Cryoprobe Guide

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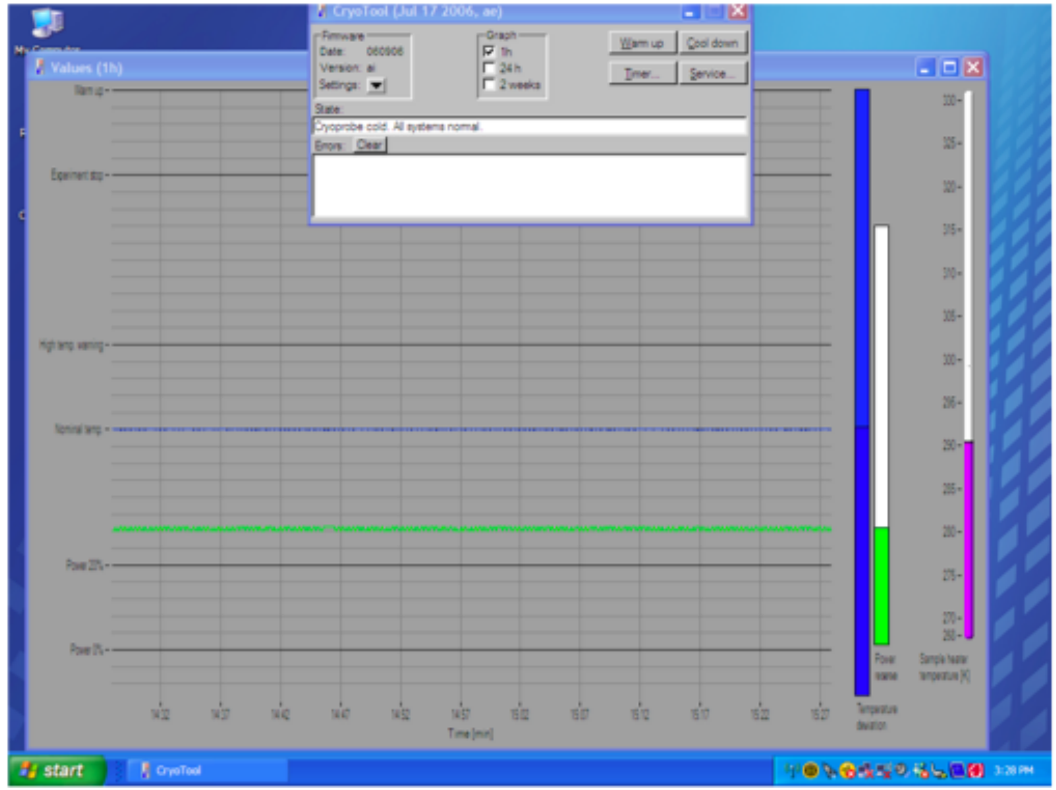
Normal operation

*The cryoprobe must be monitored whenever an experiment is started!



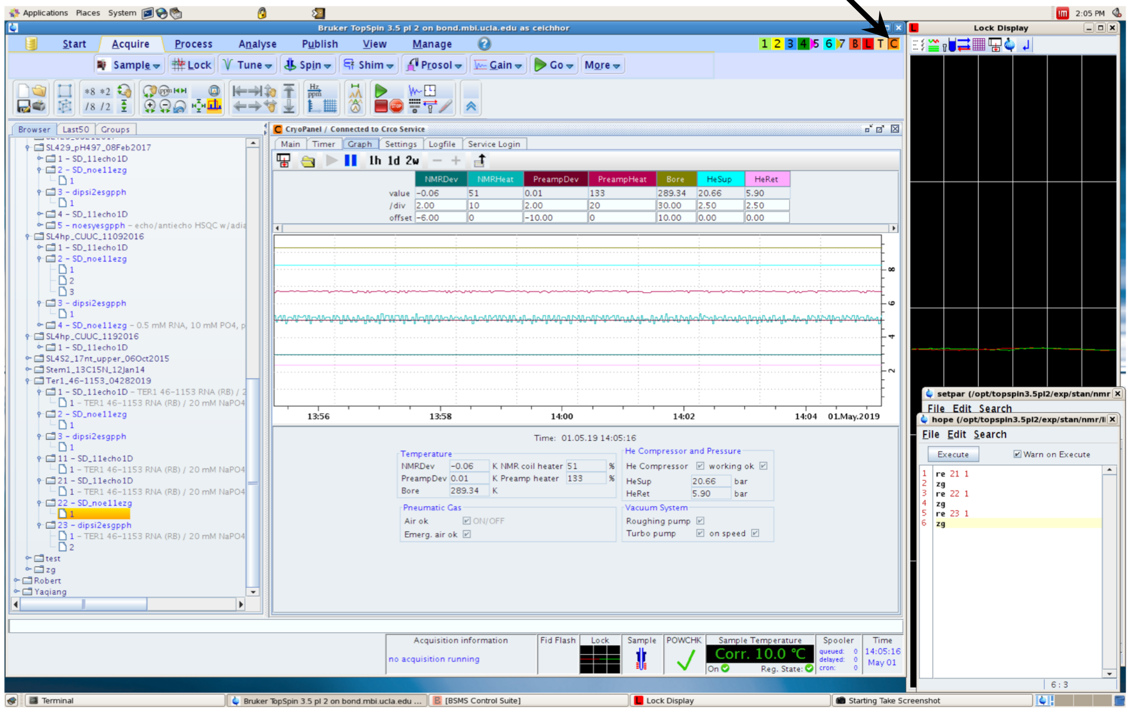
On Mulder (500), the monitoring software (cryotool) is on the cryo-laptop which sits on top of the cryoplatform. You must remember to look at the display when starting experiments.

