Variable Temperature Control for NMR

Introduction

This manual is to instruct NMR IMSERC users to do the Variable Temperature (VT) experiments on A600, Au400, and Hg400 (w/ RT probes) NMR spectrometers (-150 to +150 °C). In principle, one can follow similar procedure to use the HFCN600 (equipped with QCI-F cryoprobe, with much smaller VT range from -30 to 70 °C).

Depending on your target temperature, please consult with **fig 1** and **table 1, 2** to pick the right spinner, set up the gas flow rate, and the cooling power for the BCU II device.

When operating at elevated temperatures, internal probe heating is sufficient and precisely regulated with the new BSVT on Au400 and Hg400, which also controls (regulates) the selected VT flow rates and other auxiliary flows. Chillers are not required as long as the operating temperature is sufficiently above the room temperature. For A600, you needs manually adjustments of Shim/Flush gas. If you need temperatures below room temperature, chiller (BCU II or FTS) is required.

Always remember to redo the lock, tune, and shimming if the temperature changes more than 10 °C. The solvent boiling and/or frozen point must be considered. For high temperature, the highest temperature allowed should be at least 10 °C below the boiling point while for a low temperature experiment; the lowest temperature should be at least 5 °C above frozen point.

The real temperature and detected temperature may be slight different. Refer to the temperature calibration curve chart or calibrate temperature yourself. If you need a temperature below -60 °C, please ask NMR staff to use liquid nitrogen as a cooling source.

Operation Procedure

1) Select the right spinner based on **fig 1** and **table 1,** position and load your sample.
2) Open temperature control panel by typing command **edte**.
3) To change gas flow rate, click "**set**" under "Target Gas Flow" on BCU II (**fig 2**), put appropriate gas flow rate in the popup box. For FTS chiller, simple click on "**-**" or "**+**" to change the flow rate (**fig 3**).
4) To change the chiller power, for FTS chiller, set the desired temperature using the ▲▼arrow. For BCU II, click "**set**" under "Target Power" and select cooling power based on **table 2**.
5) To change temperature, click "**set**" under "Target Temperature" on BCU II (**fig 2**) to set your target. For FTS, click "**change**" button of "Target temp" row. Change the temperate by 10 °C increment and give around 5 minutes before doing next increment. Changing temperature too fast may damage the probe.
6) For FTS chiller, you can explicitly set the temperature. The maximum you can set is 60 °C. Please keep FTS at least 10 °C below your target temperature for above RT experiment. For extremely low experiment, you have to keep the FTS temperature 30 to 40 °C below your target.
7) Once the target temperature is regulated, please wait at least 5-10 minutes for the sample to reach equilibrium before you run your experiment. Remember to redo locking, tuning, and shimming for each different temperature.
8) Collect data.
9) After finishing, please restore the temperature setting back to default at 25 °C. Change flow rate and cooling power back to default.

Fig 1: Three types of spinners: Standard **POM** Spinner (blue, left), 0°C to +80°C; **Kel-F** Spinner (off white, middle) for elevated Temperatures, +80°C to +120°C; **Ceramics** Spinner (white, right) for high and low Temperatures, +120°C to +180°C and 0°C to -150°C (A600, Au400, and Hg400)

Table 1: The VT gas, shim gas, flush gas settings and recommended spinners for all 5 mm RT probes (P = POM ; K = Kel-F; C = Ceramics):

Sample T [°C]	-150…-80	-80…-50	-50…0	0...80	80...120	120...150
Spinner	C	C	K & C	P, K & C	K & C	C
Recom. VT gas [L/hour]	1200	1000	750	400 (P) 600 (K & C)	450	350
Shim gas [l/min]	20	20	20	0	0…20	20…60
Flush gas [l/min]	5…10	5	5	0	5	5
Chiller	LN2	LN2	BCU II (-50)			

Important:

1. The temperature of the shim system should always be -80 °C < T < 80 °C.
2. The temperature of the magnet flange (O-Ring!) has to be 3 ºC < T < 80 ºC, especially for long term experiments.
3. The shim system and the probe has to be flushed during low temperature experiments (sample temperature T < 0 °C) to prevent icing and condensed water.
4. The new BBFO smart probes on Au400 and Hg400 automatically adjust the Shim/Flush gas based on temperature setting. Only the A600 needs manually adjustments of Shim/Flush gas (ask NMR staff if you do not know how).
5. You should raise/lower the temperate incrementally by 10 °C for about every 5 minutes
6. Never turn VT gas off.

Table 2: BCU II cooling mode (off, low, medium, and strong)

25 °C to 150 °C	off
25°C down to 0°C	low
0°C to -20°C	medium
-20°C to -50°C	strong

Fig 2: Au400/Hg400 VT interface with BCU II (by type command “edte)

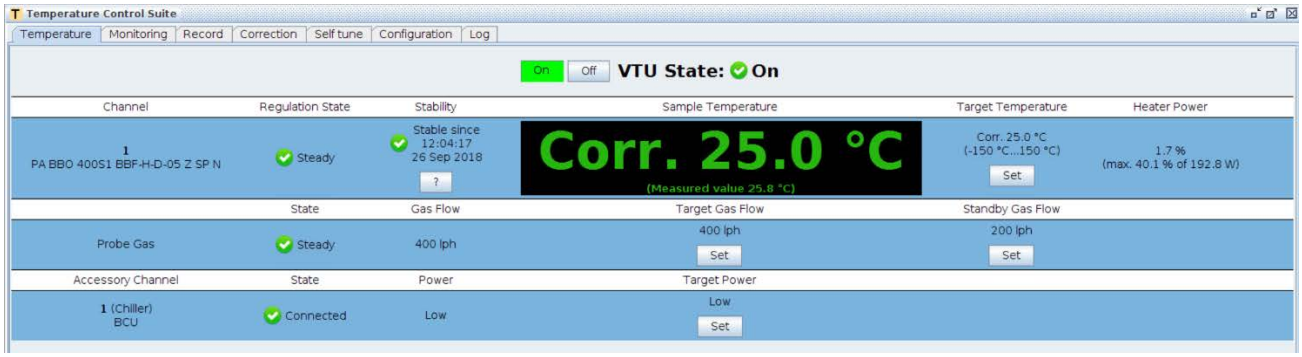

Fig 3: A600 VT interface with FTS (by type command “edte)