OBTAINING A ^{13}C DEPT 135 NMR SPECTRUM ON THE BRUKER DRX 400

http://nmr.gmu.edu/13cdept135drx400.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 DEPT 135 NMR spectrum (CH and CH_3 up; CH_2 down, 4^o C nulled) on the broadband direct probe.

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. Do a ^1H spectrum and a CPD ^{13}C spectrum.

2. File, Search, User 0startup, Name 13CDEPT135CDCl3, 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. Make sure the tuning and matching settings at the bottom of the probe are on the ^{13}C values.

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 -558, 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. If you have methylenes in your solute, you will need the baseline in the middle instead of the bottom. To move the baseline to the middle, click

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 N_2 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 N_2 is off turn on the main valve of the 230 psi liquid N_2 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 N_2, make sure the N_2 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. The purpose of the ^{1}H spectrum is to check the shimming, and the CPD ^{13}C spectrum tells you where all of your peaks are. Quaternary carbons disappear in a DEPT 135.

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 $CDCl_3$. Check the tuning and matching settings on the card that hangs below the probe, and make sure the probe is on those ^{13}C settings. Don't change the last digit on the right if it is already on ^{13}C, since a previous user has tuned and matched using a sensitive procedure. If you just did a ^{1}H or CPD ^{13}C you don't have to do steps 4, 5, and 6.

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 -558, 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. If you just did a ^{1}H or CPD ^{13}C you don't have to do steps 4, 5, and 6.

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 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. If you just did a ^{1}H or CPD ^{13}C you don't have to do steps 4, 5, and 6.

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 (^{13}C). This takes 15 seconds. Wait for it to finish. Type ii again. Type zg to zero the data file and go. A ^{13}C DEPT 135 spectrum on a concentrated sample (10% solute, 90% solvent) takes 4 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 except CH_2s.

9. If apk does not give all positive peaks for non methylenes, 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 N_2 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.