OBTAINING A ^{19}F NMR SPECTRUM DECOUPLED FROM ^{1}H ON THE BRUKER DPX 300

http://nmr.gmu.edu/19fdecoupdpx300.pdf

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GMU NMR Center

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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. Turn the nitrogen on if it is off. 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. Pull down on the slide control at the bottom of the probe to put it on 3.

3. Left click /opt/topspin1.3, 0startup, 19FDECOUPCDCl3, 1, 1. Right click the last 1. Display.

4. Left click the blank page upper left. NAME = your file name, USER = your initials, TITLE = your title. OK.

5. rsh. Choose qnpcdcl3. Read.

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 -802, and then push STDBY.

7. Right click small lock display screen. Locksignal display. Red 2 upper right. Maximize Z. Maximize Z^2. Maximize Z again. Press STDBY. Green 1 upper right.

8. ii. rga. ii. zg.

9. efp.

10. To phase (not always needed), click Processing, Phase Correction, Automatic phasing, OK.

11. To calibrate (not always needed), expand $CFCl_3$ a few times. Click

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

12. To integrate, click Analysis, Integration, Auto-find, OK.

13. layout. Choose +/0startup19F.xwp, OK. 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. Close. Click edge of paper to cancel green squares. Ctrl P, Print. Click X upper right to exit plot.

14. Push up on the slide control at the bottom of the probe to put it on 1.

15. Remove sample. Insert standard. Unspin. Check the DRX 400 by bumping the mouse. If the login screen is there, turn off the main valve of the 230 psi liquid N_2 dewar (the one with the tag). If the login screen is not there, it means someone is doing a long accumulation on the DRX 400 and you should leave the N_2 on. 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 (the one with the tag). Press LIFT on the keypad. Do not lean on the magnet as you insert and remove samples. Remove the previous sample from the blue spinner, put yours in, and adjust it in the gauge. 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 double click the TopSpin icon.

2. Position 3 is ^{19}F. The positions are on the unit near the bottom of the probe.

3. The 0startup files are write protected.

4. No notes.

5. Type rsh at bottom left to read in a shim file. Choose the qnpcdcl3 (Quad Nucleus Probe $CDCl_3$) shim file.

6. 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 -802, 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. 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. 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. Click the green 1 upper right.

8. Type ii to initialize the interface between the Linux tower and the console computer that controls the magnet. If the DQD, SW, and qsim warning appears, click Close. 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 (^{19}F). This takes 15 seconds. Wait for it to finish. Type ii again. Type zg to zero the data file and go. A ^{19}F spectrum takes 1 minute.

9. 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.

10. 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. If you cannot see the

icon, pull the right side of the window to the right.

11. To expand $CFCl_3$ (the ^{19}F 0 ppm standard), drag across it a few times. If you don't know which signal is $CFCl_3$, you should run it by itself and then add your compound of interest. The ^{19}F chemical shift range is very large. Most fluoroorganics are in the -250 to +50 ppm region. Inorganics have a much larger range. The maximum ^{19}F SW on the DPX 300 is 266 ppm. You may have to look for your peaks by changing o1p (offset channel 1 ppm). Increasing o1p by 10 ppm moves your window to the left 10 ppm.

12. If you don't like the Auto integrals, you can integrate manually with the following procedure. Click

Click the green

to select all. Click

to delete them. Click

Drag across the peaks you want. To change areas, right click any integral, Calibrate, put in New value, OK. Click

to save and return.
You can delete one integral alone by right clicking it.

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

14. The standard position is 1 (^{13}C). Please put it back on ^{13}C because most users don't know how to change the slide control.

15. Remove sample without leaning on magnet. Insert and lock a standard sample. Unspin. Check the DRX 400 by bumping the mouse. If the login screen is there, turn off the main valve of the 230 psi liquid N_2 dewar (the one with the tag). If the login screen is not there, it means someone is doing a long accumulation on the DRX 400 and you should leave the N_2 on. Type exit to exit TopSpin. 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.