

# **Variable Temperature NMR Experiments**

## 1. Introduction to Variable Temperature (VT) NMR

### 1.1 What is VT NMR?

NMR experiments in the Department of Chemistry are normally run at an ambient temperature of 25°C (298K). It is sometimes necessary to run experiments at temperatures significantly higher or lower than ambient. Such variable temperature (VT) experiments are used for a number of reasons, mainly to provide insights into the dynamic and kinetic behaviour of molecules, or to simplify spectra for compounds that are undergoing conformational exchange e.g. rotamers.

Changing the probe temperature away from ambient has the potential to cause serious damage to an NMR spectrometer. Therefore please be careful when performing a VT NMR experiment, and take the time to familiarise yourself with the contents of this document.

## You must NOT perform any VT NMR experiments unless you have been trained to do so.

Common reasons for running high temperature NMR:

- Resolving (sharpening) broad peaks due to exchange processes
- Following chemical or conformational exchange processes
- Following a high temperature reaction
- Solubility issues

Common reasons for running low temperature NMR:

- Following chemical or conformational exchange processes
- Trapping reaction intermediates
- Following a low temperature reaction
- Chemical instability

### 1.2 Important considerations for VT NMR

Please follow these instructions carefully. If you are unsure of anything *ask a member of the NMR staff for assistance.* 

- Available instruments: The instruments that are available for VT NMR work are the AVB500, AVX500 and AVD500, all of which are located in the NMR suite in the CRL basement. The AVB500 and AVX500 instruments have chiller (fridge) attachments for (limited) sample cooling.
- **2. Temperature limits:** We generally advise operating temperatures of no higher than 100°C (373K) and no lower than -80°C (193K).
- **3. Appropriate tubes:** It is essential that you use only Class A glass tubes ("pyrex") for VT work, such as Norell S400, Wilmad 507 or above. Cheaper (Class B) glass will deform at temperature extremes and may fracture; this includes the commonly used "disposable" NMR tubes which are not suitable for VT work.

**4. Appropriate spinners:** When performing VT NMR you must always use a brown PEEK or white ceramic spinner (turbine) (Figure 1) with either high or low temperatures. This is because the normal turbines can be damaged at extremes of temperature.

**Note:** Please be very careful when inserting an NMR tube into the ceramic spinners, as there is often a very tight fit and a consequent risk of injury due to breakage of the tube. The best way to insert an NMR tube into a ceramic spinner is by **gently** and **slowly** applying a twisting pressure to the tube during insertion.



Figure 1: Appropriate spinners for VT NMR (Left: ceramic, Right: PEEK).

- 5. Appropriate solvent: Make sure that you use a solvent with an appropriate boiling or freezing point i.e. it will not boil or freeze at your target temperature. Although an obvious statement, numerous chemists have been known to make this mistake. You must not get any closer than 10°C (15°C is recommended) to the boiling or freezing point (please see the NMR solvent sheets near the instruments for these). In addition, it is good practice not to use a sealed NMR tube when performing high temperature NMR. The most common solvents for high-temperature NMR are DMSO (BP 189 °C) or toluene (BP 111 °C), and for low-temperature are CD<sub>2</sub>Cl<sub>2</sub> (MP -95 °C), MeOD (MP -98 °C) or toluene (MP -95 °C).
- **6. Allow sufficient time:** When planning VT experiments, please make sure you have enough instrument time to allow the system to equilibrate at ambient temperature before the next user; this will usually require an additional 20-45 minutes after your experiments, depending on what temperature the spectrometer was set at.
- **7. Sample solubility:** Bear in mind that solubility can be reduced at low temperatures. Therefore if you suspect that your sample is coming out of solution at low temperature, it may be necessary to either dilute your sample or to use a higher temperature.
- **8.** If you want to start measurement immediately at a certain temperature (e.g. for reaction monitoring), you should have a "blank" NMR tube, with an equivalent volume of your solvent. When you have reached the desired temperature, tune, match and shim on this blank sample. You can then exchange this with your sample, which you should have cooled/heated to the correct or approximate temperature externally. You can then either start your experiment immediately without further shimming, or you can use a "quick" shimming routine, by typing in "topshim convcomp ordmax=3" instead of the usual "tshim" or "tshimvt" command.

