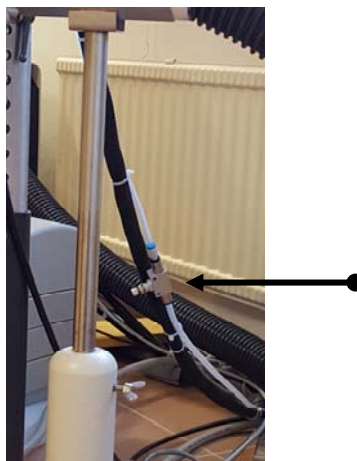


# Changing the temperature on the 500MHz

## A) To know before changing the temperature:

1. For the BBO and the BBI probes, the temperature range covers from -100 to +120 °C.
2. As a rule of thumb, you should be **at least 5 °C from the solvent boiling or freezing point**.
3. If you run below -50 °C or above 80 °C, type the AU-program **coiltemp** to follow the temperature over the shims. The temperature over the shims **must not go under 0 °C or above 80 °C**.  
The temperature over the shims will be displayed in the lower part of the screen as text.
4. If you run **below -50 °C or above 80 °C, you need to have airflow over the shims** depending on how long you will stay on that temperature. The magnet is equipped with a cooling ring set. On the backside of the NMR magnet, you can find a screw/needle valve (see the image) on the pressurized air-tube. You will hear some noise when the air flow is on.



5. **Use the white (ceramic) spinner** when running extreme temperature below -50 °C **or the beige (grey?) (plastic) spinner** when running extreme temperature above 50 °C. You can find them in the blue box on the console. Please, remember to put them back.
6. You need to change the mode on the **BCU II** (to the left of the 500 MHz) when running temperature higher than 60 °C or lower than -10 °C, to help the system to reach the temperature. **The default position is Remote**. See further instructions in section B.5 to heat up or section C.3 to cool down.

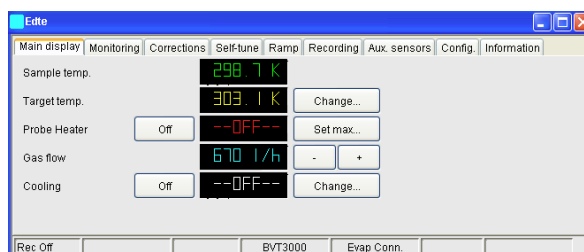
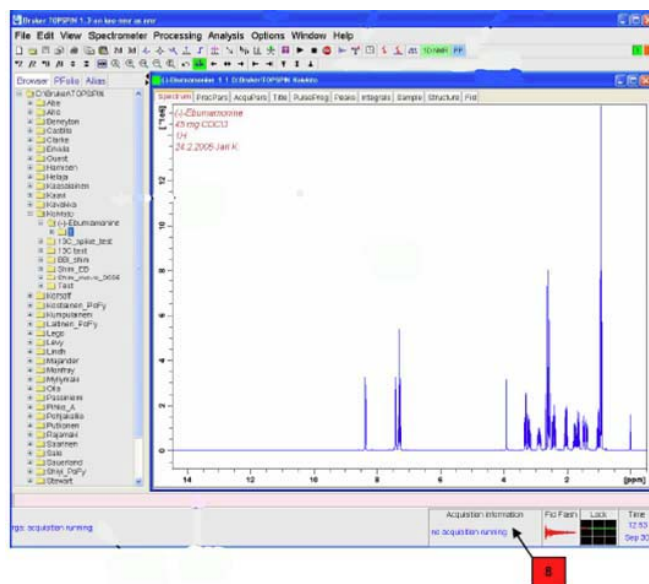


## Changing the temperature on the 500MHz

7. **Go up and down in ~20 °C to 25 °C steps.** Wait some minutes on every temperature level to prevent the glassware in the probe from breaking.
8. A temperature calibration may be needed. The set temperature is not the actual temperature; you can look in the folder for more close values.
9. First tune, match, lock and shim on a reference sample at 25 °C to see that everything is okay. You may want to do this procedure a few times when going down or up in temperature, since the tune, match, lock and shim values will change with temperature.

### B) How to change the temperature:

Open the temperature window by typing **edte** or by right-clicking on the temperature (VTU) in the Acquisition Status bar (no. 8 in the picture) below and chose “edte” to get the temperature window.



Under the “*main display*” tab, you can change “**target temp.**”, “**probe heater**” and “**gas flow**”.

Under the “*monitoring*” tab, you can follow the temperature.

