on the unit from “flush” to the “3” setting.

(C) Ultra Low Temperature Range: -55 ºC to -150 ºC

(a) Fill the LN2 holding dewar.

(b) You must check both the air regulator on the wall and the console regulator to ensure that they read 90psi and >5bar, respectively. If they do not read these values STOP and contact the facility manager immediately.

(c) When using the ultra-low temperature range, you should make sure to know the Melting point of your solvent. **DO NOT** go lower than 10 degrees from the melting point of your solvent as doing so can cause severe damage to the probe head!

(d) turn off gas flow

(e) remove BCU trunk and velcro

(f) Place the LN2 dewar in the circle outlined on the floor behind the magnet.

(g) The o-ring and the ring clamp can be found on top of the console and are shown in figure 1A. Take the LN2 VT line off of the top of the console (figure 1B) and slide the o-ring all the way up to the top of the LN2 VT line (figures 2A and B).

Figure 1 (A) O-ring and ring clamp (B) VT Line for LN2 use

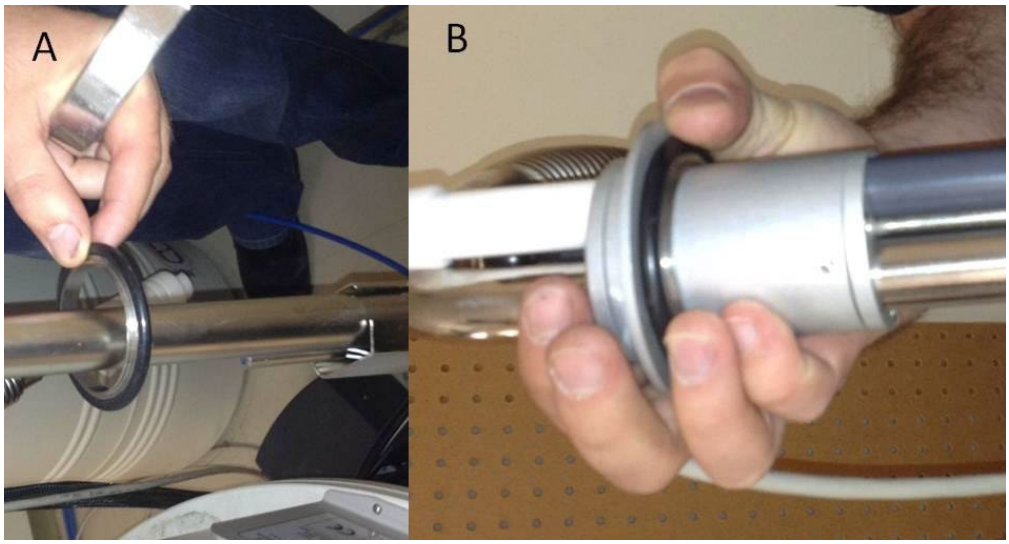

Figure 2 O-ring on (A) the bottom and (B) the top of the LN2 VT line

(h) Place the LN2 VT line in the LN2 dewar and let the line cool down. The line is cold when the pressure release valve stops hissing.

(i) Secure the LN2 VT line in the LN2 dewar by attaching the ring clamp finger tight as shown in figure 3.

Figure 3 LN2 Dewar with the LN2 VT Line in place and secured with the ring clamp

(j)Place the Velcro strap on the VT line to provide the line with support.

(k) Gently push forward on the white Teflon nozzle found at the end of the LN2 VT line and attach the line to the probe as shown in figure 4. **NOTE:** No grease is required as long as the white string is holding the line level and straight. Now that the LN2 VT line is securely connected you can begin your experiments in the ultra-low temperature range.

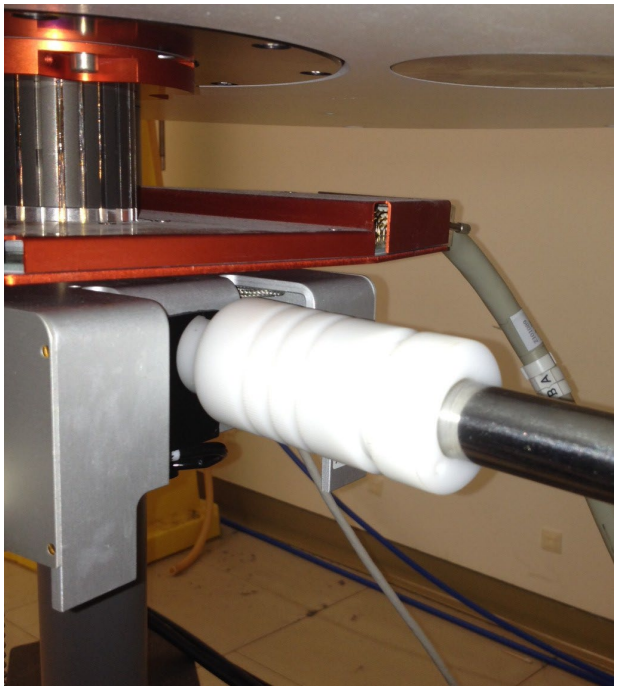

Figure 4 Picture of the LN2 VT line attached to the probe.

