TUNING AND MATCHING ¹³C AND ¹H ON THE BROADBAND DIRECT PROBE ON THE BRUKER DRX 400

http://nmr.gmu.edu/tm13c1hdrx400.pdf Nov 1, 2018 Version GMU NMR Center

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This is an advanced procedure for experienced users only. It requires a check off by Dr. Honeychuck.

Access to the DRX 400 is strictly prohibited to all persons who are not checked off. The possession of this document alone does not constitute being checked off. Read NMR MAGNET SAFETY (http://nmr.gmu.edu/nmrsafety.pdf). Read PREPARATION OF AN NMR SAMPLE (http://nmr.gmu.edu/nmrsampprep.pdf).

Use this document to tune and match ¹³C and ¹H after you have finished running another nucleus such as ²⁹Si or ³¹P on the 5 mm direct broadband probe on the Bruker DRX 400. Always leave the X nucleus tuned and matched to ¹³C when you are finished, since routine users do not know how.

Summary (Notes after Summary)

1. Sign the paper NMR log. Turn the nitrogen on if it is off. Insert a sample with CDCl₃ as solvent. Do not lean on the magnet. Press SPIN ON/OFF if your sample isn't spinning. Log in. XwinNMR.

2. Use the silver colored tool beneath the magnet to put the ¹³C tune and match numbers written on the card hanging from the probe into the slide switches. The least significant digit of these is not critical since you will be adjusting them.

3. File, Search, User 0startup, Name 13CCDCl3, Expno 1, Procno 1, Append, Apply, Close.

4. File, New, NAME = your file name, USER = your initials. SAVE. setti. Type your title. Click Save. Click Quit.

5. rsh. Choose bbdircdcl3.

6. lock cdcl3. If it fails to lock when it finishes (LOCK ON/OFF light still blinking) then push the FIELD button and change it to -424, and then push STDBY.

7. lockdisp. Maximize Z. Maximize Z². Maximize Z. Press STDBY. Click the blank black spectrum screen.

8. ii. Click the Seen button if an error message appears.

9. Turn the spinning off by pressing SPIN ON/OFF. If you don't do this the signal will be jumpy.

10. Type wbsw. It should be 4 MHz. Type wbst. It should be 1 k = 1024.

11. Type wobb. Click the Seen button. Type acqu.

12. Turn the monitor around so you can see it from the magnet. It should have a dip (a negative peak). Center the peak horizontally on the blue vertical line by adjusting the least significant **tuning** slide switch. Make the negative peak go as low as possible on the screen by adjusting the least significant **matching** slide switch. Tuning and matching are interdependent so go back and readjust the tuning and then matching as much as possible.

Now look at the HPPR on the floor next to the magnet. Final adjustments will be made using the display on top. Try to achieve as few LEDs as possible going back and forth between tuning and matching. As with the monitor, horizontal changes on the HPPR display represent tuning and vertical changes represent matching. The ¹³C channel is difficult (small changes with your fingers produce large changes on the HPPR). With practice you may be able to achieve 3 green matching LEDs and 1 green + 1 orange tuning LEDs.

13. Click the wobb-SW button on the left side of the monitor. Change nucleus? yes

14. Now you will tune and match ¹H. The probe is color coded. The ¹H BNC is yellow and so are the ¹H tuning and matching rods. Look at the monitor and HPPR. The ¹H channel may not need to be adjusted since the X channel was changed by the user (you) before observing the non-¹³C X nucleus, and you may not have changed the ¹H tuning and matching. If you need to adjust the ¹H tuning and matching (T and M) rods, turn them with the red Al tool underneath the magnet. The same monitor first and HPPR second procedure you used with ¹³C applies here. Tuning will be sensitive to tiny finger movements, but matching will be easier. You should be able to obtain 3 green matching LEDs and 1 green tuning LED.

15. Type stop. Click the return button at lower left to exit acqu.

16. Spin the sample and check your work by doing a ¹³C CPD spectrum, which is what you read in with

File, Search

above. Do a ¹H. Remember you are leaving the next user, who may be inexperienced, high and dry if these 2 routine nuclei don't work.