## 1.3 General procedures

It is usual procedure to run VT experiments under manual operation only using manual sample handling and **not** through ICONNMR.

## 1.3.1 Removing or disabling the autosampler

<u>SampleXpress lite</u>: Ensure no sample is present in the spectrometer before starting. To stop and disconnect the <u>SampleXpress lite</u> autosampler device, hold down the on/off button until the light goes out, then gently **lift** the carousel off the top of the magnet (Figure 2). Make sure that you place it safely on a table.

<u>SampleCase</u>: These autosamplers cannot be removed. Eject any sample in the magnet beforehand, then turn it off by pressing the red on/off button (Figure 3).

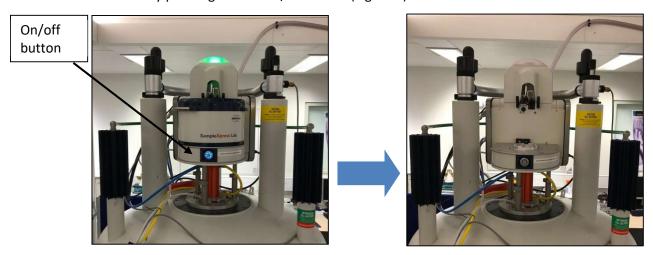
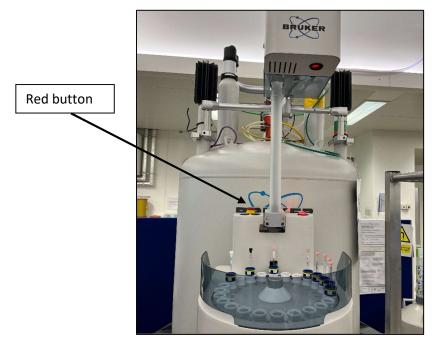


Figure 2: Removing the autosampler carousel from the SampleXpress Lite.



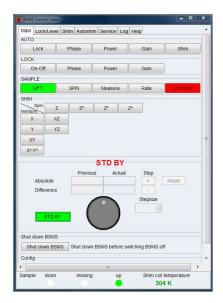
**Figure 3:** Disabling the *SampleCase* autosampler on the AVX500. The red light indicates the unit is disabled.

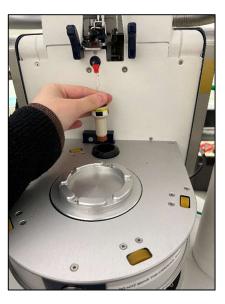
### 1.3.2 Manual sample insertion (2 methods)

Insert using the BSMS control suite: To manually insert your sample, open the BSMS display by either

typing "bsmsdisp" into the command line in Topspin, or by clicking on the keypad icon "Sample" section under the "Main" tab, click on the "Lift" button, which should turn green (Figure 4). Wait until you can hear air coming out of the bore on top of the magnet. Place your sample into the bore, and click on the "Lift" button once again. The button colour will change to grey, and the sample should slowly descend into the magnet.

Insert using ej/ij command: Type 'ej' into the command line to turn on the lift gas. Wait until you can hear air coming out of the bore on top of the magnet. Place your sample into the bore, then type 'ij' into the command line. The sample should slowly descend into the magnet.





**Figure 4:** Manual sample insertion: *Left*: the BSMS panel and *Right*: sample insertion to the AVB500 magnet bore.

If the sample fails to descend into the magnet when the lift gas is turned off, do not touch the sample. This can happen when the spinner gets caught on the autosampler hardware. Repeat the same procedure as above. If the sample still fails to descend, repeat the procedure however when turning the lift gas off, press a small amount of pressure on the top of the NMR tube to guide the tube into the bore.