In the CryoTool window, there are three graphs normally available - 1hour, 24 hours, and 2 weeks. In the 1 hour graph there is a blue line that indicates the current temperature of the coil. Ordinarily this will be on the ‘nominal temperature’ line. There are also lines at 5, 15, and 20 grid units above the ‘optimal temperature’ line called ‘high temp. warning’, ‘experiment stop’, and ‘warm up’.

The maximum average power can easily be exceeded by long pulses or pulse trains. If running your experiment causes the coil temperature to rise at all, you should stop it and check all pulses and pulse trains.

Normal operation

On Bond (600), the monitoring software is in topspin. You can start it by typing cryopanel. You can bring it to the front quickly by clicking the orange C at the top right. Or you can have it as a separate window.



You must remember to look at the display when starting experiments. Have the "Graph" tab open and pay attention to "NMRDev" and "NMRHeat".

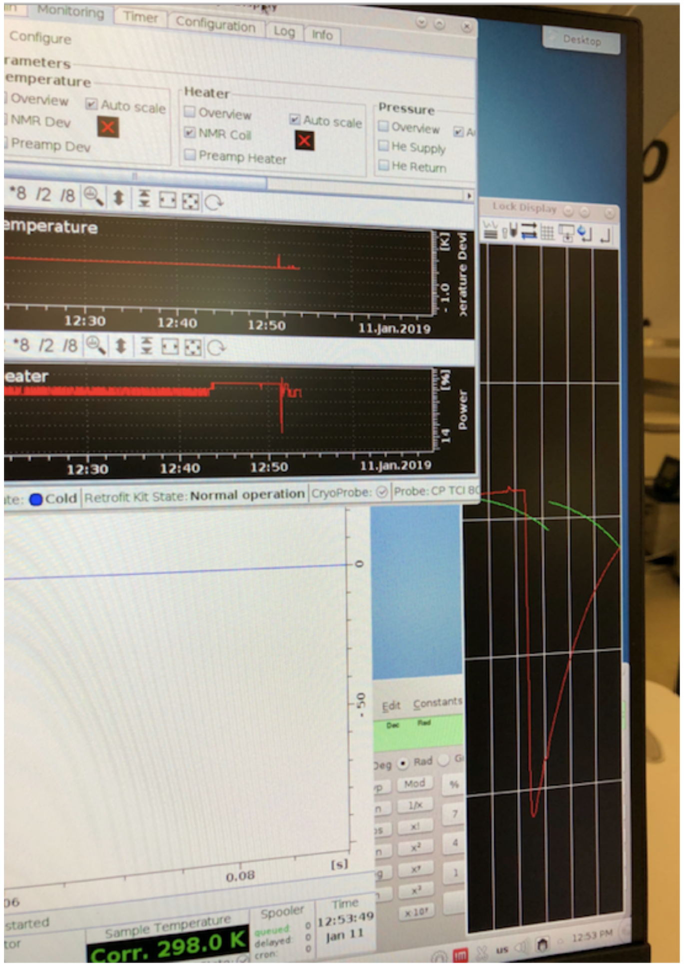
The maximum average power can easily be exceeded by long pulses or pulse trains. If running your experiment causes the coil temperature (NMRDev) to rise quickly, or makes NMRHeat drop to zero, you should stop it and check all pulses and pulse trains.

