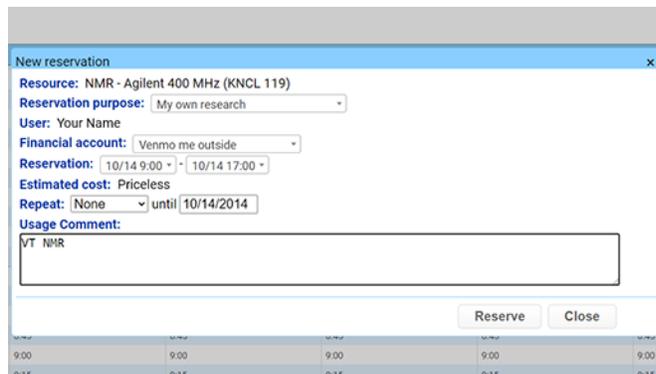# Variable Temperature (VT) Operation on the 400 MHz

1. All requests for VT NMR must be approved by MRC staff before scheduling.

   When you schedule on FOM, enter "VT NMR" into the **Usage Comment:** box so other users know that the extended reservation is approved use.

2. **Before changing the temperature,** YOU SHOULD ALWAYS VERIFY THAT THE INSTRUMENT IS SWITCHED TO THE NITROGEN PNEUMATIC SUPPLY.

   The copper line is labeled as NITROGEN and the black line is standard compressed air. The yellow handles should appear as shown.

2a. If the yellow ball valve handles are parallel to the supply line, that line is open. If perpendicular, the supply is closed. If the instrument is still on the compressed air supply, the handles will be positioned as shown.

2b. To change supply, first turn the nitrogen supply's ball valve handle 90° counterclockwise so both supply lines are open.

2c. Then turn the air supply's ball valve handle 90° clockwise to close it. These steps should be done stepwise to avoid cutting off all pneumatic supply to the instrument.

You should wait at least 5 minutes to flush the system with nitrogen before continuing.

3. The VT spinner is stored in the "Strd samples" drawer of the cabinet below the pneumatic lines. This spinner has extra holes to promote air flow and should be used for all VT samples. When loading samples into this spinner, be careful not to dislodge the bottom O-ring or break its support fingers.
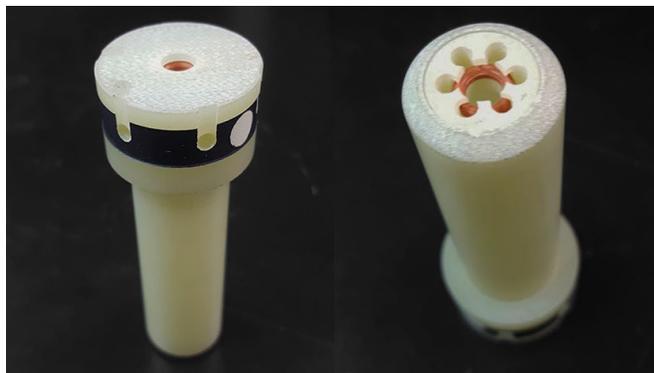
4. Quickly load your sample into the magnet. You should not leave the eject air on for more than is necessary when loading samples at low/high temperature.

If your experiment requires the instrument to be at temperature before inserting your sample (e.g. kinetics), load another sample, or an empty NMR tube, into the VT spinner first and insert it into the magnet while adjusting temperature.

Always verify the freezing/boiling point of the sample in the magnet *before* **changing the temperature. The sample should remain liquid at the final set point.** Any experiment to be ran with a sample close to the freezing/boiling point must be discussed with the MRC staff before the sample goes in the manget.

Never adjust the temperature on the standard. Do not assume it is D_2O.

