NMR OPERATOR CHECK-OUT

Avance-DRX 500 at MedSci 1D

Review safety rules.

1. Log in. Use your chosen ID and password to access your CentOS account

2. Start-up. double click on Topspin 1.3 Icon on the desktop

3. Set initial parameters. Open up an old experiment, ie an old Proton experiment. From this experiment, type **edc** or **new** to create a new experiment. Set new experiment name, start with experiment number 1, set your solvent, execute **getprosol**, click ok.

> Note, after you have created a new dataset, if you wish you can read in a standard parameter set. For a routine proton experiment, type **rpar PROTON.500 all** on the command line. This will load routine Proton acquisition and processing parameters into your workspace.

4. Sample insertion. Place your sample in blue spinner at correct sample height using the depth gauge. Remove black cap on the top of the magnet. Type **ej** to eject / start air flow, place your sample in the magnet, type **ij** to insert your sample.

5. Lock. Type **lock** to open up the lock page. Select your solvent. Wait until the lock step has completed (usually takes 10-15 seconds).

6. Tune probe. Type **atma** to automatically tune the nucleus or nuclei that are open in your experiment. Example, if you have opened up a Proton experiment in Step 4, **atma** will tune and match the Proton channel. A 1H / 13C HSQC experiment would tune and match both the 1H and 13C channels during **atma**. You can also use **atmm** to control the tuning and matching stepping motors manually.

7. Shim. First, it will be helpful to manually shim on Z and Z2 using the BSMS Panel. You will need to drop the lock gain (also using the BSMS panel) if the lock level rises about 100%. After shimming Z1 and Z2 manually, save your shims as GSHIM by typing **wsh GSHIM**. Then you can do Gradient shimming: open up the gradient shimming gui by typing **gradshim**, and select Start Gradient Shimming. When completed (usually takes about 1 minute), close out of gradshim and return to your experimental parameters page. If the lock display has again reached above 100%, lower the lock gain using the BSMS panel.

8. OPTIONAL: Trial spectrum (usually one scan). Check linewidth and FID. This step is optional but useful. Double check your initial parameters in your acquisition window. To look at your important acquisition parameters, type **ased** on the command line. Use 0 dummy scans and 1 scan (DS = 0, NS = 1). Check and set your spectral width **sw** and your carrier frequency **o1p** (center of your spectrum). Type **zg** to collect your trial spectrum. Take a look at your raw FID – is your acquisition time set appropriately? Should you collect data for longer time? Shorter time? To view your trial spectrum, process your raw FID by performing a Fourier transform using the **fp** or **efp** command. Make a note of your signal intensity, spectral width, center of your spectrum, etc.

9. Select proper carrier position and sweepwidth. Based on the results of your trial spectrum or prior knowledge of your system, you should move the carrier frequency **o1p** to the center of your spectrum, and set the sweep width **sw** so that you see all resonances. You can change your sweep width if needed by typing **sw** on the command line, and setting chosen sweep width when prompted.

10. Select all proper parameters for run (rep rate, etc.). To access the condensed acquisition parameters page, type **ased** on the command line. Based on your sample concentration, you may need to change the number of scans **ns** requested. Also, set your relaxation delay **d1**. If you want to be quantitative, be sure that the total rep rate (**d1** plus acquisition time **aq**) equal at least 5 times T1. If you use the routine **zg30** pulse program, a **d1** of 2 seconds is usually adequate.

11. Check gain. Type **rga** to set the receiver gain automatically.

12. Collect data. Double check your parameters. Go through your mental checklist, ie "sample is in, locked to solvent, probe is tuned and matched, I have shimmed, I have set appropriate acquisition parameters). When you are ready to collect your data, type **zg** on the command line

13. Data process, including apodization, phasing, window function, peak shift reference, etc. Type **efp** to perform Fourier transform. Change apodization (line broadening) by typing **lb** and changing the value. Typically, one uses 0.1 to 0.3 Hz line broadening for a Proton spectrum. Alternatively, you my elect not to use any apodization by typing **fp** instead of **efp.** Type **apk** to perform automatic phasing of your spectrum. Often you will have to manually phase your data by selecting the Adjust Phase icon under processing.

To reference your data, type **sref** on the command line. This will attempt to reference all chemical shifts to TMS, if present. If TMS is not present, **sref** will still get you close. If you want to reference your chemical shifts to a specific peak, first zoom into that peak, and then type **.cal** on the command line. Select the peak of interest, and type in the desired chemical shift when prompted.

14. Leaving procedure. Remove your sample from the spectrometer by typing **ej**, and replace the black protective cap. For the 800, 600 and 500 instruments we do not use a "blank" sample, so simply type **ij** to insert no sample. Close out of topspin when you are done.

15. Log out, user bill. Be sure to sign the log book and billing slip, and to log out of your account.