(8) Run your spectra at each desired temperature and be sure to check both your tuning and shimming at each temperature as these are highly temperature dependant. The temperature is varied by opening the VT interface by typing edte in the command line and hitting enter.

Normal and high temperature operating ranges: you just need to set your target temperature and wait for the sample and probe to warm up. If the probe is taking an excessive amount of time to warm up you can increase the max power setting for the heater but this setting should not exceed 25%. The gas flow should remain at 535 L/h and the cooling setting should remain at empty and off.

NOTE: Do not exceed the boiling point of your solvent.

Low temperature range: The default gas flow of 535 L/Hour will allow you to reach -15 ºC. You will need to add the mass-multiplier ring to the top of the spinner for all temperatures in this range and you will also need to adjust the VT gas flow according to Appendix A. Please pay attention to the time required to cool-down and warm-up the systems when booking your spectrometer time.

Ultra-Low temperature range: Gas flow should be set to 0 L/h as the console gas line is disconnected from the probe (step e). Turn the cooling setting on and set your target temperature. You should adjust/increase the cooling flow according to your needs as detailed in Appendix B. In other words, the cooling setting should be increased to allow you to reach your target temperature but not go below it.

NOTE: Do not go lower than 10 degrees from the melting point of the solvent!

(9) Once your VT experiments are completed, open up the VT interface by typing edte in the command line and hitting enter. Please be patient and wait while the VT interface opens. Return the VT parameters to the default settings shown below:

Target temp = 22ºC

Probe heater = ON

Max power = 5%

Gas flow = 535 L/h

Cooling = empty and off

NOTE: Be sure that when you book your VT experimental time you allow enough time after you are completed for the system to return to 22ºC.

(10) Lastly, use the following procedures for properly removing your VT setup and returning the instrument to normal use:

(A) Normal and High Temperature Operational Range: +20 ºC to +150 ºC

For this VT range no setup was required and the system is already configured for normal use, therefore no removal is required. **NOTE:** please make sure you have set the EDTE settings to those given in step 9 above.

(B) Low Temperature Range: +20 ºC to -55 ºC

Ensure that the EDTE settings have been returned back to the default values given in step 9 above and then go to the back of the BCU II unit and change the dial setting on the unit from “3” to the “flush” setting. Be sure to put the mass multiplier ring back in its case.

(C) Ultra-Low Temperature Range: -50 ºC to -150 ºC

Ensure that the EDTE settings have been returned back to the default values given in step 9 above. Once the system has warmed back up to 22ºC you need to complete the following steps to return the system back to normal operation:

(a) Disconnect the LN2 VT line from the probe head and replace it with the trunk line from the BCU that you disconnected earlier.

(b) Remove the ring clamp from the top of the LN2 VT line and pull the line out of the dewar. Cap the dewar and place the VT line, ring clamp and o-ring back up on top of the console and allow them to warm up.

(11) Load the initial room temperature shim that you saved in step 3 above. This can be done by typing "rsh RoomTemp-VT" into the command line and hitting enter. This will reload the initial room temperature shim for the instrument. This step is ESSENTIAL to ensure the instrument is ready for the next user.

(12) The System is now back in normal operational mode and should be ready for the next user of the instrument!

NOTE: Be sure that when you finish your VT experiments the edte settings are set back to the default Values shown in step 9 above!!! If you do not return the system to these values you could lose your VT privileges.

BE SURE to allow adequate time following your last experiment for the system to return back to the default temperature. In other words **YOU MUST** book enough time that when the next scheduled user begins the temperature of the system is back at 22ºC. Failure to do so leads to wasted time for the next user and could result in the loss of your VT privileges.

Appendix A – Low Temperature Range Table

Cool down by switching the BCU-II setting from “flush” to “3”. The mass-multiplier ring should always be used for low temperature experiments.

Target Temperature	VT Gas Flow (L/Hr)	Max Heater %	Time to Stabilize from 22 °C (minutes)
+20 to -15 °C	535	15	20
-15 to -25 °C	670	10	25
-25 to -30 °C	800	10	30
-30 to -35 °C	935	10	35
-35 to -40 °C	1200	10	40
-40 to -45 °C	1335	10	45
-45 to -50 °C	1470	10	50
-50 to -55 °C*	1600	10	60

* a consistent temperature of -55 might not be attainable. If not, drop to -54 degrees.

Appendix B – Ultra-low Temperature Range Table

Cool down using 8% cooling to cool quickly then adjust to the following settings

Allow at least 3min for every 10° Step to Stabilize.

Target Temperature	Cooling %	Max Heater %	Time to Stabilize from 22 °C (minutes)
10 °C	4	3	8 - 10
0 °C	4	9	8 - 10
-10 °C	4	9	10 - 15
-20 °C	6	9	
-30 °C	7	9	
-40 °C	8	9	
-50 °C	10	9	
-60 °C	13	9	
-70 °C	15	9	