# **High Temperature Experiments**

### 2.1 Overview

The upper temperature limit for experiments is typically 100°C. In occasional circumstances, it may be possible to exceed 100°C for short periods of time, but this **must only be done with the agreement of the NMR staff**. Once again, you are reminded to check the boiling point of your solvent, and to **not** use a temperature within 10-15°C of this boiling point. The shim coil system has an upper limit of 80 °C and this must never be exceeded.

### 2.2 Preparation

### 2.2.1 Inserting your sample

You must use **manual** mode to insert your sample, which should now have the appropriate turbine attached (see section 1.2.4). Remove or disable the autosampler as described in section 1.3.1. Insert your sample into the magnet as described in section 1.3.2.

## 2.2.2 Run a room temperature experiment.

Now that your sample is in the magnet, it is good practice to run an experiment at ambient temperature, to ensure sample integrity and optimal spectrometer operation.

## 2.3 Establishing high temperature

### 2.3.1 Switch off the chiller unit

If using the AVB500 or AVX500, it is important to switch off the chiller unit before any heat is applied to the probe. This is done by selecting the "Flush/0" setting, as shown in Figure 5.



Figure 5: Turning off the chiller units on the AVB500 and AVX500- turn dial to Flush / 0.

If the Switch on the chiller unit is set to 'Remote', the settings can be changed within TopSpin, using the **Set** button highlighted in Figure 6. Change the Target Power from Low to Off.

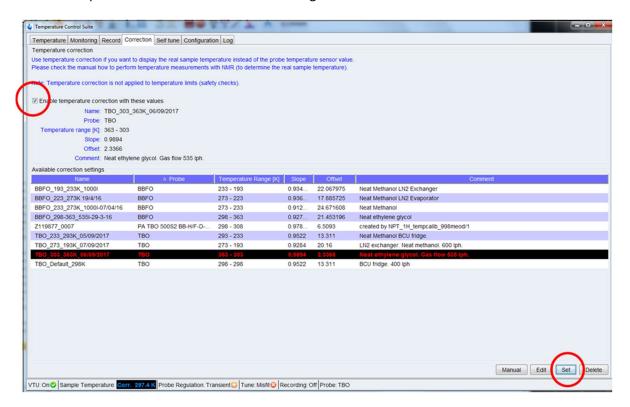


Figure 6: Turning off the chiller remotely by changing Target Power to Off.

## 2.3.2 Set the correct temperature calibration

To access the VT temperature control unit, type **edte** on the top right hand corner of TOPSPIN, or double click the sample temperature window on the bottom right-hand side.

Load the temperature correction and calibration settings, accessible from the "Correction" tab of the Temperature Control Panel, as shown in Figure 7. Select the calibration file for the probe (see Table 1) and desired temperature range, and click on the **Set** button. Make sure there is a tick in the box for "Enable temperature correction with these settings".



**Figure 7:** Setting the temperature calibration file.

### 2.3.3 Setting the temperature and gas flow

The temperature control panel is shown in Figure 8, viewed with the "Temperature" tab. The VTU status should be on. Ensure the Target Gas Flow is set to the required lph specified in the 'Comment' section in the 'Correction' tab (see Figure 7). It is **extremely important** to ensure correct gas flow, as failure to do so could overheat the probe and shims. If no value is specified, set to 535 lph. Set the Target Temperature to the desired value using the **Set** button. It is advisable to only change temperature in steps of 10-20°C to minimise thermal shock to the probe, and progress step-wise to your desired temperature. To change the units of temperature (K to °C, or vice versa), right click on the sample temperature window on the bottom right-hand side, click 'Options' and under Temperature Unit, select the desired 'Kelvin' or 'Celsius'. If changed, ensure this is set back to Kelvin once finished.

**Note:** The Sample Temperature indicator is colour coded. When the sample temperature is regulated, it turns green. When the temperature is too low or too high, the colour becomes blue or red, respectively.

