

OBTAINING A ^{13}C NMR SPECTRUM ON THE BRUKER DPX 300

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

<http://nmr.gmu.edu/13cdpx300.pdf>

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Do not photocopy this document. Instead, print out the current version at the above address.

Access to the DPX 300 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>).

Summary (Notes after Summary)

1. Sign the paper NMR log. Insert sample. Do not lean on the magnet. It will not support your weight. If you lean on the magnet, you may cause a quench. Press SPIN ON/OFF if your sample isn't spinning. Log in. TopSpin.
2. Left click /opt/topspin1.3, 0startup, 13CCDC13, 1, 1. Right click the last 1. Display.
3. Left click the blank page upper left. NAME = your file name, USER = your initials, TITLE = your title. OK.
4. rsh. Choose qnpcdcl3. Read.
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 9602, and then push STDBY.
6. Right click small lock display screen. Locksignal display. Red 2 upper right. Maximize Z, then Z^2 , then Z. Press STDBY. Green 1 upper right.
7. ii. rga. ii. zg.
8. efp.
9. To phase (not always needed), click Processing, Phase Correction, Automatic phasing, OK.
10. To calibrate (not always needed), expand TMS a few times. Click

Put cursor on top of peak, left click, type 0. OK. Click

11. layout. Choose +/0startup13C.xwp, OK.

12. To plot, type plot. 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. Move your spectrum to the bottom with



Close. Click edge of paper to cancel green squares. Ctrl P, Print. Click X upper right to exit plot.

13. Remove sample. Insert standard. Exit. Logout. All users must logout from Linux.

Notes

1. Sign the NMR log on the clipboard. Wallet, keys, pens, watch. Are they still on you? Press LIFT on the keypad. The magnet is on donuts. Do not lean on it as you insert and remove samples. It will not support your weight. If you lean on the magnet, you may cause a quench. Remove the previous sample from the blue spinner, put yours in, and adjust it in the gauge. Making sure the air will support your sample, let it go at the top of the magnet. Press LIFT on the keypad. Your sample won't go all the way down (listen for the click) unless the spinning air is off. After your sample is down press SPIN ON/OFF to spin your sample. Log in. Left double click the TopSpin icon.

2. The 0startup files are write protected.

4. Type rsh at bottom left to read in a shim file. Choose the qnpdc13 (Quad Nucleus Probe CDCl₃) shim file.

If you just did rsh for a ¹H spectrum, you don't have to do it again.

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 9602, 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 lock cdcl3 for a ¹H spectrum, you don't have to do it again.

6. Right click the small black lock display screen. Choose Locksignal display. Bring the lock display screen to the front by clicking the red 2 upper right. 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 moving the wheel counterclockwise or clockwise. If it goes off the top, press LOCK GAIN on the keypad, move the wheel counterclockwise, press Z again, and continue maximizing. Then maximize Z², and then Z again. Always press STDBY after maximizing with Z the last time to prevent accidental changing of it later if you bump the wheel. Click the green 1 upper right.

If you just maximized Z, Z², and Z for a ¹H spectrum, you don't have to do it again.

7. Type ii to initialize the interface between the Linux tower and the console computer that controls the magnet. 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 spectrum takes 12 minutes.

You do have to ii, rga, ii, and zg even if you just did a ^1H spectrum.

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 (Processing, Phase Correction, Automatic phasing, OK) does not give all positive peaks, phase manually by clicking



Hold down the left mouse button on 0 while moving mouse up or down until the red line peak is phased. If peaks far away from the red line peak are out of phase, adjust 1 until they are in phase. Click



to save the phasing information and return to the spectrum.

10. To expand TMS, drag across it a few times.

11. This layout has the spectrum, title, parameters, etc. positioned so they don't overlap.

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

13. Remove sample without leaning on magnet. Insert and lock a standard sample. You can help to extend the compressor life by not spinning the sample. Type exit to exit TopSpin. To exit Linux (you must do this; never leave the instrument logged in), click the Red Hat lower left, Logout, Logout.