

# Extreme Low Temperature Protocol (down to -150°C)

## Bruker 400 in Stepan, SampleCase in Manual Insertion mode

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**Instrument:** Bruker 400 MHz (Stepan)

**Probes:**

- 5 mm Bruker BBFOplus, VT to -150
- 5 mm Nalorac VT to -80

**Sample tube:** regular, 5 mm

**Spinners:** Ceramic (white) -150°C to +150°C

### Critical requirements and warnings

**SAFETY REQUIREMENTS:** You **must** wear **closed shoes**, **goggles** and **cryogenic gloves** when handling liquid nitrogen dewar and heat exchanger.

**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:

- The Shim Coil Temperature must stay within -80°C to +80°C. If your Shim Coil Temperature reached one of these limits 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 operate EDTE manually: Probe Temperature may be changed **by a maximum of 10°C at a time**, and **you must wait 3 min** before initiating the next change.

- When you increase temperature from low temp to room temperature, **you must disconnect the liquid nitrogen cooler before you increase temperature further.**
- You may **change samples only while above -80°C.** Below this range you must warm up 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 be **lower** than the VTU setting by as much as 60°C at a low end of the temperature range!

Current calibrations are at [nmr.nd.edu](http://nmr.nd.edu) in **NMR Training and Resources : Temperature Calibrations**

## Preparations

- Insert your sample in the **Ceramic** spinner
- Insert sample in the probe using **Manual Insertion** protocol for the SampleCase
- lock, tune and shim on your sample at room temperature
- record 1D of your sample to verify that the instrument is fully functional at room temperature.
- Fill a dewar (TA26) with liquid nitrogen, at least, to 1/2 volume. Bringing too little nitrogen may result in your experiment not reaching a desired low temperature! A full dewar supplies cold gas for about 12 hours.
- Check the front of the BCU: the dial must be in "Flush/0" position
- Turn VTU off
- Detach gas supply from BCU
- Attach gas supply to the heat exchanger to flush it from moisture
- Detach BCU from the probe
- Move dewar to proper location
- Disconnect gas supply from heat exchanger
- Insert the heat exchanger into the dewar
- Attach heat exchanger to the probe
- Attach gas supply to heat exchanger
- Set temperature in EDTE to **295 K**, gas flow target to **200 LPH**
- Turn VTU on

## 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**  
(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:

VTU setting, K	VT Gas Flow, LPH
300	290
280	350
270	440
250	500
230	600
210	700
190	800
170	1070
150	1400

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 1 to 2 hours for complete stabilization of the hardware at the lowest temperature!

### 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** fully (clockwise)
- Turn off VTU
- Detach gas supply from heat exchanger
- Detach heat exchanger from the probe
- Remove heat exchanger from dewar
- Pull out dewar
- Attach BCU to the probe
- attach gas supply to the BCU
- Switch the system back from Manual Insertion mode to the **automatic SampleCase operation**.
- change sample for the standard
- take the tube out of the Ceramic spinner
- wipe off the Ceramic spinner with 70% ethanol

- in EDTE, set target gas flow to **200 LPH**, target temp to **298K (25C)**
- Lock on the  $\text{CDCl}_3$
- Quit TopSpin and your account
- Report temperature range you used.