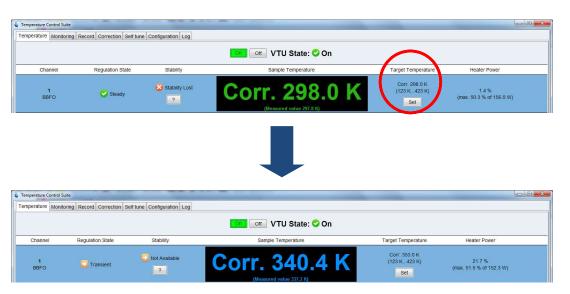


Figure 8: Setting the temperature for high-temperature NMR

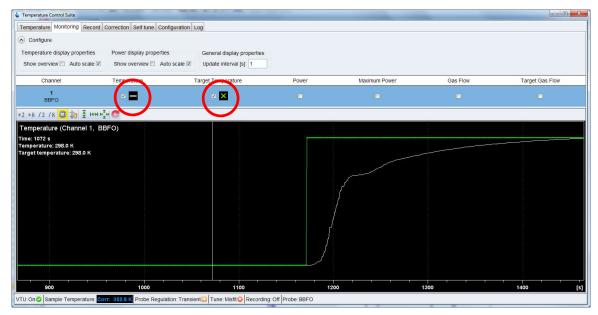
Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	535	303K_373K_535lph_09092024
AVX500	ТВО	535	TBO_303K-373K_600lph_11092024
AVD500	BBFO	400	High temp Ethylene Glycol

**Table 1**. Gas flow settings and probes for high-temperature NMR. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

### 2.3.4 Equilibrating the temperature

Allow the system to equilibrate at the final temperature, which can take up to 20-30 minutes. The shim coil temperature will also increase during this time. A stable temperature is indicated by a completely horizontal line in the "Monitoring" tab, when the "Temperature" and "Target Temperature" fields are selected (Figure 9A), and by a green tick in the Temperature Control Panel (Figure 9B).

(a)



(b)

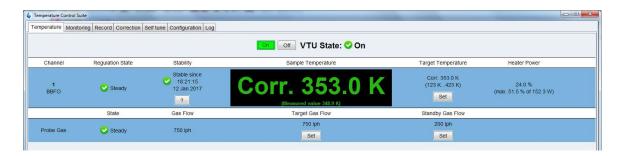


Figure 9: a) Monitoring the temperature during equilibration, b) display when temperature has stabilised.

## 2.3.5 Running high temperature NMR experiments

Now that the temperature has equilibrated, you are ready to perform your high temperature NMR experiment. Data acquisition is done as normal, including all locking, tuning/matching, and shimming steps. For shimming, you should use the *tshimvt* routine in place of *tshim*. If leaving the instrument unattended, it is advisable to leave a note warning the instrument is running in VT mode.

## 2.3.6 Returning the spectrometer to ambient temperature

Having performed all your high temperature experiments, **please remember** to return the VT unit back to ambient temperature by carrying out the above procedures in reverse:

- a) Set the temperature correction back to the default calibration file (refer to Table 2).
- b) Set the chiller/fridge unit back to the original "1" position, or "Low" Target Power.
- c) Decrease the temperature using the set button to 298K, in steps of 20 K.
- d) Decrease the airflow back to the default value (refer to Table 2).
- e) Wait until the temperature is near ambient.
- f) Eject the sample manually from probe when it is safe to do so.
- g) Replace the autosampler to its original state in the 'on' position for the next user.

Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	535	Default_298K_535lph_09092024
AVX500	ТВО	400	TBO_Default_298K_05092024
AVD500	BBFO	400	High temp Ethylene Glycol

**Table 2**. Temperature calibration files and gas flow settings for ambient temperature (default) NMR. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

# 3. Low Temperature Experiments using the Chiller

#### 3.1 Overview

There are two ways to perform a low temperature experiment. For temperatures from room temperature down to -40°C (233 K) on AVX500, it is advisable to use the attached chiller unit (BCU II) as described here. For temperatures below -40°C (233 K), and for all low temperature experiments on all other instruments, it is necessary to use a liquid nitrogen exchanger (see Section 4).