Normal operation

On Bosch (800), the monitoring software is in topspin. You can start it by typing cryopanel or cryodisp.

The default is to display it as a separate window. In the window, go into the “Monitoring” tab and click “NMR dev” under Temperature, and “NMR coil” under Heater. Then scroll using the mouse to get the scale to a reasonable level. Make sure the window is visible as shown in the picture.

The maximum average power can easily be exceeded by long pulses or pulse trains. If running your experiment causes the coil temperature (NMR dev) to rise quickly, or makes NMR coil drop to zero, you should stop it and check all pulses and pulse trains.



For all spectrometers: if the coil warms quickly and/or the heater power drops to zero, but none of the power levels exceeds the maximum allowed power level, stop the experiment. Check all pulse trains, such as TOCSY and CPD sequences. If any pulses (including shaped pulses) have long durations (msec range), check the power levels.

Any problems that occur will generate an error message in the software. The software is very good and it will automatically handle any problems so as to prevent damage to the system.

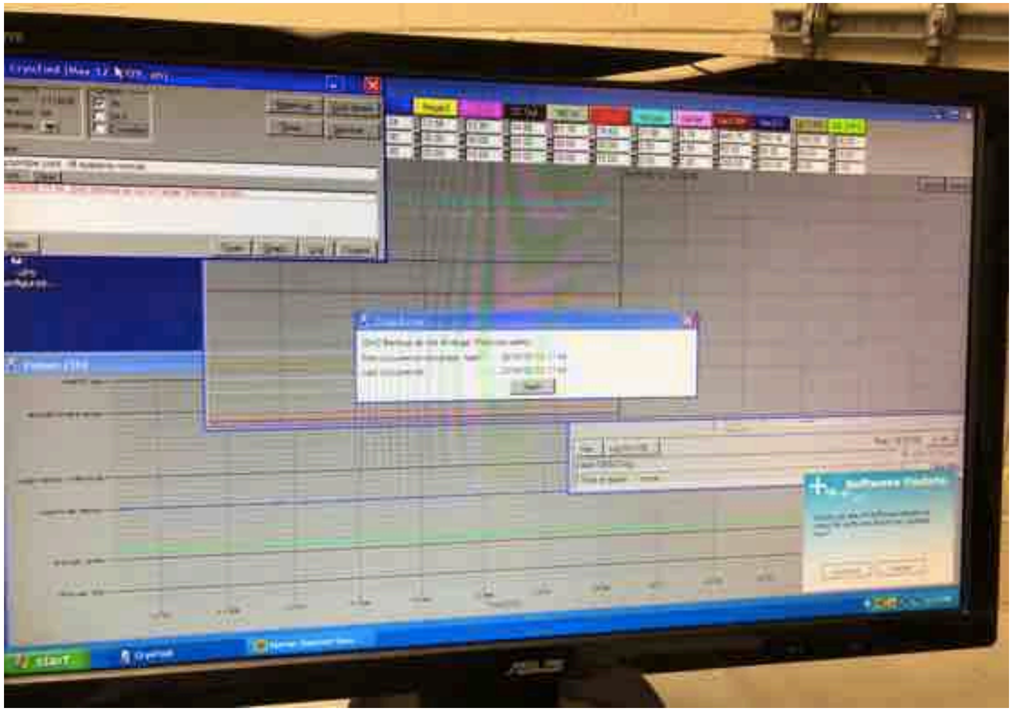
The overall system status can be seen in the main window of the software, or by looking at the lights on the front of the cryoplatform. If there's any type of error at all, the red "error" light will be on. If the system is cold, the green "cold" light will be lit, and if it's warm, the "warm" and "unplug" lights will both be on. If the system is warming up or cooling down, the "warm" or "cold" light will be flashing.

To initiate a warmup, press the "Warm up" button on the front of the cryoplatform. To initiate a cooldown, press the "Cool down" button. You can also click "warm up" or "cool down" in the cryotool or cryopanel window. *see page on no-flush cooldown.

There are many errors that can occur. Some of the errors are inconsequential ("pressure drop in system" for instance). If there is an error but the system is still cold, try to get assistance, but it's usually safe to leave it alone.

One error that's very common on Mulder (500) is shown here:

"Backup air out of range", or similar errors pop up often – usually several times a day. They don't seem to mean anything, so don't worry if you see them. They can be cleared from the software by clicking "seen" and "clear". And they can be cleared from the cryoplatform by pushing and holding the "Cooldown" button.



