Variable-temperature NMR
Room temperature to +150°C

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Instrument: Bruker 400 MHz (Stepan) or 500 MHz (McCourtney)
Probes:

- 5 mm Bruker BBFOplus, ambient temperature to +146 °C
- 5 mm Nalorac, ambient temperature to +130 °C

Sample tube: regular, 5 mm
Spinners: POM (regular blue spinner) up to +80°C or Ceramic (white) up to +150°C
Tube caps: Make sure to use the cap that can withstand temperature of the experiment!

Critical requirements and warnings

WARNING: Variable temperature operation stresses the **glass part** of the probe, which may easily crack due to steep thermal gradients. **Follow directions exactly** to avoid damaging the probe!

CRITICAL GUIDELINES:

- The Shim Coil Temperature must stay below +80°C. If your Shim Coil Temperature reached 80C, while the Shim Gas valve is fully open - **you may not proceed** with the next temperature change. **This is the limit of a possible range, YOU MUST STOP.**

- If you cannot see Shim Coil temperature on the bottom toolbar, type **bsmsdisp** and check it there (bottom of the window).

- If you change temperature through EDTE: Probe Temperature may be changed **by a maximum of 10°C at a time.** After a change **you must wait**, at least, **3 minutes** before initiating the next change.

- You may **change samples only while below or at 80°C.** Above this range you must cool down the probe to +80 first before ejecting the sample.

Obtain current calibrations

IMPORTANT: You **must** use the calibration graphs when choosing VTU settings because **real temperature may exceed VTU setting** by more than 20°C outside of ambient temperature range!

Current calibrations are at **nmr.nd.edu** in **NMR Training and Resources** : **Temperature Calibrations**

Preparations

- If you use
    - Stepan 400
        - if you plan to go above 80C thus use a Ceramic spinner, you must switch SampleCase to the **Manual Insertion** mode
        - if you need to use tubes longer than 9 inch (ie, J.Young tubes) , you must switch SampleCase to the **Manual Insertion** mode
    - McCourtney 500:
        - No action needed: SampleExpress Lite changer is compatible with any spinner or a tube.
- insert your sample
- lock, tune, and shim at room temperature
- record 1D of your sample to verify that the instrument is fully functional at room temperature.
- Type **edte** to open VTU control window
- On 500 in McCourtney:
    - check in **edte** window if BCU is "off"
- On 400 in Stepan:
    - Check the front of the BCU: the dial must be in "Flush/0" position

Increase the Shim Gas flow

The room-temperature Shim Gas keeps the shim stack from overheating. The Shim Gas valve is at the top of the magnet body. **If you intend to work at a real temperature of 80°C or above:**

- Fully close the valve by rotating clockwise.
- Rotate the knob counter-clockwise (watch the mark on the knob)
    - **9 full turns** on Stepan 400
    - **12.5 full turns** on McCourtney 500

Operation using EDTE window (manual temperature change)

Ramping to work temperature

- Click **Monitoring** tab, display **Target temperature**, **Coil Temperature**, and **Current Power;**
- Record **Probe** and **Shim Coil temperature** as well as **Gas Flow** in the table as you go;
- **Change temperature by no more than 10 degrees at a time!**

Time													
Probe T													
Gas Flow													

Shim Coil T													

1. Set the **Target Gas Flow** for the next temperature in the **Temperature** tab:
    - below 80C (353 K) using a POM spinner: 400 LPH
    - using a Ceramic spinner:

VTU setting, K	VT Gas Flow, LPH
293	600
353	500
393	400
423	300

2. Increase Probe Temperature by 10 degrees in the **Temperature** tab
3. Wait 3 minutes
4. Repeat these steps 1-3 till you reach your desired temperature.
5. Once the final temperature is reached, **equilibrate for, at least, 15 minutes** for stabilization of hardware
6. Perform **self-tuning** if prompted by EDTE

Experiments at the working temperature

Allow spectrometer to equilibrate for, **at least**, 15 minutes before you start locking/tuning/shimming.

NOTE: Bruker manuals require 30 to 120 minutes for complete stabilization of the hardware (the higher temperature - the longer)!

Ramping back to room temperature

1. Reduce **Target Temperature** by 10^{o}C
2. Change the **Target Gas Flow** as according to the table above
3. Wait 3 min
4. Record **Current Temperature**, **Shim Coil Temperature**, and **Gas Flow** in the table
5. Repeat 1-4 till reach the room temperature (25C)
6. Perform VTU self-tuning procedure if prompted by EDTE

Time													
Probe T													
Target Gas Flow													
Shim Coil T													

Operation using KovriginNMR (automatic temperature change)

KovriginNMR automates temperature ramps taking into account the parameters of the probe, spinner, solvent, and chiller. It automatically changes VTU setting in small increments simultaneously adjusting the VT gas flow and taking into account the temperature calibrations. To use KovriginNMR, please, contact Evgenii Kovrigin for additional training.

Ending your work

- Close the **Shim Gas Valve** fully (clockwise)
- If you were on Stepan 400 and used **Manual Insertion** protocol with the SampleCase, you must switch the system back to the **automatic SampleCase operation**.
- Turn off VTU
- Set **Target Gas Flow** to 200 LPH
- Insert the standard and lock on the $CDCl_3$
- Quit TopSpin and your account
- Report temperature range you used.