

## Routine 1D Acquisition on Bruker AV-500 & AV-600

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### GENERAL PROCEDURE FOR DATA ACQUISITION

The general procedure for acquiring NMR data on all spectrometers is similar, but commands differ depending on the software. For every sample, you must perform all of these steps. However, if you are running multiple experiments on the same sample (e.g., first  $^1\text{H}$ , then  $^{13}\text{C}$ , then HSQC), you do not need to re-lock or re-shim unless you change the probe temperature or remove the sample from the instrument.

- 1) Log into computer and open Topspin
- 2) Insert sample: remove dummy sample, set depth of your sample
- 3) Set-up initial acquisition parameters by typing **new**
- 4) **Lock** on to deuterated solvent
- 5) Tune the probe: **atma**
- 6) Shimming: **rsh LAST; topshim**
- 7) Check acquisition parameters: **ased**
- 8) Set the receiver gain: **rga**
- 9) Acquire data: **zg**
- 10) Process data: **ef;apk;abs n**
- 11) Remove sample and replace with dummy sample
- 12) Log off spectrometer: **CTRL + ALT + BACKSPACE**

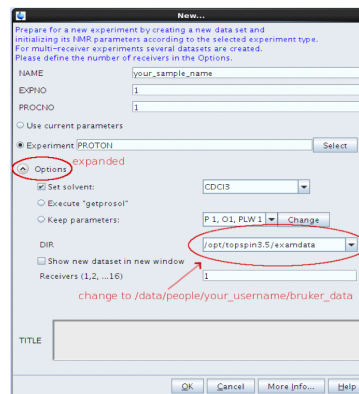
### LOGIN AND INSERT YOUR SAMPLE

If no one was logged into the spectrometer computer, there will be no data acquisition active. Login with your username and password. Double-click on the TopSpin icon to launch TopSpin.

- 1) Check to make sure that the spinner is clean. Avoid touching below the black and white tape with your bare hands.
- 2) Wipe the outside of the sample tube with a clean KimWipe and insert it into the spinner. Make sure the tube fits snugly in the spinner. If a tube is loose in the spinner, do NOT use it. Please use the other spinner. Loose tube/spinner combinations can get stuck in the instrument or easily break in the probe, which will cause extended downtime and require expensive repairs.
- 3) Set the depth using the depth gauge. After you set the depth, remove the sample and spinner from the depth gauge, and wipe the outside of the sample tube again.
  - a. If your sample height is not  $\geq 40$  mm, make sure to center the sample around the line marking the center of the *rf*-coils when using the depth gauge.
  - b. Improperly-positioned samples can get stuck in the instrument or easily break in the probe, which will cause extended downtime and require expensive repairs.
- 4) Place the sample in the SampleCase injection system. Eject the dummy sample by hitting the inject button.
- 5) Rotate the Sample Case so that the sample is in the appropriate position and hit the inject button. The sample will auto-inject into the instrument. You may hear a clicking sound once the spinner properly settles into the instrument.

## SET UP INITIAL ACQUISITION PARAMETERS

- 1) Type **new** to create a new data set. This will open the file creation dialogue. There are three fields available for you to describe your experiment:
  - a. **NAME**: experiment name, usually something descriptive, for example a lab notebook number. This field permits letters, numbers, dashes, and underscores. Do not use special characters (spaces, #, ?, !, \*, etc)
  - b. **EXPNO**: experiment number. Can be any number 1-999. Many people use 1 for  $^1\text{H}$ , 13 for  $^{13}\text{C}$ , 31 for  $^{31}\text{P}$ , etc.
  - c. **DIR**: make sure this says  
`/data/people/your_username/bruker_data`
- 2) Next to **Experiment**, hit the Select Button. A list of experiments will appear. Click on the experiment you'd like to run and then hit the "Set selected item in editor" button.
  - a. There is a shorter list in `Source = /opt/topspin/exp/stan/nmr/par/user`. We have a UO default proton, carbon, fluorine, and phosphorus parameter set. The naming scheme is nucleus.instrument, e.g., `1h.av500` for a  $^1\text{H}$  experiment on the 500, `13c.av600` for a  $^{13}\text{C}$  data set on the 600.
  - b. If you know the name of the parameter set you want, you can also type the name into the empty box.
- 3) **Select your solvent** and **Execute 'getprosol'**. The remaining fields can be left as is. You can always overwrite these parameters later if needed.



## LOCK ONTO DEUTERATED SOLVENT

The NMR lock is a  $^2\text{H}$  (deuterium) lock, which compensates for small changes in the magnetic field that might occur during data acquisition. The NMR lock will only work if you use a deuterated solvent. Automatic locking is available on both the AV-500 and AV-600.

