

Moderate Low-Temperature Protocol

500 in McCourtney Hall

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Instrument: Bruker 500 MHz (McCourtney), automatic BCU-II chiller (temperature to -58 °C)

Probes: 5 mm Bruker BBFOplus, VT to -150 °C

Sample tube: regular, 5 mm

Spinners: POM (blue) to 0°C; Ceramic (white) to -150°C

Critical requirements and warnings

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

CRITICAL GUIDELINES:

- If you change temperature through EDTE window: Probe Temperature may be incremented **by a maximum of 10°C at a time**. After a change **you must wait 3 minutes** before initiating the next change.
- After you finished your work, when you increased temperature from low temp to room temperature, **you must turn BCU "Off" before you increase temperature further**.
- You can use the **POM (blue) spinner down to 0°C**. You must use the **Ceramic spinner if you intend to go below 0°C**.

Obtain current calibrations

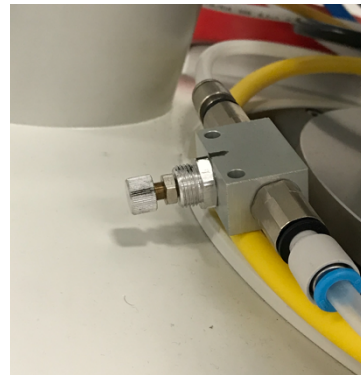
IMPORTANT: You **must** use the calibration graphs when choosing VTU settings because **real temperature may be significantly lower** than the VTU setting at a low end of the temperature range!

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

Preparations

- Lock, tune, and shim at room temperature
- record 1D of your sample to verify that the instrument is fully functional at room temperature.

- Switch BCU-II to a "Medium" setting.
NOTE: It takes about 15 minutes for BCU to achieve sufficient cooling power.
- Set the first "Target Temperature" to 20°C (293K)
- Set Target Shim Gas value to 400 LPH
- Start VTU (click On button)



Shim Gas

If your **real** sample temperature will be at 0°C or below:
open Shim Gas Valve by **12.5 turns counter-clockwise from fully closed state.**
(You need to open the gas flow to 1200 LPH = 0.8 CFM)

You hear a hiss of nitrogen Shim Gas passing through the Shim Stack.

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:
 - If you are using **POM** spinner and work above 0°C (273 K): use **400 LPH**
 - If you use a Ceramic spinner, use:

VTU setting, K	VT Gas Flow, LPH
220	850
233	800
253	750
273	700
293	650

2. Reduce Probe Temperature **by 10 degrees** in the **Temperature** tab
NOTE: When you step VTU setting to below -20C (263 K), turn BCU-II to "Strong" setting.
3. **Wait 3 minutes**
4. Repeat these steps 1-3 till you reach your desired temperature.
5. 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 60 minutes for complete stabilization of the hardware!

Ramping back to room temperature

1. Increase **Target Temperature** by 10°C
NOTE: As you increase temperature to above -20C, switch BCU-II to "Medium" setting.
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)

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** (clockwise till stop)
- Turn BCU-II to "Off" mode

- If there will be the next user after you: **wait, at least, 10 minutes** to allow the BCU to warm up with VTU working.
- Turn off the VTU
- Set **Target Flow** to **200 L/h**
- Insert a standard sample and lock on the CDCl_3
- Quit TopSpin and your account
- Report temperature range you used