

Notes on using the Bruker AC-200- TECMAG 200MHz NMR



Precautions:

WARNING!!!! Remove your wallet, watches, etc. before approaching the magnet! Never bring any ferromagnetic items into the magnet room. Be careful with metal objects in the room. Avoid taking your credit or ATM cards too close to the magnet (approx. 2 feet). **No loose staples or paper clips allowed.** People with medical implants should not approach the magnet. People with pacemakers are not allowed in the room. You must be trained either by the class instructor or by an NMR staff member.

This instrument is also known as the Bruker AC200. It has a Tecmag DSPECT hardware-computer interface module. The NTNMR software is run under Windows-NT. The magnet strength is 4.7 Tesla with a ^1H Larmor frequency of 200MHz.

Sample Preparation in the NMR room:

At the NMR console you will find the spinner (a plastic collar) that fits snugly over the NMR tube. Slide the spinner over the NMR tube to the appropriate place using the depth gauge (also located near the console). (Note: Be careful not to get oil from your fingertips on the NMR tube or spinner. Use a Chemwipe to clean the outside of the tube before inserting into the magnet.)

Insert the sample tube in the spinner, use the depth gauge to adjust the depth of the NMR tube in the spinner



Spinner on an NMR tube. The placement is too high. No signal would be seen.



Use a depth gauge to determine where the spinner should be.



Approximate height of spinner on an NMR tube. Note that the person is not touching the spinner.

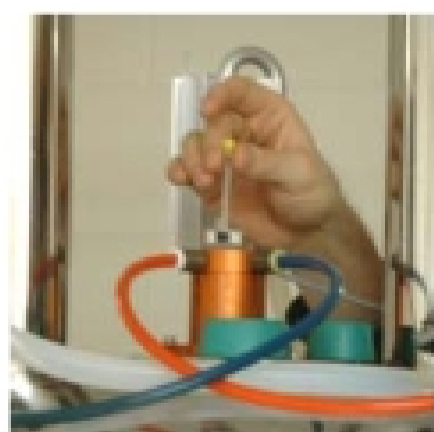
Collecting the Data:

The instrument is in room 2407. There are 7 parts you should be able to identify before acquiring your data. There are two monitors. The one on the right is connected to the computer. The one on the left is connected to the instrument console and will be used to monitor the deuterium lock signal. There are two keyboards. There is a standard QWERTY keyboard to operate the software. The other is to the right and is used to insert the sample and to lock and shim your samples. It will be referred to as the SCM keyboard. The monitors and keyboards are on the instrument console. While it looks like a big metal box, it contains all the electronics (amplifiers, receiver, etc.) for the instrument. On the front, left-hand side of the console, there is a small black button. It is used to stop spinning of the sample at the end of your acquisitions. Finally, the magnet is the big silver object in the back of the room.