Unfortunately the system often warms up because of supposed problems with the pressure of the backup air. If you find the system warm, and the error messages say "main air below limit", or something similar. Try just cooling it down again.

*(see page on how to do a no-flush cooldown)

RF power

RF coil maximum power

The RF coil can easily be damaged by excessive pulse power.

Bond and Bosch have software controls on the power so it's not possible to exceed the maximum allowed power. However, it is possible to exceed the maximum average power.

The maximum allowed power level on each channel has also been set by the installation engineer. This power appears in the edprosol table, and on the pulse calibration list.

Here are representative maximum power levels and 90º pulse lengths:

^{1}H	8μsec	-5dB
^{13}C	15μsec	-5.5dB
^{15}N	40μsec	-5.2dB

This means you should never exceed these power levels. For example, you should never use -6dB on the carbon channel.

There are small books labeled **Typical Pulses for the 5mm CryoProbe** on each console. They contain typical allowed combinations of power levels and lengths.

For each nucleus it contains information like this:

^{13}C	
Hard pulse (max length 360º)	15μsec
CC spin lock	20 msec @ 25μsec
GARP4 decoupling	140 msec @ 65μsec

Here is what it means, taking the GARP4 decoupling as an example:

It's OK to apply GARP decoupling on ^{13}C for 140 msec, using the power necessary for a 55 μsec 90º pulse. So if the power for a 55 μsec 90º pulse on ^{13}C is -6.33dB, then you could set pcpd2=55 μsec, and pl12=-6.33dB. Then if GARP4 decoupling was set during acquisition, your acquisition time could be up to 250 msec long.

*The limits are different for each spectrometer. Check the typical pulses guide.

The **Typical Pulses** book gives examples of allowed power levels and durations that are meant as a guide. If you need different power levels and/or lengths than are listed there, you can interpolate to some extent (i.e. apply GARP decoupling for a longer time with less power/longer pulses). **Be very conservative if you need to use higher power levels than listed** (and of course never exceed the maximum power). If you're not sure, ask me.

Miscellaneous:

Total recycle delay should be 1 second or longer if you're anywhere even close to any of the maximum or average power limits.

If you apply simultaneous ^{13}C and ^{15}N hard pulses, you must reduce the power of both by 3dB. Of course you must also adjust the pulse length. For example:

Max.	length	power	reduced length	power
^{13}C	15μsec	-5.5dB	21.2μsec	-2.5dB
^{15}N	40μsec	-5.2dB	56.5μsec	-2.2dB

The same is true for simultaneous ^{13}C and ^{15}N decoupling (see next page).

The most common way that people exceed the maximum average power is by applying decoupling for too long. This happens because decoupling is applied during acquisition, and the acquisition time is too long.

In general, you should never decouple for longer than about 150 msec (or 250 msec on the new probes. Before starting your experiment, type aq. If it's longer than 150/250 msec, you must decrease it (or decrease TD such that aq is less than 150/250 msec). Verify that aq is less than 150/250msec before starting the experiment.

*If you're decoupling on two channels at the same time, then you must reduce the decoupling power.

As a general rule, you can use pcpd2=100μsec (^{13}C) and pcpd3=200μsec (^{15}N), with the appropriate power levels.

Another option to reduce the decoupling power is to use **adiabatic decoupling**. To use adiabatic decoupling on ^{13}C, in eda set CPDPRG2 to "p5m4sp180". Then set pcpd2=1.5msec and set spnam15 to "CHIRP95". The power level for this decoupling sequence is sp15.

sp15 should be set to pl12+2dB. ← This means 2dB higher than the power normally used for GARP decoupling with pcpd2=65μsec.

Adiabatic decoupling has the dual advantages that it uses less power and it decouples a larger bandwidth.