### 3.2 Preparation

## 3.2.1 Inserting your sample

You must use **manual** mode to insert your sample, which should now have the appropriate turbine attached (see section 1.2). Remove the autosampler if using the *SampleXpress lite*, or disable the *SampleCase* autosampler, as described in section 1.3.1. Insert your sample into the magnet as described in section 1.3.2.

### 3.2.2 Run a room temperature experiment

Now that your sample is in the magnet, it is good practice to run an experiment at ambient temperature, to ensure sample integrity and optimal spectrometer operation.

3.3 Low temperature experiments using the BCU chiller unit on the AVX500 (BCU II) and AVB500 (BCU I).

## 3.3.1 Overview

The BCU II chiller unit may be used for temperatures down to -40°C. In general, the use of the chiller unit is less complicated and time-consuming than the use of a nitrogen exchanger. Therefore for temperatures down to -40°C we advise using this procedure. A low-temperature chiller unit is only available on the AVX500 (that on the AVB500 cools to only  $^{\sim}5$  °C). If you are using other instruments for low-temperature NMR, you should use a nitrogen exchanger as described in Section 4.

## 3.3.2 Adjust the chiller unit

Switch the chiller unit to the "3" setting, as shown in Figure 10, on the AVX500. For the AVB500, switch the chiller unit to the "2" setting. Alternatively, if the chiller is set to Remote, change the Target Power to "Max" using the **Set** button highlighted in Figure 6 above.



Figure 10: Switch the chiller unit to the "3" position on the BCU II chiller unit.

## 3.3.3 Setting the temperature and gas flow

Open the VT temperature control window. Set the appropriate temperature calibration file and gas flow, as specified in Table 3.

Set the Target Temperature to the desired value using the **Set** button. It is advisable to change temperature in steps of 10-20°C only to minimise thermal shock to the probe, and progress step-wise to your desired temperature.

Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	535	273K_298K_535lph_12092024
AVX500	TBO	600	TBO_253_293K_05092024

**Table 3.** Temperature calibration files and gas flow settings for low-temperature NMR using the chiller unit. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

### 3.3.4 Temperature equilibration

Allow the temperature to equilibrate, which can take up to 20 minutes and can be checked using the "Monitoring" tab. Temperature stability is achieved when a completely horizontal line is achieved in the Temperature Monitoring Panel and when a green tick appears in the Temperature Control Panel (Figure 9 above). If the system is at a sub-zero temperature for a number of hours, the probe may be unable to tune/match. It is therefore recommended to tune/match to your desired nuclei as soon as the spectrometer has reached the desired temperature.

**Note:** The Sample Temperature indicator is colour coded. When the sample temperature is regulated, it turns green. When the temperature is too low or too high, the colour becomes blue or red respectively.

## 3.3.5 Running low temperature NMR experiments

Now that the temperature has equilibrated, you are ready to perform your low temperature NMR experiment. Data acquisition is done as normal, including all locking, tuning/matching, and shimming steps. For shimming, you should use the **tshimvt** routine in place of **tshim**. If leaving the instrument unattended, it is advisable to leave a note warning the instrument is running in VT mode.

### 3.3.6 Returning the spectrometer to ambient temperature

Having performed all your low temperature experiments, **please remember** to return the VT unit back to ambient temperature by carrying out the above procedures in reverse:

- a) Set the temperature correction back to the default calibration file (refer to Table 4).
- b) Set the chiller/fridge unit back to the original "1" position, or "Low" Target Power. Note the BCU may take up to 20 minutes to kick in.
- c) Increase the temperature using **set** button to 298K, in steps of 20 K.
- d) Decrease the airflow back to the default value (refer to Table 4).
- e) Wait until the temperature is near ambient.
- f) Eject the sample manually from when it is safe to do so.
- g) Replace the autosampler to its original state in the 'on' position for the next user.

Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	535	Default_298K_535lph_09092024
AVX500	TBO	400	TBO_Default_298K_05092024
AVD500	BBFO	400	High temp Ethylene Glycol

**Table 4.** Temperature calibration files and gas flow settings for ambient temperature (default) NMR. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

## 4. Low Temperature Experiments using the Nitrogen Exchanger

#### 4.1 Overview

For temperatures below -40°C on the AVX500, and for all low temperature work down to -80°C on other instruments, it is necessary to use a nitrogen exchanger. This device uses a tank of liquid nitrogen to cool a flow of nitrogen gas, which is then fed to the probe.

## 4.2 Liquid Nitrogen Dewar

The first step is to fill a 25-litre LN<sub>2</sub> dewar (Figure 11) with liquid nitrogen, using a filling station, ensuring to follow the safety procedures for handling liquid cryogens. The closest filling station to the NMR laboratory is located adjacent to the goods lift. Use the wooden trolley shown in Figure 10 to transport the dewar to and from the filling station. Once full, return the dewar to the NMR laboratory, remove it from the trolley, and place it adjacent to the magnet, as shown in Figure 11.

**Note:** Follow all appropriate safety guidelines, including the wearing of safety glasses and thermal gloves, when filling the dewar with liquid nitrogen. If you are transporting the dewar to a filling station on another floor, **NEVER** travel with the dewar in a lift.





**Figure 11:** *Left*: Liquid nitrogen dewar and trolley. *Right*: Appropriate positioning of liquid nitrogen dewar with respect to the magnet.

### 4.3 Low temperature experiments

## 4.3.1 Inserting your sample

You must use **manual** mode to insert your sample in an **appropriate tube**, which should now have the **appropriate turbine** attached (see Section 1.2). Remove or disable the autosampler as described in Section 1.3.1. Insert your sample into the magnet as described in Section 1.3.2.

## 4.3.2 Run a room temperature experiment

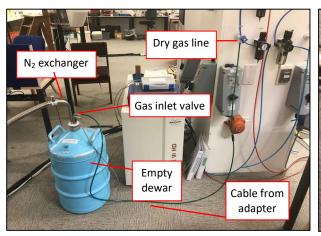
Now that your sample is in the magnet, it is good practice to run an experiment at ambient temperature, to ensure sample integrity and optimal spectrometer operation. Remember to use a correct VT spinner and Class A NMR tube, as only these are tolerant of temperature extremes.

## 4.3.3 Prepare the transfer Line

Before and after use, the transfer line must be flushed through with dry gas in order to remove any condensation (see Figures 12 and 13). Use the nitrogen gas line adjacent to the AVB400. First, place the nitrogen exchanger in an empty dewar. Then connect the gas cable line (leads to a ball and cup joint on the N2 exchanger) securely to the gas inlet valve on the  $LN_2$  exchanger and ensure a good seal. Ensure the ball and cup joint on the  $N_2$  exchanger is correctly positioned to allow adequate gas flow. Open the  $N_2$  gas valve **very slightly and carefully**, so that you can hear  $N_2$  gas flowing out of the  $N_2$  exchanger. Leave the line flushing for a minimum of 5 minutes.



Figure 12: Nitrogen exchanger and transfer line for low temperature NMR.



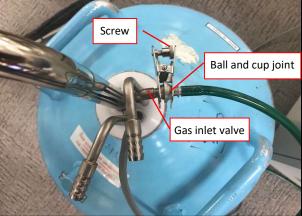


Figure 13: Flush dry gas through the transfer line.

