## Low Temperature NMR Experiments in the 500MHz

USE THE CERAMIC SPINNERS FOR ALL VARIABLE TEMPERATURE EXPERIMENTS!

The lower limit for cooling is -50°C with consideration of the melting point of the solvent.

Never leave the NMR room for more than 20 minute intervals while conducting low temperature NMR experiments.

How to set up a low temperature experiment:

1. In the command line of TopSpin software interface, type "edte" to start the Temperature Control Suite
2. Click on OFF button to turn off the Variable Temperature Unit (VTU):
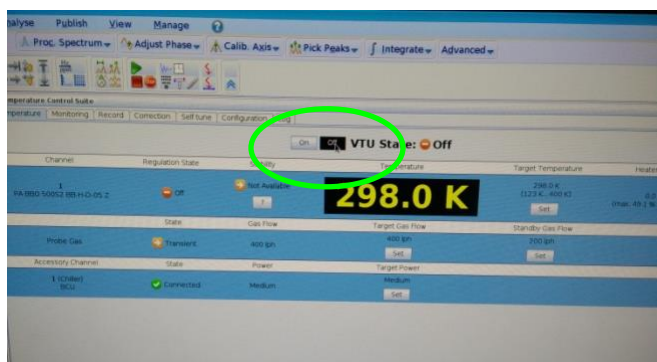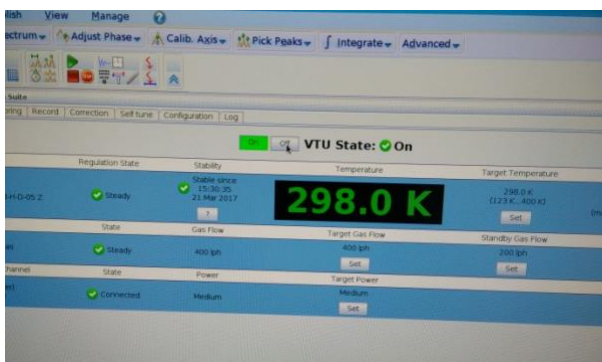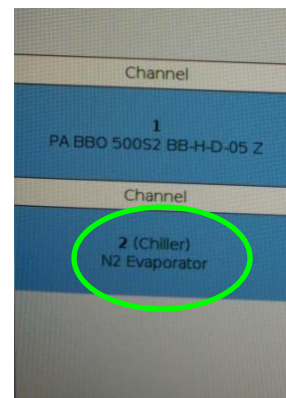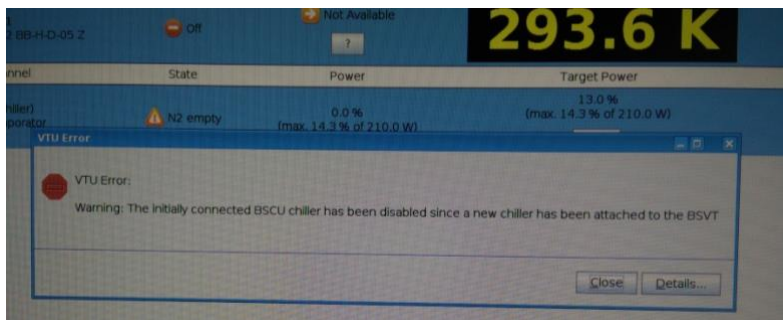

