Moderate Low Temperature (to -40°C) Bruker 400 in Stepan, SampleCase

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Instrument: Bruker 400 MHz (Stepan), BCU-I manual chiller
Probes:

- 5 mm Bruker BBFOplus, VT to -150 °C (main probe)
- 5 mm Nalorac VT to -80 °C (requires an adaptor for BCU-I)

Sample tube: regular, 5 mm
Spinners: POM (blue) is rated down to 0°C or Ceramic (white) is rated 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.

- When you increase temperature from low temp to room temperature, **you must turn BCU-I into "Flush/0" mode before you increase temperature further.**

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

- If your **real** sample temperature will be
  - below 0°C:
    - insert your sample in the **Ceramic** spinner.
    - use **Manual Insertion** protocol with SampleCase
  - Above 0°C:

    - use **POM** spinner (regular blue spinner). No changes to the SampleCase operation are required.
- Lock, tune, and shim at room temperature
- record 1D of your sample to verify that the instrument is fully functional at room temperature.
- Check if the BCU-I is
    - powered ON (status light lit)
    - attached to the probe
    - VT gas supply is attached to the back inlet
- Set the selector knob of BCU-I to "2" position

Shim Gas

If your **real** sample temperature will be at 0°C or below: open Shim Gas Valve by **9 turns counter-clockwise from fully closed state.**
(You need to open the gas flow to 1200 LPH = 0.8 CFM)

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: use **400 LPH**
    - If you use a Ceramic spinner, use:

VTU setting, K	VT Gas Flow, LPH
250	1400
260	1400
270	800
280	650
290	450

300	300

2. Reduce 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. 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
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-I to "Flush/0" mode
- If there will be the next user after you: wait 10 minutes to allow the BCU-I to warm up

- Turn off the VTU
- If you were using **Manual Insertion** protocol with SampleCase, you must switch the system back to **automatic SampleCase operation**.
- Set **Target Flow** to **200** L/h
- Insert standard and lock on the $CDCl_3$
- Quit TopSpin and your account
- Report temperature range you used