If the coil overheats and a warmup cycle starts accidentally:

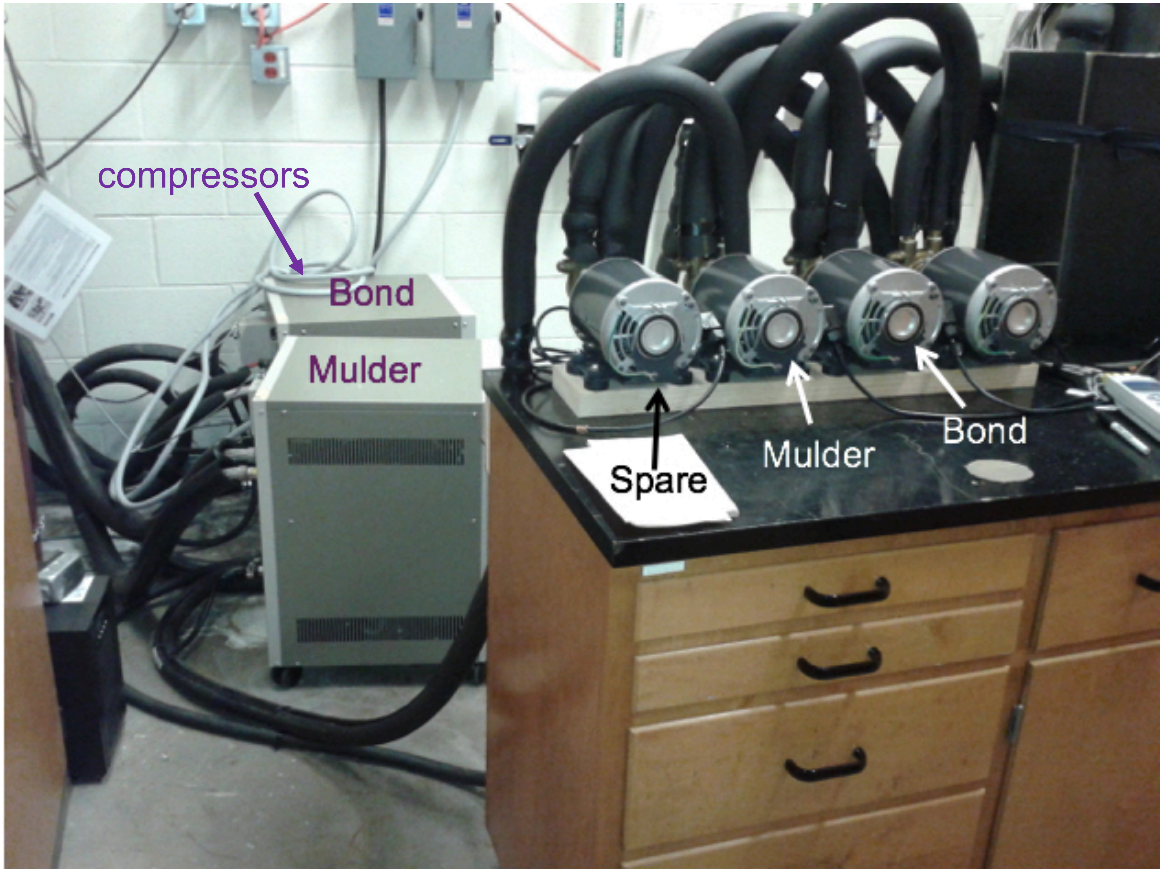
Immediately stop the experiment then push the "Cool down" button on the front of the CryoPlatform. If you do this quickly enough, it will abort the warm up cycle and cool back down.

Chilled water problems

Our helium compressors are water-cooled. They are cooled by heat exchangers which themselves are cooled by our building chilled water. One very common thing that causes a warmup is that there's a problem with the chilled water and the helium compressor overheats. This usually results in the error message "Trouble with compressor".