3. Fill the liquid nitrogen dewar.
4. Disconnect the black VT gas hose from the probe:

5. Connect the nitrogen transfer line cable to the LN2 box adapter (hidden under the black sponge to protect it from the humidity)
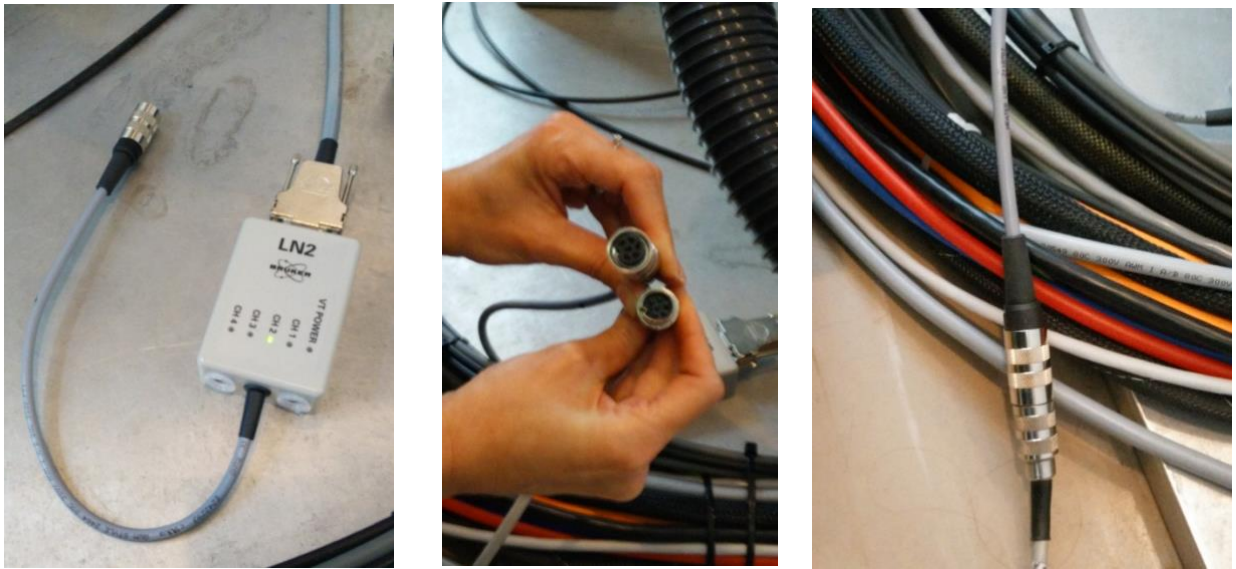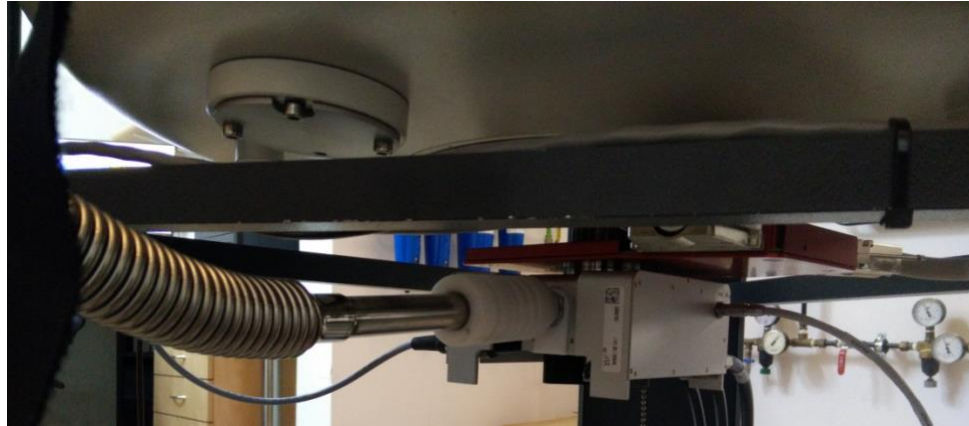

At this point, the Temperature Control Suite shows the Chiller N2 Evaporator added to the channel 2 as shown in the Temperature Control Suite:


6. Make sure the o-ring is on the heater end of the nitrogen transfer line and insert the heater of the nitrogen gas transfer line into the LN2 dewar SLOWLY. After the transfer line is in the dewar, use a clamp to seal the heater on the top of dewar. The clamp must be screwed down tightly using a butterfly nut.

7. Attach the transfer line to the probe. Keep the transfer line horizontal when connecting the transfer line to the probe.


