

OBTAINING A ^{13}C NMR SPECTRUM ON THE BRUKER DRX 400

<http://nmr.gmu.edu/13cdrx400.pdf>

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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 generate a ^{13}C NMR spectrum decoupled from protons using composite pulse decoupling (CPD).

Summary (Notes after Summary)

1. Sign the paper NMR log. Turn the nitrogen on if it is off. Insert sample. Do not lean on the magnet. Press SPIN ON/OFF if your sample isn't spinning. Log in. XwinNMR.
2. File, Search, User 0startup, Name 13CCDCI3, Expno 1, Procno 1, Append, Apply, Close.
3. File, New, NAME = your file name, USER = your initials. SAVE. setti. Type your title. Click Save. Click Quit.
4. rsh. Choose bbdircdcl3.
5. 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 -574, and then push STDBY.
6. lockdisp. Maximize Z. Maximize Z^2 . Maximize Z. Press STDBY. Click the blank black spectrum screen.
7. ii. rga. ii. zg.
8. efp.
9. To phase (not always needed), type apk.
10. To calibrate (not always needed), expand TMS a few times. Click the calibrate button. Put cursor on top of peak, center click, type 0 Enter. Click exp, all.
11. To plot, type xwinplot. Usually your peaks are the wrong size at this point. To turn them up or down, click button upper left with 8 green squares. Click your spectrum. 1D/2D-Edit upper

right. Turn your peaks up or down with *2 or /2. Close. Click edge of paper to cancel green squares. Ctrl P, Print. Click X upper right to exit xwinplot.

12. Remove sample. Unspin. Turn off the main valve of the 230 psi liquid N₂ dewar. Exit. Logout. All users must logout from Linux. Don't turn off tower or screen.

Notes

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 press SPIN ON/OFF to spin your sample. Log in. Left single click the XwinNMR icon.

2. The 0startup files are write protected.

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

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

5. Type lock cdcl3, even if it has already locked itself. Wait for it to finish locking to go on to step 6. If it fails to lock (LOCK ON/OFF light still blinking) then push the FIELD button and change it to -574, 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.

6. 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 Z² and then Z again. Press STDBY after maximizing to prevent accidental changing of Z or Z² later if you bump the wheel.

7. Type ii to initialize the interface between the Linux tower and the console computer that controls the magnet. Usually a DQD error message appears at this point. Click the Seen button. Type rga. This stands for receiver gain automatic, and an iterative process starts which chooses a value for rg (the receiver gain) matched to your concentration and observe nucleus (¹³C). This takes 15 seconds. Wait for it to finish. Type ii again. Type zg to zero the data file and go. A ¹³C spectrum takes 2 minutes.

8. Type efp. This stands for em, ft, and pk, which in turn stand for exponential multiplication, Fourier transform, and phase correct. Exponential multiplication increases the signal to noise ratio in the frequency domain spectrum by non-constant multiplication of the time domain spectrum. Then the time domain data is Fourier transformed to the frequency domain. After that the spectrum is automatically phase corrected so all peaks are positive.

9. If apk does not give all positive peaks, phase manually by clicking the phase button on the left. Click biggest. Hold down the left mouse button on PH0 while moving up or down. You may also have to do this with PH1. Click return. Click Save & return.

10. To expand TMS, box it in a few times with the box at upper left.

11. For expansions, click the EXPAND button and box in the peaks you want. Ctrl P, Print.

12. 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 power failures.