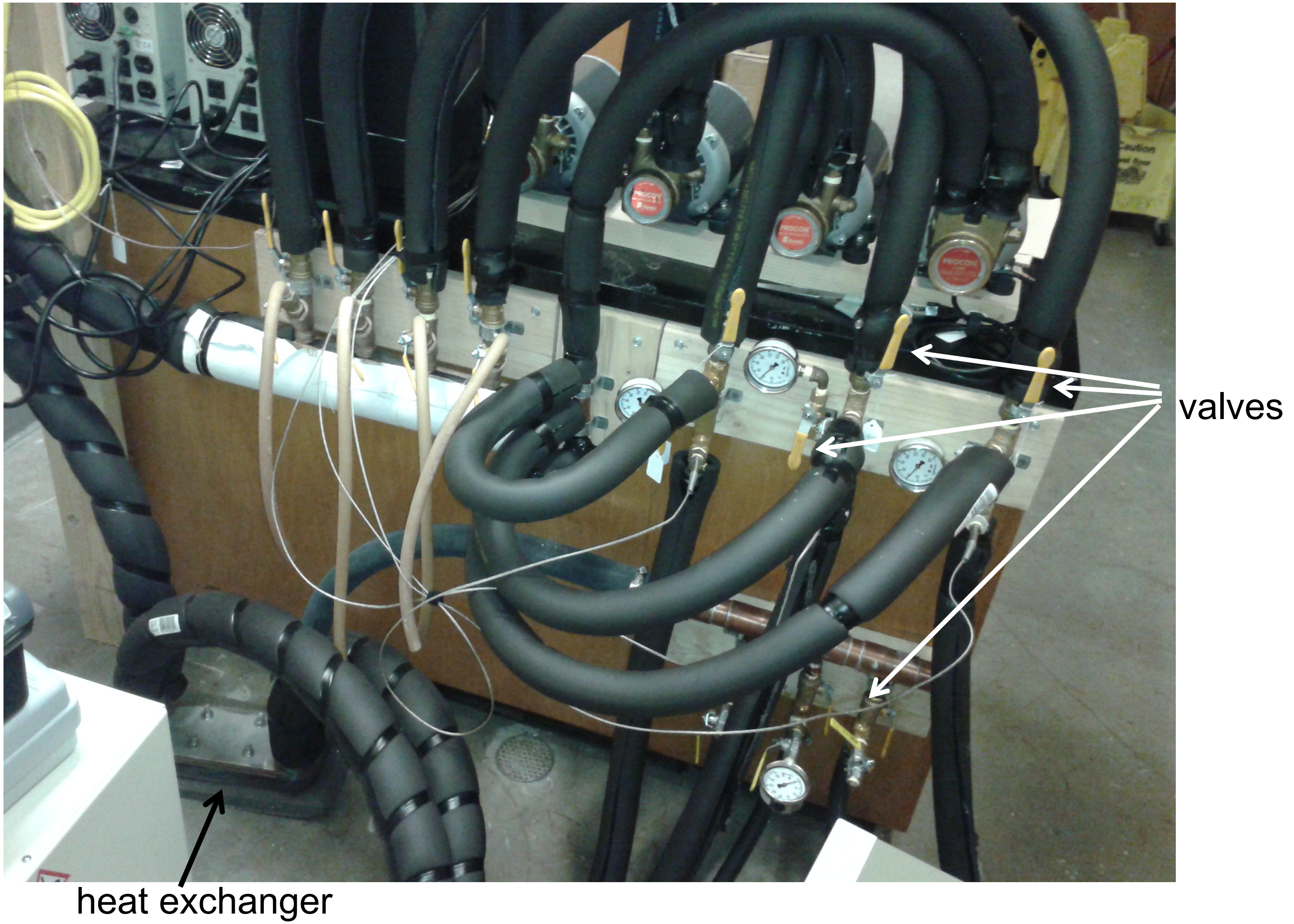
Bond's and Mulder's cryoprobes (as well as the departmental cryoprobe) are cooled by a home-built system:

This is located in the small service room right next to Mulder (MSB 1425).