## Changing the temperature on the 500MHz

### C) To heat up:

1. Use the **white** (ceramic) or the **beige** (plastic) **spinner** when the target temperature is **over 50 °C**.
2. In the temperature window (**edte**), under the “*main display*” tab, reduce the airflow to 400 L/h.
3. In the temperature window (**edte**), under the “*main display*” tab, change “**target temp.**” Remember to reach your desired sample temperature by heating up in **~20-25 °C steps**.
4. If you want to go **above 60 °C**, change the **mode to 0** on the **BCU II** unit (see section A.6).
5. When **running experiments above 80 °C**, type the AU-program **coiltemp**. The temperature over the shims **must not go over 75 °C**. Depending on how long you will stay on the temperature, **you need to have airflow over the shims** (see section A.4).
6. **Go up in ~20-25 °C steps** until reaching the target temperature. **Wait some minutes** on each temperature level so that the glassware in the probe doesn't break. Don't forget to tune, match, lock and shim some times on your way up.
7. Check that the sample temperature increases in the **edte** window at the bottom of the screen under the “*monitoring tab*”. If you do not reach the temperature, you may set a higher maximum at the “*probe heater*”.
8. A temperature calibration may be needed. The set temperature is not the actual temperature; you can look in the folder for more close values in the already made calibration curves. If you want a more exact actual temperature you need to calibrate.
9. After finishing your measurements, you must always **go back to 25 °C**.
  - a. Go down in ~20-25 °C steps.
  - b. If the mode on **BCU II** unit is changed, change **back to Remote mode**.
  - c. If you have **airflow over the shims**, close the needle valve.
  - d. Change to the **default gasflow**: 535 L/h.
  - e. If you have changed maximum for the “**probe heater**”, go back to 10%.

## Changing the temperature on the 500MHz

### D) To cool down:

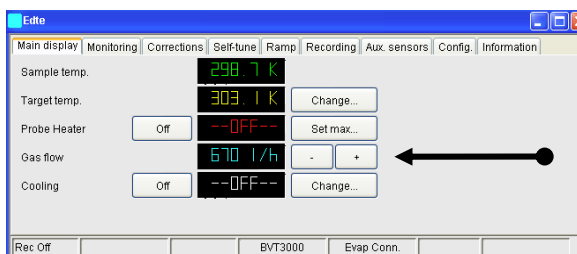
You can cool down to nearly  $-40^{\circ}\text{C}$  without changing to nitrogen cooling. If you need lower temperature than  $-40^{\circ}\text{C}$ , ask Kristina to show you how to do this.

1. Mind the airflow:

The airflow change depends on how low you want to measure. If the flow is too high, it can affect the spinner and the lock level jumps. If this happens, lower the flow or change to the ceramic spinner. Take into account that going down the last  $10\text{--}15^{\circ}\text{C}$  (to  $-38^{\circ}\text{C}$ ) consumes quite much time.

In the temperature window (**edte**) under the “*main display*” tab, increase the “**gas flow**”.

- Down to  $10^{\circ}\text{C}$ , use flow 535 L/h.
- Down to  $-10^{\circ}\text{C}$ , use flow 670 L/h.
- Down to  $-30^{\circ}\text{C}$ , use flow 800 L/h.
- Down to  $-38^{\circ}\text{C}$ , (actual  $-41^{\circ}\text{C}$ ), use flow 935 L/h.



- In the temperature window (**edte**) under the “*main display*” tab, change “sample temp”. Remember to reach your desired “**sample temp.**” by cooling in  **$20\text{--}25^{\circ}\text{C}$  steps**. Wait some minutes on every temperature level so that the glassware in the probe doesn’t break.
- If you go want to go **below  $-10^{\circ}\text{C}$**  change the **mode to 3** on the **BCU II** unit (section A.4).
- Don’t forget to tune, match, lock and shim some times on your way down.
- Check that the sample temperature drops in the **edte** window under the “*monitoring*” tab. If not, check that you have mode 3 on the **BCU II** unit or increase the “**gas flow**”.
- A temperature calibration may be needed. The set temperature is not the actual temperature; you can look in the folder for more close values in the already made calibration curves. If you want a more exact actual temperature you need to calibrate, see section E.
- After finishing your measurements, you must always **go back to  $25^{\circ}\text{C}$** .
  - Go up in  $\sim 20\text{--}25^{\circ}\text{C}$  steps.
  - If the mode on **BCU II** unit is changed, change **back to Remote mode**.
  - Change to the **default airflow**: 535 L/h.

# Changing the temperature on the 500MHz

## E) Temperature calibrations:

### Temperature calibration with a methanol-d<sub>4</sub> sample

1. **atma, lock, shim**
2. Run an ordinary proton (**ns=1, rga, zg, ef, apk**) on three temperatures covering the area you want to run in and make a curve out of the data.
3. You should see two peaks. Calculate the shift difference between the peaks.
4. Calculate the temperature according to the formula:

$$T (\text{Kelvin}) = -16.17467 * (\Delta \text{ in ppm})^2 - 52.5130 * (\Delta \text{ in ppm}) + 419.1381$$

### Temperature calibration with a 4% methanol-d<sub>4</sub> in a methanol sample

5. **atma, lock, shim**
6. Run an ordinary proton (**ns=1, rga, zg, ef, apk**) on three temperatures covering the area you want to run in and make a curve out of the data.
7. You should see two peaks.
8. Write **calctemp**, this AU-programme calculates temperatures down to -94°C.

Or

Write **pps**. Use the given shift values in ppm to calculate the actual temperature in Kelvins (0 °C = 273.15 K).

According to the following formula:

$$175-225\text{K } T = 537.4 - 2,380 * (\Delta \text{ in ppm}) * 60$$

$$-98\text{ °C} \rightarrow -48\text{ °C}$$

$$220-270\text{K } T = 498.4 - 2.083 * (\Delta \text{ in ppm}) * 60$$

$$-53\text{ °C} \rightarrow -3\text{ °C}$$

$$265-313\text{K } T = 468.1 - 1.810 * (\Delta \text{ in ppm}) * 60$$

$$-8\text{ °C} \rightarrow 40\text{ °C}$$