17. Remove sample. Unspin. Turn off the main valve of the 230 psi liquid N_2 dewar. Exit. Logout. All users must logout from Linux. Don't turn off tower or screen.

1. Sign the NMR log on the clipboard. Wallet, keys, pens, watch, cell phone. Are they still on you? If the N₂ is off turn on the main valve of the 230 psi liquid N₂ dewar. Press LIFT on the keypad. Do not lean on the magnet as you insert and remove samples. Put your sample into the blue spinner and gauge it. Make sure you can hear the rushing N₂, make sure the N₂ will support your sample, and let it go at the top of the magnet. Press LIFT on the keypad. Your sample may not go all the way down (listen for the click) unless the spinning is off. After your sample is down (look for the green "down" LED on the BSMS (the keypad)), press SPIN ON/OFF to spin your sample. Log in. Left single click the XwinNMR icon.

2. "Tuning and Matching" an NMR probe is an important skill. Recent probes do this automatically, but many probes must be tuned and matched manually. Put this skill on your resume.

Tuning is not the same as shimming. When you shim (Z and Z^2 , for example), you are changing the shape and intensity of the applied magnetic field so it is uniform in the region of the sample irradiated by the transmitter with RF radiation. When you tune, you are tuning the frequency of the probe to match the frequency (radio frequency) of that transmitter.

When you match, you are matching the impedance of the probe to that of the transmitter.

3. The 0startup files are write protected.

4. Many commands typed in the white field at the bottom, like setti, require Enter on the keyboard.

5. Type rsh at bottom to read in a shim file. bbdircdcl3 = broadband direct probe CDCl₃.

6. Type lock cdcl3, even if it has already locked itself. Wait for it to finish locking to go on to step 7. If it fails to lock (LOCK ON/OFF light still blinking) then push the FIELD button and change it to -424, and then push STDBY. If it still is not locked, push the LOCK ON/OFF button to stop the locking attempt, wait a few seconds, and push it again to lock it.

7. Type lockdisp to turn on the lock display. The distance from the bottom of the screen to the trace represents the deuterium lock amplitude. Press Z on the keypad. Maximize the deuterium lock amplitude by turning the wheel counterclockwise or clockwise. If it goes off the top, press LOCK GAIN on the keypad, turn the wheel counterclockwise, press Z again, and continue maximizing. When Z is maximized, maximize Z^2 and then Z again. Press STDBY after maximizing to prevent accidental changing of Z or Z^2 later if you bump the wheel.

8. Type ii to initialize the interface between the Linux tower and the console computer that controls the magnet. Usually a DQD error message related to file size appears at this point. Click the Seen button.

9. No notes.

10. wbsw = Wobble Sweep Width. The wobble curve is the signal you will observe on the monitor. wbst = Number of Wobble Steps. This is the number of individual radio frequencies measured by wobb.

11. wobb starts the process and automatically refreshes.

12. The HPPR colors are stop light colors: green (good), yellow (bad), and red (terrible). The yellow looks more orange than yellow. Horizontal = Tuning. Vertical = Matching. HPPR = High Performance Preamp, the box on the floor.

13. wobb goes through the nuclei from low frequency to high. It knows to do ¹³C and ¹H because these are the nuclei in the CPD ¹³C you read in with File, Search above. Here it switches from ¹³C to ¹H.

14. No notes.

15. Stop stops the wobb process.

16. The standard configuration of the DRX 400 is with this probe inserted, and ready for 1 H and 13 C.

17. Remove sample without leaning on magnet. Leave the blue spinner in the gauge with no nmr tube underneath the screen so the next user can find it in this standard place. Unspin. Turn off the main valve of the 230 psi liquid N₂ dewar. Type exit to exit XwinNMR. To exit Linux (you must do this; never leave the instrument logged in), right click a blank part of the screen, Logout, Logout. Do not turn off tower or screen. Do not restart tower. Do not Shutdown tower. This is a Linux computer which is never turned off except during planned power outages.