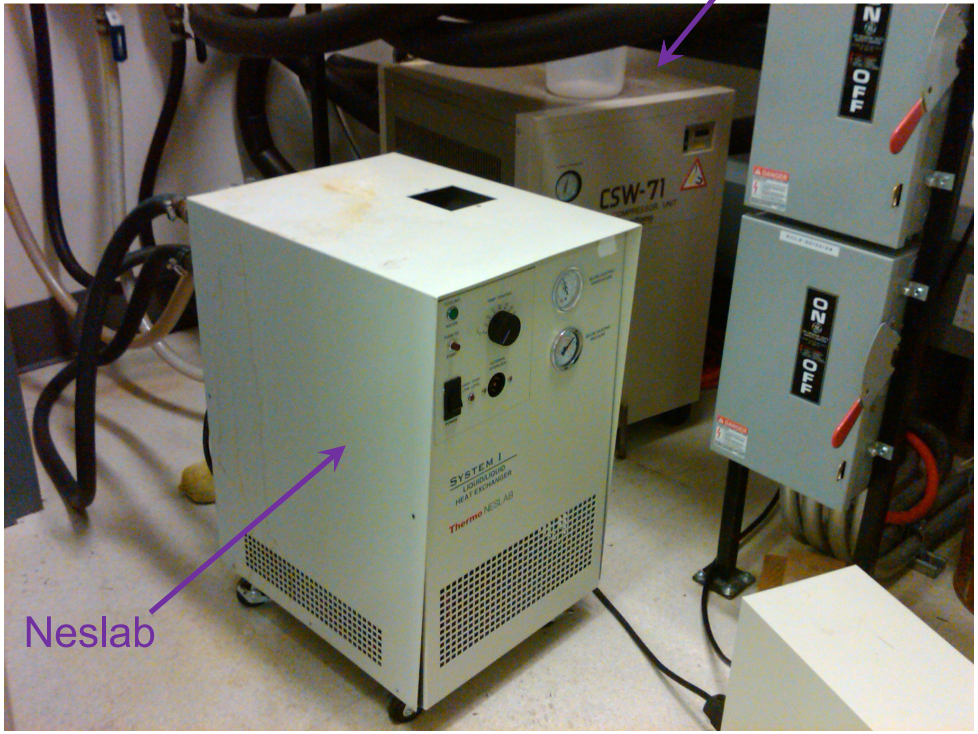
Chilled water problems

The pumps pump water from one common reservoir into the three separate compressors and then into a heat exchanger. The pumps are cleverly plumbed together so that the spare pump can be used for any of the three compressors:



Chilled water problems

Bosch's cryoprobe is cooled by a small Neslab water-to-water heat exchanger located in the closet in the 800 room:

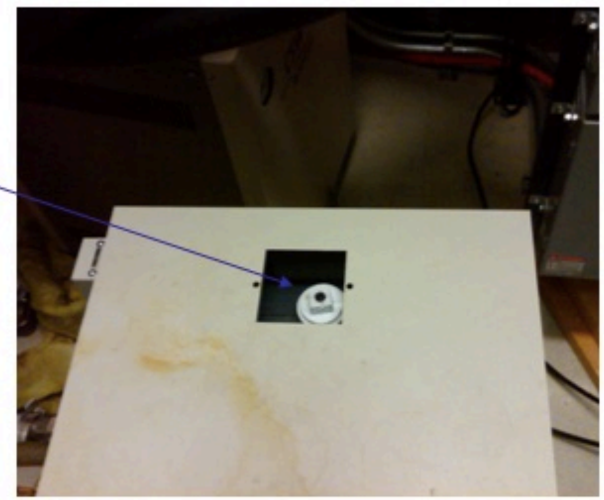


Chilled water problems

To turn it on, you have to first turn on the power.

Then push the red button marked “push to start”.

Refill water reservoir here:



*Problems with the chilled water are often easily solved.

800: check whether the Neslab is on, and whether the water reservoir is filled.

500/600: check whether the appropriate pumps are running and whether the water reservoir is filled.

Chilled water problems

In the past, the most common problem with the chilled water system was the building chilled water itself becoming too warm.

The temperature of the building chilled water can be monitored from the thermometer attached to the "Chilled Water Supply" pipe in MSB1425 (the room right next to Mulder).

Since the building heat exchanger was replaced and new electronic controls were installed, this thermometer always reads ~53º F.

If this temperature is too high (above ~65º F), the water to the compressors will be too hot and they won't stay on.

So if the chilled water is hot, don't attempt to cool the cryoprobes.

If the laptop crashes (Mulder/500 only)
Don't worry – it won't affect the cryoplatform. Just restart the laptop (the username is CryoUser and there's no password). Then double click the CryoTool_AG icon and select com port 6.

CryoPlatform
There is an emergency off switch on the front of the CryoPlatform. In the event of an emergency, this switch can be turned to the off position. This will return all valves to their default position and initiate a passive warmup.

*If the switch is accidentally turned to the off position (which can happen if you just brush against it), just turn it back to the on position.

Miscellaneous
The maximum sample depth is 21mm on all cryoprobes.