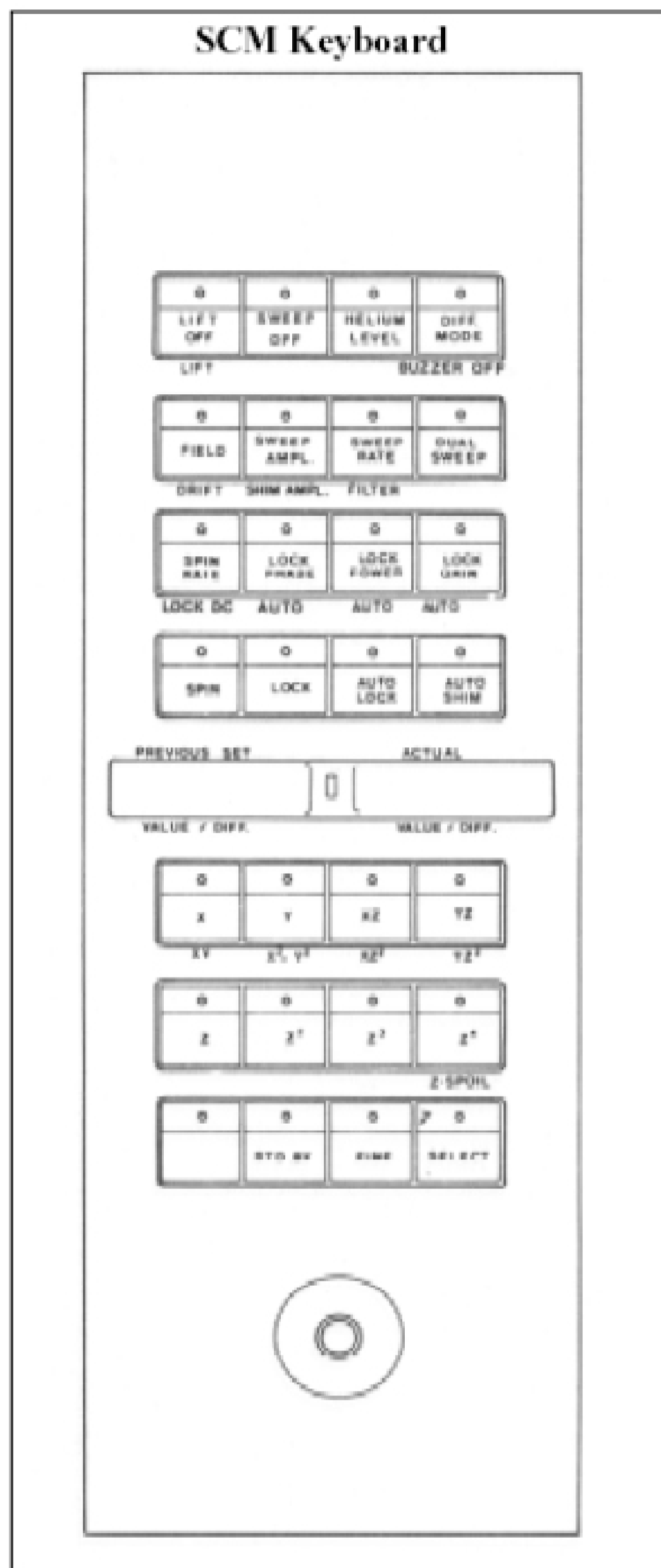
(If you want to know more about the constituent parts of the magnet, check out this part of the JEOL website: http://www.jeol.com/nmr/mag_view/magnet_destruction.html)

On the computer keyboard, press Ctrl-Alt-Del simultaneously, then login.

- On the computer keyboard, press Ctrl-Alt-Del simultaneously, then login.
- On the SCM (NMR) keyboard, press the orange button, then press the **Lift** button to turn the lift air on. Put the sample on the column of air on top of the magnet. Press the **Lift off**



button to lower the sample into the magnet.



Inserting the sample

Once the sample has been properly inserted into the magnet the normal procedure is to perform three steps to achieve optimum magnet homogeneity 1) spinning the NMR tube, 2) set the deuterium lock parameters, 3) optimize the shims currents

- Press **Spin** button to spin the sample. The light on the **Spin** button should turn on.
- On the NTNMR software window click on the **Console** button to open the Console Toolbar window, Click on **load**, select **standard.shm** file, then Click **Open**.
- This will load the standard shim set on the SCM keyboard.
- Look at the computer screen on the left. A deuterium signal should appear in the center of the screen. If not, press **Lock Power** on the SCM keyboard and set the value between 30 and 35 by turning the knob on the keyboard. (The range of 30-35 is appropriate for CDCl₃ solvent; for Acetone-d₆ use 10-15).
- Press **Lock Gain** and adjust the value between 100-104.
- Press the **Lock Phase** button and adjust the value to 253 (This value may change in the future; look for a note on the computer with the current value).
- Press **Field** and adjust the field value till the deuterium signal appears in the middle of the window. (January 17, 2003: Field = 4200 for CDCl₃, Field = 4010 for Acetone-d₆).
- Press **Lock** (the deuterium signal will disappear and the light on the **Sweep OFF** button will turn on). The **Lock** button light will blink.
- If the lock light is blinking, Press **Field** and slowly adjust the field until the lock light stops blinking. (Generally, for a solid lock the line should be over one grid line above the center).
- Now shim the magnet.

Shimming Procedure:

Check to make sure that the **Fine** button has a green light indicating it is engaged. As when locking, try to maximize the lock signal level by selecting a shim key (see below) and very slowly adjusting the knob at the bottom of the SCM keyboard.

- If you have difficulty shimming, try reading the latest stored shim file (ask GSI).
- If the lock level disappears off the top of the screen, Press **Lock Power** and lower the current value until the lock level is one to two grids below the top.
- Press **Z** and adjust the value in whichever direction increases the lock level.