

Variable Temperature NMR on the Bruker 400 MHz NMR

Low Temperatures: Temperatures between 0 and -130 °C can be achieved with the nitrogen exchanger.

Must Remember:

- Ensure sample tubes and solvent are compatible with the temperature range desired
- Switch to nitrogen gas ~15 minutes prior to dropping temperature below 5 °C
- Use only the Ceramic sample holder
- Purge the exchanger with nitrogen prior to submerging in the Dewar
- Turn on shim gas if coil falls below 5°C

Hookup Procedure

- 1) Turn off air supply, count to 5, turn on nitrogen supply (On wall labeled "N")
- 2) Open VT control by double-clicking on the temperature readout at the bottom of the screen
- 3) Turn VT control off then disconnect VT gas line from probe
- 4) Connect VT gas to exchanger and set standby gas to 800 L/hr. Let purge for 30 seconds to dry line.
- 5) Slowly lower exchanger into liquid nitrogen dewar until seated (watch out for splash!).
- 6) Set standby gas back to 200 L/hr and connect exchanger hose to probe (finger tight – don't forget to remove black cap!)
- 7) Turn VT control back on and select 0 °C as your first temperature.

Operation Procedure

- 1) Set the desired temperature (move in 20 degree increments when dropping)
- 2) On the Self tune tab, either "restore" a previous tune file or run a new "self tune" if your conditions have not been tuned before.
- 3) Wait at least 10-20 minutes after you have reached a stable temperature to ensure the sample and electronics have all equilibrated – then collect spectrum as normal

- Bruker recommends 500 L/hr for temperatures between -20 and -60 °C, and 600 L/hr below -60 °C.
- You must open the shim gas needle valve if the coil temperature drops below 5 °C. You can monitor coil temperature on the "Level" tab of the BSMS.

- Make sure Ice is not forming on the probe block or the bottom of the magnet!!!

Removal Procedure

- 1) Set temperature to 0 °C and wait for equilibration.
- 2) Turn off VT control, disconnect exchanger hose and reconnect VT gas to probe.
- 3) Turn VT control on and ensure it is set at 25 °C with 400 L/hr
- 4) Switch back to Air at the wall manifold after a stable temperature is reached.

(see next page)

High Temperatures: temperatures between room temperature and 150 °C can be achieved.

Must Remember:

- Ensure sample tubes and solvent are compatible with the temperature range desired
- Switch to nitrogen gas ~15 minutes prior to raising temperature above 35 °C
- Use only the Ceramic sample holder for
- Turn on shim gas for any temperature that rises above 80 °C
- The shim coil temperature must not rise above 80 °C (Check on the BSMS "level" tab).

Operation procedure

- 1) Double click on the VT temperature display at the bottom of the topspin screen.
- 2) Set your desired temperature and run self-tune.
- 3) move in 20 °C increments for temperatures above 80 °C and pay careful attention to the shim coil temperature.
- 4) Turn on the shim gas if you use a temperature above 80 °C. Monitor the temperature of the shim coil and temporarily discontinue use if the shim coil temperatures rise above 80 °C.