5. In VnmrJ, type `setvt` in the command line.

It will then prompt you for the set point temperature. Enter the desired sample regulation temperature (°C).
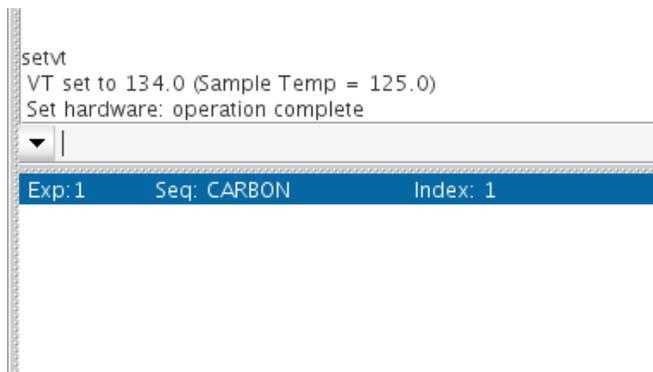

5a. If you set the temperature outside of the probe's limits, you will see the following error message. Rerun `setvt` with a lower temperature or contact the MRC staff to unlock the instrument software for VT use.
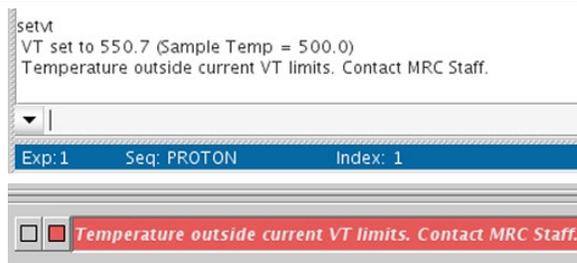
5b. If the entered temperature is within the probe's limits, the output will display the VT controller set point as calculated from the system calibration and begin heating/cooling to that point.

> In this example, when the VT controller reads 134.0, the calibrated sample temperature will be about 125 °C.


```
setvt
VT set to 134.0 (Sample Temp = 125.0)
Set hardware: operation complete
▼ |
Exp:1        Seq: CARBON        Index: 1
```

---

6. **Low Temperature VT NMR:**

If regulating below 25°C, attach the polystyrene bucket. Rotate the bucket so that the tabs protrude from the sides of the lid to ensure it is properly locked in place.

Remove the rubber stopper and pour liquid nitrogen from a narrow-mouthed dewar until the liquid covers the coils by a few inches. Do not fill the bucket fully. The initial fill will require about one full 4L dewar. You will need to monitor the liquid level to ensure the coils stay covered; typically filling every 30 minutes once the coils are cold.

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7. Monitor the temperature change with the external status display or in software. You can display a temperature chart by clicking the **Temp** button in the lower left.

The temperature change should continue at a constant rate and slow as it approaches the VT set point. If it starts changing rapidly, you should disable VT regulation and contact the MRC staff.
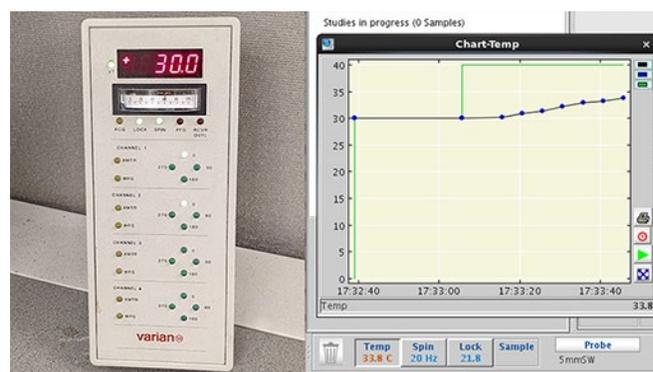
7a. If the temperature status is rapidly increasing, fluctuating by 1°+, or maxes out (999.9), switch the heater to OFF on the VT controller.

The VT controller is located on the back of the console. The most accessible pathway is shown. Be mindful to not step on any cable lines.

The heater switch is to the left of the status display on the VT controller.