## 4.3.4 Connect the N<sub>2</sub> transfer line

If you are using an instrument equipped with a chiller unit (BCU) you should set this to "flush" mode and leave this for 5 mins to purge the BCU. You will then need to detach the  $N_2$  gas feed into the chiller and connect this to the gas cable line coming from the adapter ("cup") that connects to the  $N_2$  exchanger (Figure 13 above). If the instrument does not use a chiller, you will only need to connect the usual  $N_2$  gas line to the  $N_2$  gas cable line. Then, to set-up the  $N_2$  transfer line, follow these steps:

**1.** In TopSpin open the Temperature Control Suite by typing "edte" in the command line, or by clicking on the licon. Turn off the Variable Temperature Unit (VTU) as shown in Figure 14.

**Note:** it is important to turn off the VTU before connecting the LN<sub>2</sub> transfer line in order to prevent overheating of the probe.



**Figure 14:** Turning off the VTU.

**2.** Disconnect either the black VT gas connector on BCU hose from the probe (Figure 15a) or disconnect and remove the VT adaptor (Figure 15b), according to instrument set-up. Connect the gas line terminated with the cup adaptor to the probe  $N_2$  gas line (Figure 15b, bottom right).

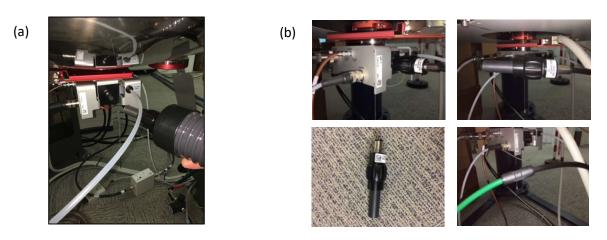


Figure 15: Remove from the probe either a) the BCU hose or b) the VT adaptor.

3. SLOWLY insert the coils of the nitrogen gas exchanger into the LN<sub>2</sub> dewar, as shown in Figure 16.

**Note:** Make sure the two nozzles on the cap are pointing away from you, as these can spray jets of liquid nitrogen if the probe is inserted too quickly. Make sure that you wear **gloves** and **safety glasses** while doing this.



Figure 16: Inserting the N<sub>2</sub> exchanger into the LN<sub>2</sub> Dewar.

**4.** Attach the transfer line to the probe <u>GENTLY</u>. Keep the transfer line horizontal when inserting it into the probe. There should be no resistance if the positioning of the dewar and transfer line is correct (see Figure 17). Once the transfer line is in place, screw the end into place. This is more difficult to do on the AVX500 due to the position of the grey bar connecting the three legs of the magnet.

**Note:** never exert force to push the transfer line into the probe. Ask for help if needed.

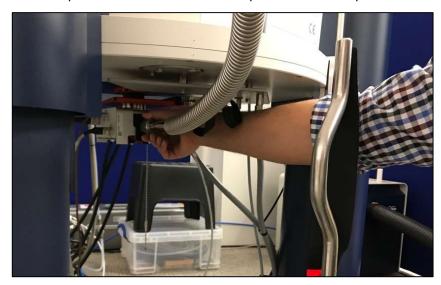


Figure 17: Insertion of the  $N_2$  exchanger transfer line into the probe.

5. Connect the  $N_2$  gas supply line to the  $N_2$  exchanger unit using a "cup" adapter and clamp this in place (see Figure 13 above).

## 4.3.5 Setup for low temperature NMR

To set up your low temperature NMR experiment(s), follow these steps:

1. In the Temperature Control Suite, click on the "Correction" tab. Select the appropriate temperature correction file according to temperature range and probe, as specified in Table 5, and click on the **Set** button (Figure 18).

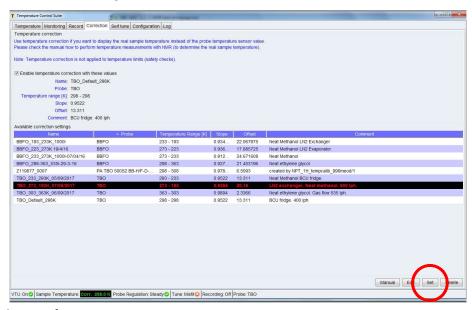


Figure 18: Selection of temperature correction parameters.