- 1) Lock on to your deuterated solvent with the command **lock**. Select your solvent from the menu that pops up, then select "OK".
  - a. You can skip the solvent selection menu and call the solvent directly by invoking its "short" name, which appears in the first column. For example, **lock cdcl3**.
- 2) When this finishes, the message "lockn:done" is output above the command line.
  - a. If you have difficulty locking, please make sure you read in the most recent shim file. Type **rsh LAST** to read in the most recent shim file. Then try **lock** again.

## SHIMMING

To produce an NMR spectrum with good resolution, the magnetic field must be homogeneous throughout the volume of sample. Shimming is a way of minimizing the magnetic field inhomogeneity by using magnetic field gradients.

- 1) Read in the most recent shim file by typing **rsh LAST**.
- 2) Type **topshim** to utilize Bruker's gradient shimming algorithm.
- 3) If you have problems with topshim, you can manually adjust the field gradients to maximize the lock level.

## TUNE THE PROBE

Tuning the probe is important for maximum sensitivity. For example, with selective 1D, 2D, or DOSY experiments, you also want to be sure that the pulse times actually correspond to the expected pulse angles! All nuclei that will be observed or pulsed must be reasonably well-tuned. Both Bruker

instruments have automatic tuning available. The probes have stepping motors that automatically adjust the tuning rods.

Type **atma** to activate automatic tuning of the probe.

### CHECK THE ACQUISITION STATUS PARAMETERS

For routine data acquisition, you can use the default parameters. Do not change any acquisition parameters that you do not understand. Type **eda** or **ased** to see the full or short list of acquisition parameters. In general, we suggest using **ns** as a multiple of 8 (the default value). For additional signal averaging, please change **td0** to whatever value you need. The total number of scans acquired will be  $ns \times td0$ , e.g., if you require 32 scans, leave  $ns = 8$ , and set  $td0 = 4$ . If you change the number of scans, you can type **expt** to check how long the experiment will take.

### SET THE RECEIVER GAIN

For all nuclei except natural abundance  $^{13}\text{C}$ , you will need to set the receiver gain (rg). (The receiver gain is already set to the maximum for  $^{13}\text{C}$ ). If the receiver gain is too low, you will miss small signals. If it is set too high, you will get artifacts in your spectrum.

Type **rga** to start the automatic receiver gain routine. When this is finished, the message "rga:finished" is output above the command line.

### ACQUIRE DATA

- 1) Type **zg** to start data acquisition. The **zg** command always zeroes the data before starting, then executes the current pulse program. The sequence is repeated  $ns \times td0$  times with appropriate phase cycling and the data points are added. The data are stored after the completion of  $ns$  scans.
- 2) If the signal to noise ratio for the resulting spectrum is not optimal, you can restart acquisition and add on to the existing data with the **go** command. The command **go** will not zero the existing data, but instead will add another  $ns \times td0$  scans to it, yielding higher SNR.
- 3) To check the progress of the data acquisition, type **tr**. This will transfer the data in the acquisition memory and add it to the disk file identified by your current data set name and expno. You can then process the data as usual while the acquisition continues. You can repeat this process as long as needed to obtain the desired signal to noise ratio.
- 4) To terminate data acquisition prior to completion of all the scans, type **halt** to stop at the end of the current scan and store the data. Do not type stop or the data acquired up to this point will be lost!

### PROCESS DATA

- 1) The command **ef** will perform an exponential multiplication and Fourier transform.
  - a. The **lb** (line broadening) parameter will be applied with the command **ef**.
  - b. To perform Fourier transform without apodization function, type **ft**.
- 2) **apk** will automatically adjust the phase of the spectrum. You can type **.ph** to enter the manual phasing routine if automatic phasing does not work for you.
- 3) **abs** or **abs n** will correct the baseline.
- 4) You can string together all of the commands and separate them with a semicolon, i.e., **ef;apk;abs n**
- 5) TopSpin automatically saves your data for you. You do not need to take any extra steps.

### REMOVE SAMPLE + LOG OFF SPECTROMETER

- 1) Walk over to the SampleCase injection system. Hit the inject/eject button.
- 2) Once your sample is in the SampleCase, rotate the SampleCase so that the sample is in the appropriate position to remove.
- 3) When you are done, please put the dummy sample back into the instrument.
- 4) Close TopSpin by either clicking the "X" in the upper right corner or type **exit**.
- 5) Log off the spectrometer. Be sure to "Confirm Logout" (do NOT use "switch user"). A keyboard shortcut you can use is **CTRL + Alt + Backspace**.