Low Temp: Carefully unlock and lower the liquid nitrogen bucket and leave seated directly below the coil.
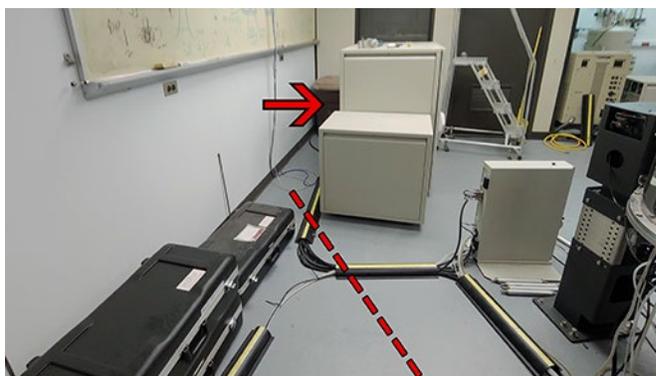
8. Wait at least 10 minutes once the instrument reaches the set point for the temperature to equilibrate across your sample. If changing the temperature by 10°C or more, or running quantitative NMR, you should tune the probe.
    • See the tuning SOP for instructions.

You should touch up shimming after all temperature changes.

9. Run your experiments.

10. If someone is scheduled to run after you, insert either their sample or an empty tube into the VT spinner and load it into the magnet.

When you logout you will be prompted about leaving the magnet below/above 30°C. Click OK.

Otherwise click Cancel and continue to the next step.

Returning the instrument to standard operating temperature (30°C)

11. **Low Temperature VT NMR:**

    If regulating below 25°C, unlock and lower the polystyrene liquid nitrogen bucket. Wait until the set point stabilizes above 10°C to allow the lines to warm before continuing to the next step.
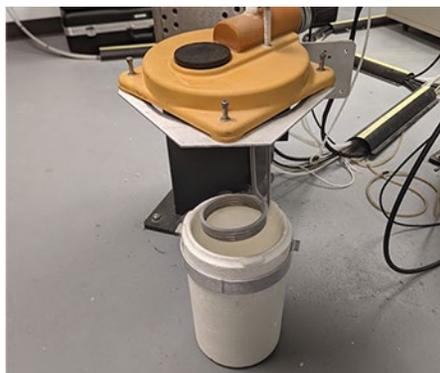
12. Return the set point to 30.

    If you run `setvt` without arguments, you will be prompted if you want to set the VT controller to 30. Enter `y` (entering `n` will result in placing the set point at the calibration for a sample temp of 30°C).

    You can skip this prompt by entering `setvt(30)` to set the temperature controller to 30 directly.
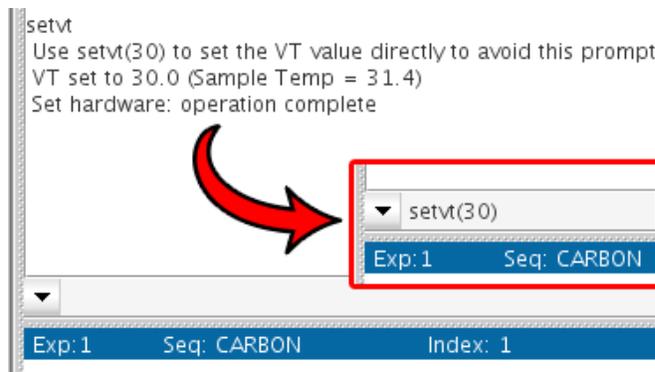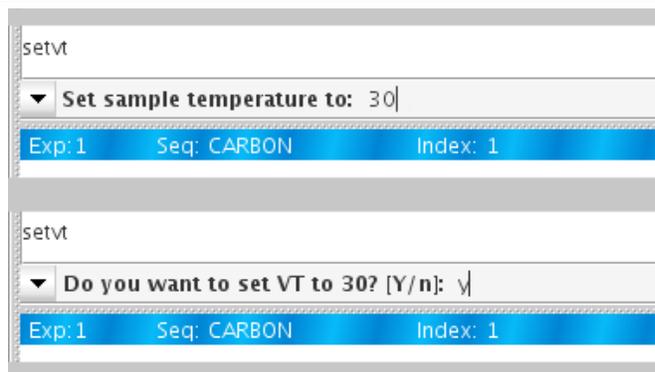
13. Load the standard into the standard spinner, insert it into the magnet, and then retune the probe to the standard.

14. Log out.

15. Return the VT spinner to the "Strd samples" drawer.

16. Switch the pneumatic supply back to compressed air by opening the compressed air supply (Step #2b) and then closing the nitrogen supply (Step #2a).