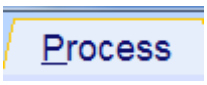
Operation procedure for Bruker 400 NMR

By Jianjun Chen

Phone: 448-1550; Office: Room 327A, Johnson Bldg

- Click on “Topspin” on desktop, this brings you to the Topspin 3.0 interface
- Click on “iconnmr”, a small window pops up
- Click on “Automation” and another small window pops up
- Select your username
- Type password, this brings you to the IconNMR interface
- Click on the 1st row in the IconNMR interface (The row numbers correspond to the position in the sample tray of autosampler. Right now we don’t have an autosampler. Once the autosampler is installed, you can put multiple samples on the sample tray and define experiments for the corresponding rows).
- Click on “add”
- Type sample name
- Select solvent
- Select experiment (select “N PROTON” for 1H experiment, “N C13CPD” for 13C experiment, etc…), you can run a set of experiments (for example 1H, 13C, COSY….) in the 1st row by clicking on “add” and specify the experiments.
- The default scan number for 1H is 16 and 1024 for 13C. But you can always change it by clicking on  which is “Here to edit specific parameters” icon and type in the desired scan numbers
- Type a title for your experiment (for example, chemical name of the compound) and select “set & copy title” so that all the information will be copied to next experiment.
- Click on “Submit”
- Click on “Start”
- A small window pops up, click on “Start” again on this window
- Acquisition starts and wait until finished

Data processing

- **Open file**. Go back Topspin 3.0 interface. Under the “Browser”, go to the submenu of “C:\Bruker\TopSpin3.0”, find your “username”, select the file and drag it to the right window
- **Go to "Process"** 

- **Adjust phase.** Click on “ 

”. Normally you can skip “adjust phase”. But if the baseline of a peak is asymmetrically disposed on either side of the peak. This peak needs to be phased. You can type “apk” in the command line (on the bottom) and hit “enter” in the keyboard. If this doesn’t work well, you have to manually adjust the phase. Go to “Process” on the top of the Topspin 3.0 interface and click on “Adjust Phase”. Select either “0”(coarse) or “1” (fine) and select the desired peak that you want to phase, hold the mouse, then drag it up and down to make the baseline of the peak symmetrical. Click on  , which is the “Return & save phased spectrum” icon when you are done
- **Calib. Axis**. Click on “ 

”. This is to reference your solvent peak to a standard chemical shift (for example DMSO peak is 2.5 ppm). Place cursor on a peak and click, type the value and hit “enter” in the keyboard
- **Pick peaks**. Click on “ 

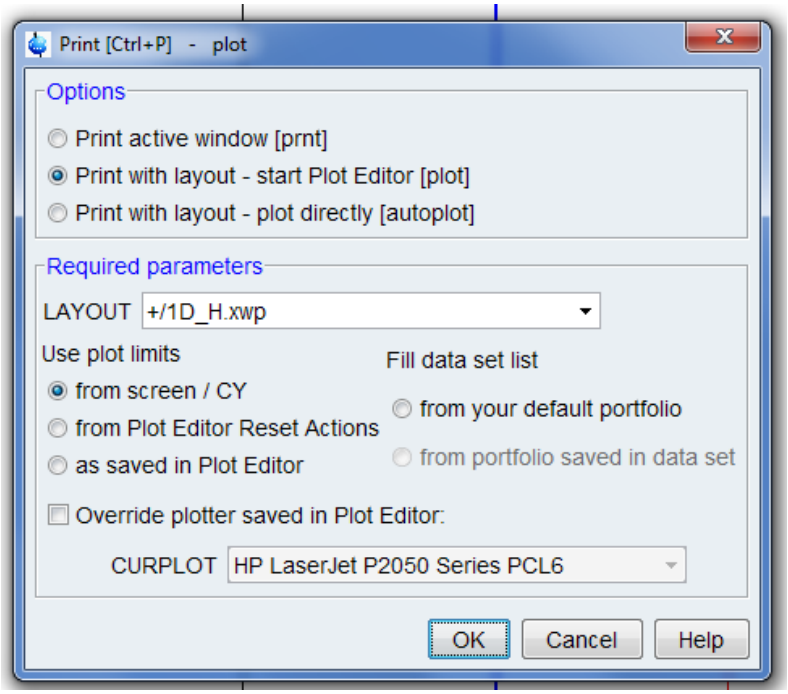
”. This is to define the peak threshold
    - Click on  which is “Delete all peak” icon to removal the previous peak labels
    - Click on the second icon:  which is “Define new pick picking range”, then drag the mouse to the peaks and the bottom line is the threshold above which all peaks will be labeled
    - Click on  to return and save your changes
- **Integration**
    - Click on “ 

”.
    - Click on  which is “Delete all integrals, no question” icon to remove all previous integration

- Click on  which is “Define new region using cursor” icon and do the integration by dragging the cursor from left to right of a peak
- To set integral values, select a peak and right click, then select “Calibrate current integral”, type in the value and hit “enter”
- Click on “Return, save changes”

- **Plot**

Select the printer icon  on the top left corner of the Topspin 3.0 interface or type “print” in the command line, a small window pops up, select “print with layout – start plot editor [plot]”, S



click “ok”, and spectrum is displayed in another window. Right click on anywhere of the spectrum, select “Edit”, then “1D Spectrum”, “peaks”, you can check or uncheck the “show peak labels”, “show integrals”, “show integral labels” according to your preference

- **Print**

Click on print icon in the spectrum window to print out your spectrum. You can also expand the peak region and print. To go back to full spectrum, click “undo”. Close this window when you are done with printing, the computer asks “Save changes to 1D_H.xwp?”, select “no”

- **Take your sample out**

  Your sample will be ejected automatically when finished. DONOT take sample out when it is right underbeneath the transfer tube. First, press on the rotation button (blue one on the leftmost side) to move your sample away from the transfer tube, then take you sample out from the holder. If your sample doesnot come out automatically, type “ej” to eject it.

- **Save Data to your flash drive**

  You can copy the data to your USB by going to “Computer\C:\Bruker\TopSpin3.0\Data\Your username\nmr”.

- **Processing data using ACD software in the offline computer.**

  The data for Bruker 400 NMR is mapped to the offline computer. You can find your data in the “z” drive which is a network drive and the name is “Data\\172.23.19.163\Z”. You can also save your data to a USB and process it in this offline computer using ACD software.