8. Click on self-tune Tab of the Temperature Control Suite, select a file that suitable to the target temperature and click on "restore to channel 1" to load the parameters.
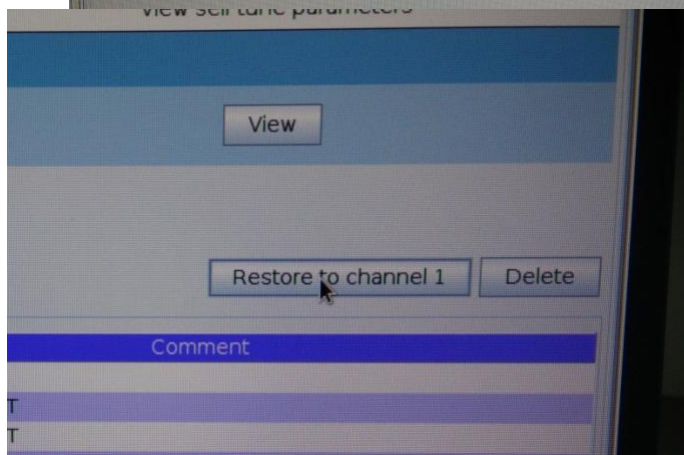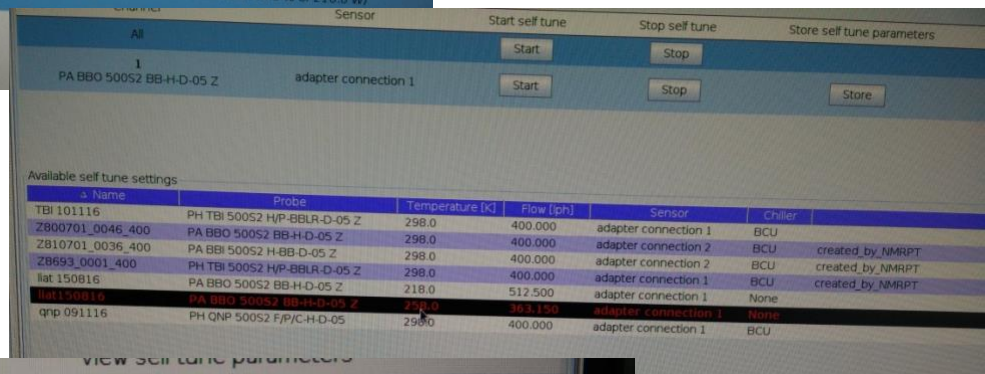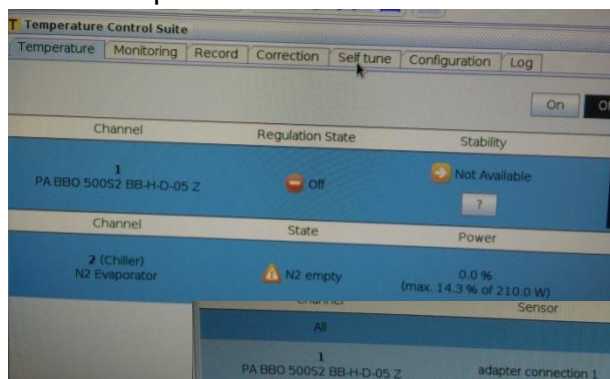
9. In Temperature Tab and in Target Temperature column, click Set to set a sample temperature in Kelvin. Click on OK to finish setting the sample target temperature and then click on ON button at the top row of the Temperature Tab to turn on the VTU.
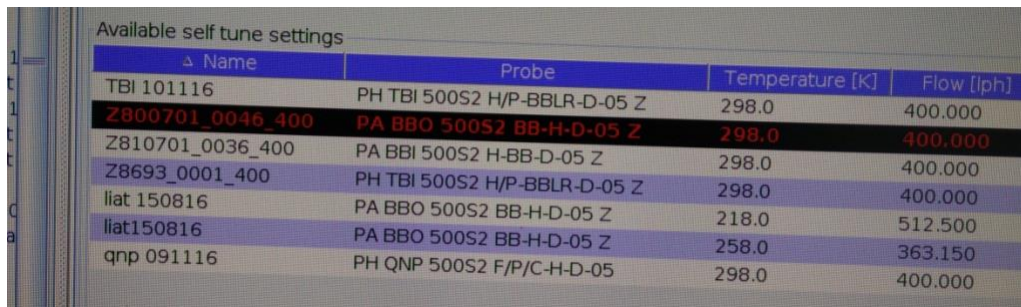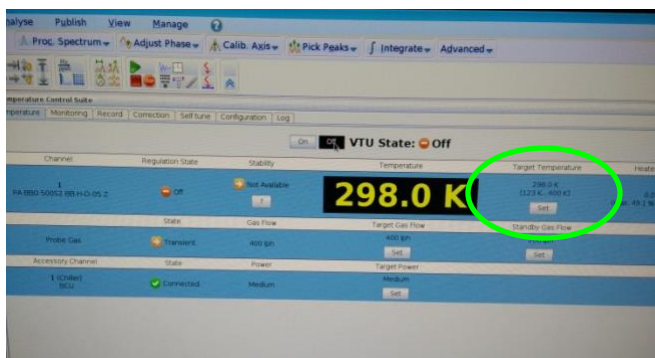