2. In the "Temperature" tab of the Temperature Control Suite, set the appropriate gas flow rate according to Table 4 then the desired temperature by clicking on the Set button, entering the temperature, and clicking on the OK button. Remember to decrease temperature in progressive steps of ~ 20°C.

Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	600	193K_273K_600lph_09092024
AVX500	TBO	600	TBO_193K-273K_600lph_11092024
AVD500	BBFO	600	N2 Exchanger low temp 600I/h gas flow

**Table 5**. Temperature calibration files and gas flow settings for low temperature NMR for the LN2 exchanger unit. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

**3.** Turn on the VTU by clicking the **ON** button in the top row of the "Temperature" tab (Figure 19). Allow the temperature to decrease to the set point and then reduce this further in stages as required.



Figure 19: Turning on the VTU

**4.** Allow the temperature to equilibrate, which can take up to 20 minutes and can be checked using the "Monitoring" tab. Temperature stability is achieved when a completely horizontal line is achieved in the Temperature Monitoring Panel and when a green tick appears in the Temperature Control Panel (Figure 9 above).

**Note:** the Sample Temperature indicator is colour coded. When the sample temperature is regulated, it turns green. When the temperature is too low or too high, the colour becomes blue or red respectively.

Now that the temperature has equilibrated, you are ready to perform your low temperature NMR experiment. Data acquisition is done as normal, including all locking, tuning/matching, and shimming steps. For shimming, you should use the **tshimvt** routine in place of **tshim**. If leaving the instrument unattended, it is advisable to leave a note warning the instrument is running in VT mode.

## 4.3.6 Returning the system to room temperature

Having performed all your low temperature experiments, **please remember** to return the VT unit back to ambient temperature by carrying out the above procedures in reverse:

- a) Set the temperature correction back to the default calibration file (refer to Table 6)
- b) Increase the temperature using **set** button to 298K, in steps of 20 K.
- c) Decrease the airflow back to the default value (refer to Table 6).
- d) When the temperature reaches 298K, turn the VTU off by clicking the **Off** button in the **Temperature** tab of the Temperature Control Suite.
- e) Disconnect the nitrogen transfer line from the probe and detach the N<sub>2</sub> gas line.
- f) Connect the N<sub>2</sub> gas line to the black VT adaptor and attach this to the probe, making sure to do this as quickly and safely as possible [NB: If a chiller is available, reconnect the N<sub>2</sub> gas feed to the chiller and set the unit back to the original "1" (low) position].
- g) Turn the VTU on by clicking the **On** button in the **Temperature** tab of the Temperature Control Suite.
- h) Eject the sample manually from when it is safe to do so.
- i) Replace the autosampler to its original state in the 'on' position for the next user.

Spectrometer	Probe	Gas Flow (lph)	Calibration File
AVB500	BBFO	535	Default_298K_535lph_09092024
AVX500	TBO	400	TBO_Default_298K_05092024
AVD500	BBFO	400	High temp Ethylene Glycol

**Table 6**. Temperature calibration files and gas flow settings for ambient temperature (default) NMR. These parameters were correct as of February 2025. If the values differ on the spectrometer, use those values rather than those specified in this table.

## 4.3.7 Flush the $N_2$ exchanger line

After using the  $N_2$  exchanger it is advisable to flush the line with dry gas to prevent the build-up of condensation inside the line, which could lead to ice formation and blockage on subsequent low-temperature use. Connect the exchanger as in Figure 12 above and follow the procedure described in section 4.3.3 to purge the line. The line should be purged for five minutes, or until there is no ice/frost remaining on the exchanger. Note whilst the exchanger line warms up to ambient temperature, the faint whistle will change in pitch.

If in doubt about any aspect of VT NMR, please consult a member of the NMR staff for advice or assistance.