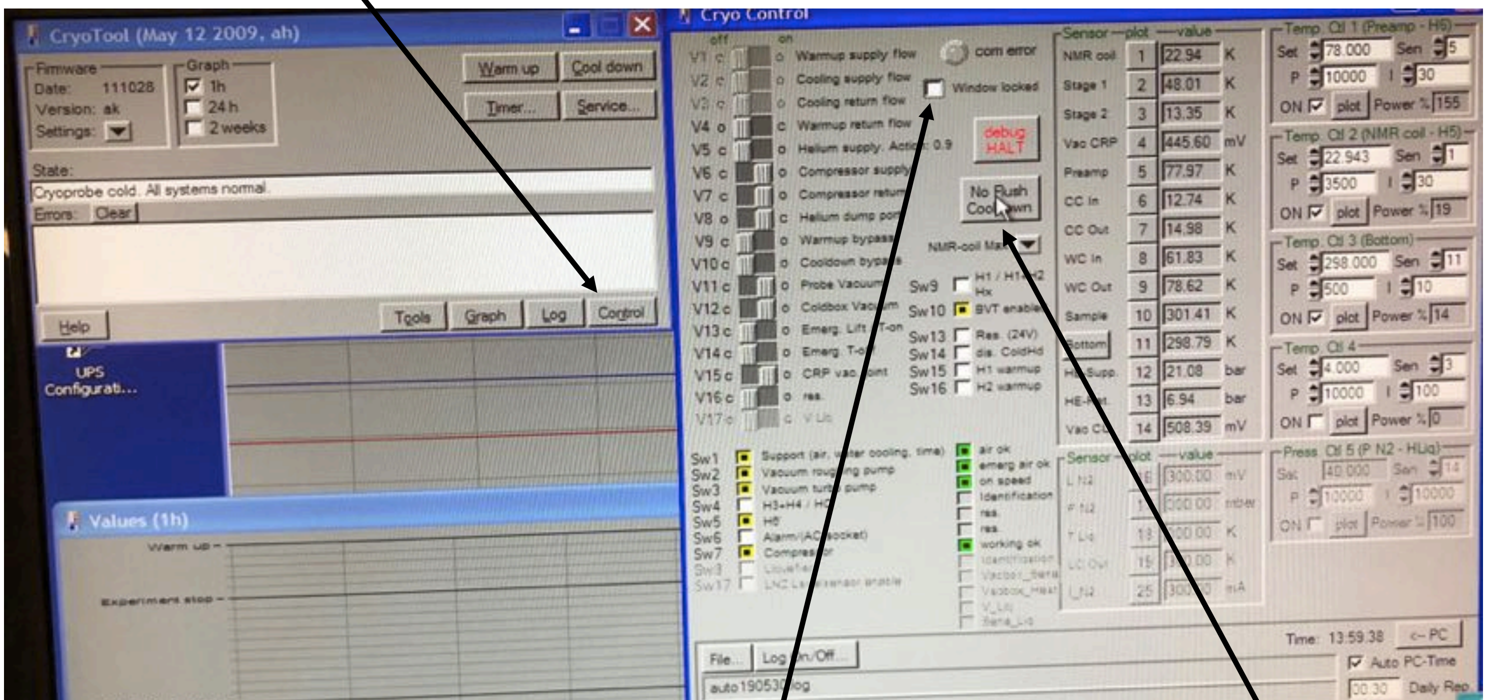
The command loopadj can be helpful in stabilizing the lock signal.

The CryoProbes are only equipped with Z gradients. Be sure to only use Z gradients in your experiments. If you use X or Y gradients, there won't be any error messages - the gradients just won't work.

No flush cooldown

In a normal cooldown, the system performs a series of helium flushes. It is not necessary to do these often, so to save helium, you should perform a "no flush cooldown".

To do this, first bring up the control panel by pressing "Control" in the main window.



Then in the control panel, press "Window locked", then press "No Flush Cooldown". After you've started the cooldown, press the "Window locked" button again to lock the window.

***Don't touch anything else in the control window! You can destroy the system.

<u>Cryoprobe checklist</u>

1. Maximum sample depth = 21mm on all cryoprobes. Use the white or blue plastic spinners. Do not use ceramic spinners.

2. Don't use the black cap.

3. EDTE: air flow=670L/hour, heater should be on at all times.

4. Do not exceed any of the maximum power levels. Generally, the power corresponding to 8μsec (^{1}H), 15μsec (^{13}C), and 40μsec (^{15}N) 90º pulses is the maximum allowed power (8/12/35 on newer probes). Simultaneous ^{13}C and ^{15}N pulses requires that the power for each be dropped by 3dB (and pulse lengths recalculated).

5. Do not exceed the maximum average power. Acquisition time (aq) should not exceed 150msec if decoupling is used (250msec on newer probes). Consult the *Typical pulses for 5mm CryoProbe* guides.

6. Use only Z gradients. All cryoprobes have only Z gradients. 3D gradient shimming can be done on Bosch and Mulder, but not Bond.

7. **Monitor the sample temperature** when starting any experiment. Mulder: the blue line (RF coil temp) in the 24 hour graph on the laptop should not move above the "nominal temp." line. Bond/Bosch: watch NMRDev and the NMR heater power. If the RF coil temp rises, stop the experiment and check all parameters.