10. Click on Monitoring Tab and check the boxes of Current Temperature and Target Temperature. The current sample temperature slowly approaches the target temperature. To prevent a decrease of the shim coil temperature the external gas flow valve must be opened.

11. A methanol sample is used for low temperature calibration. Use the calibration curve to determine the actual temperature.
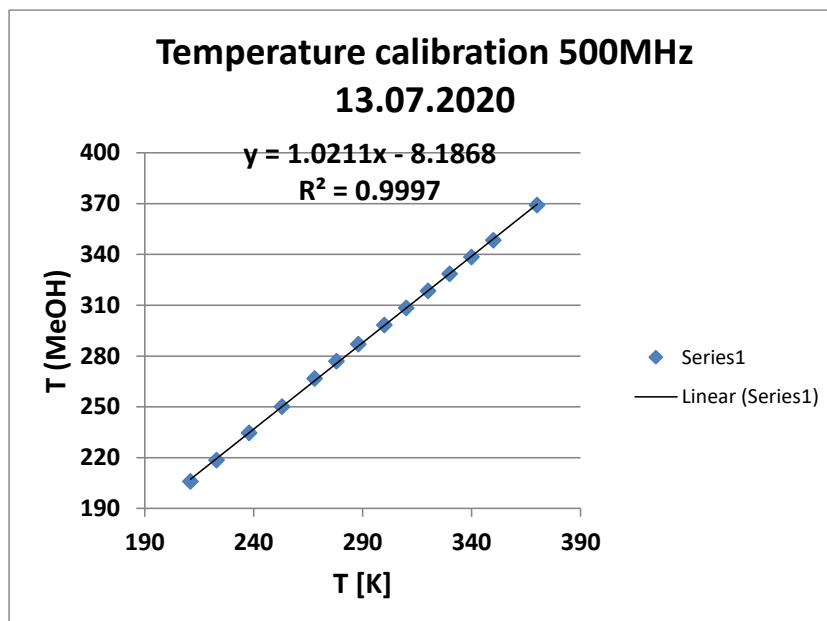
12. When the low temperature measurements have completed increase the temperature back to 298 K gradually (don't need to stabilize the temperature), take out the sample, turn off the VTU unit (click on OFF),

detach the transfer line from the probe and connect the black VT gas hose, disconnect the nitrogen transfer line cable from the LN2 box adapter, take out the transfer line from the dewar, close the external gas flow valve, click on Self Tune Tab of the Temperature Control Suite, select the self-tune setting file:

△ Name	Probe	Temperature [K]	Flow [lph]
TBI 101116	PH TBI 500S2 H/P-BBLR-D-05 Z	298.0	400.000
Z800701_0046_400	PA BBO 500S2 BB-H-D-05 Z	298.0	400.000
Z810701_0036_400	PA BBI 500S2 H-BB-D-05 Z	298.0	400.000
Z8693_0001_400	PH TBI 500S2 H/P-BBLR-D-05 Z	298.0	400.000
liat 150816	PA BBO 500S2 BB-H-D-05 Z	218.0	512.500
liat150816	PA BBO 500S2 BB-H-D-05 Z	258.0	363.150
qnp 091116	PH QNP 500S2 F/P/C-H-D-05	298.0	400.000

Available self tune settings

then click on Restore to Channel 1 button to load the self-tune parameter for room temperature operation. Click on ON button in the Temperature Tab of the Temperature Control Suite to turn on the VTU. Wait until the target temperature reaches and stabilizes at 298 K.