Basic NMR Operation

Guide for the Bruker AV-III 600 MHz NMR Spectrometer
TopSpin 3.X Instruction

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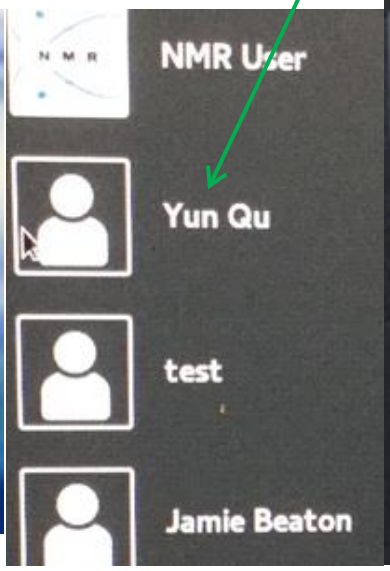
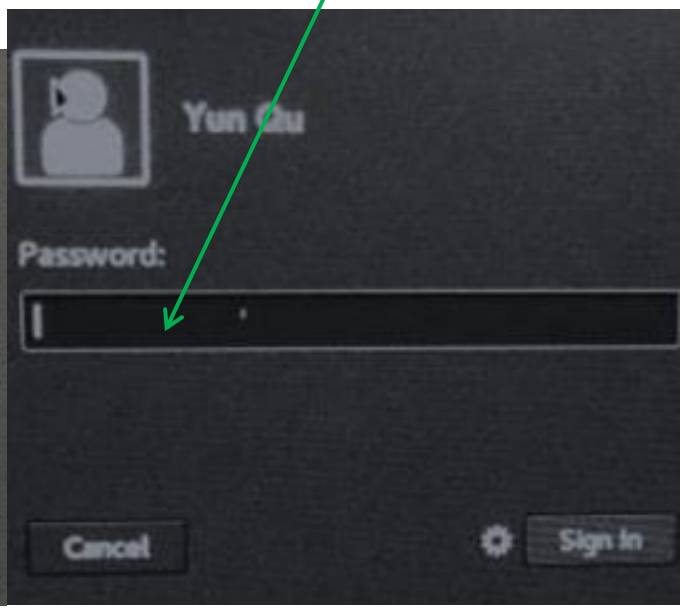
1. Start TopSpin

Press any key to turn the screen back on. Click on your account and type in **password** in pop-up form to login. Click on TopSpin 3.x icon to open TopSpin window

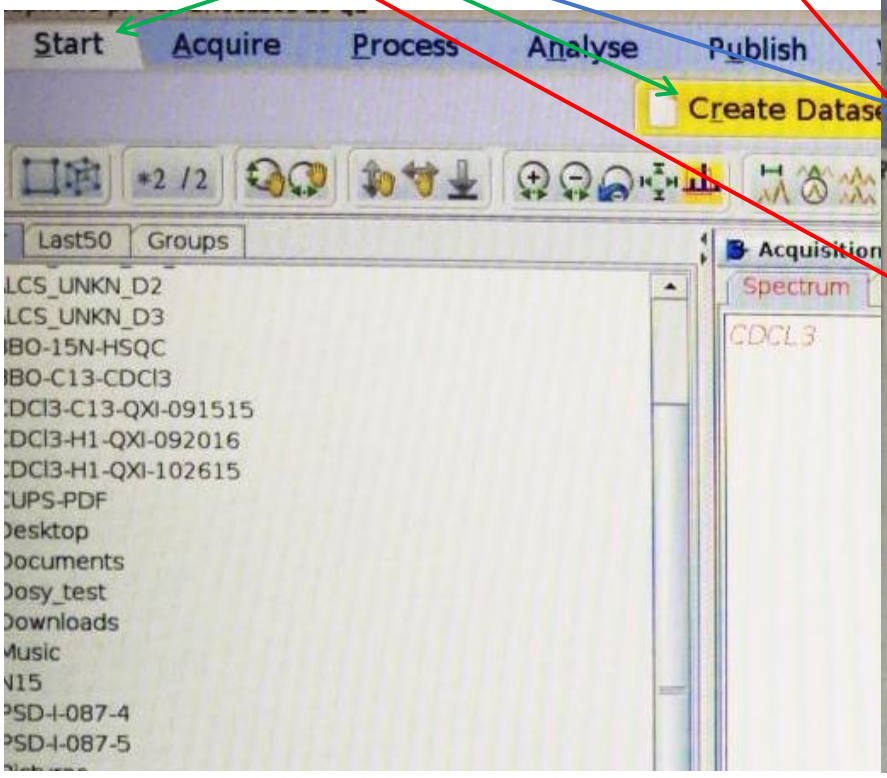


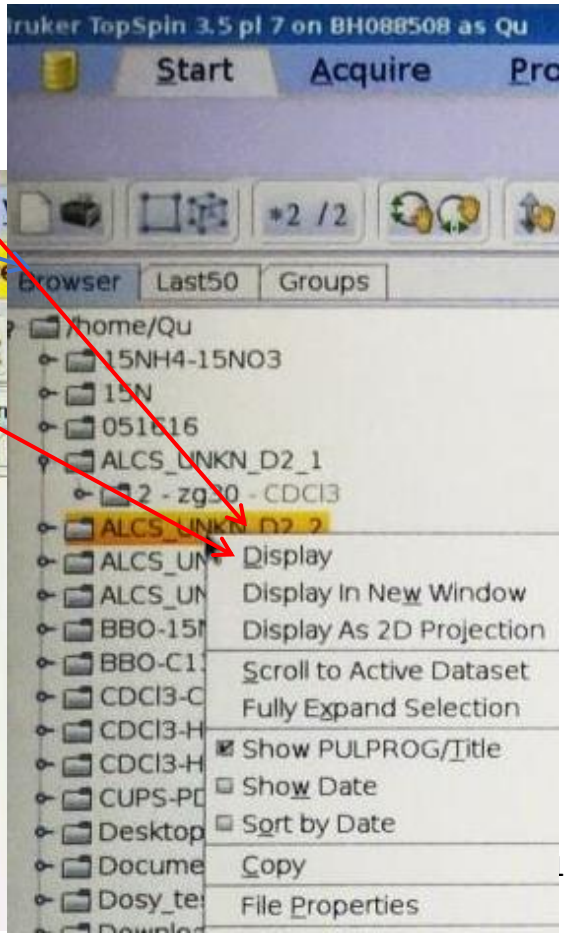


2. Setup Experiment

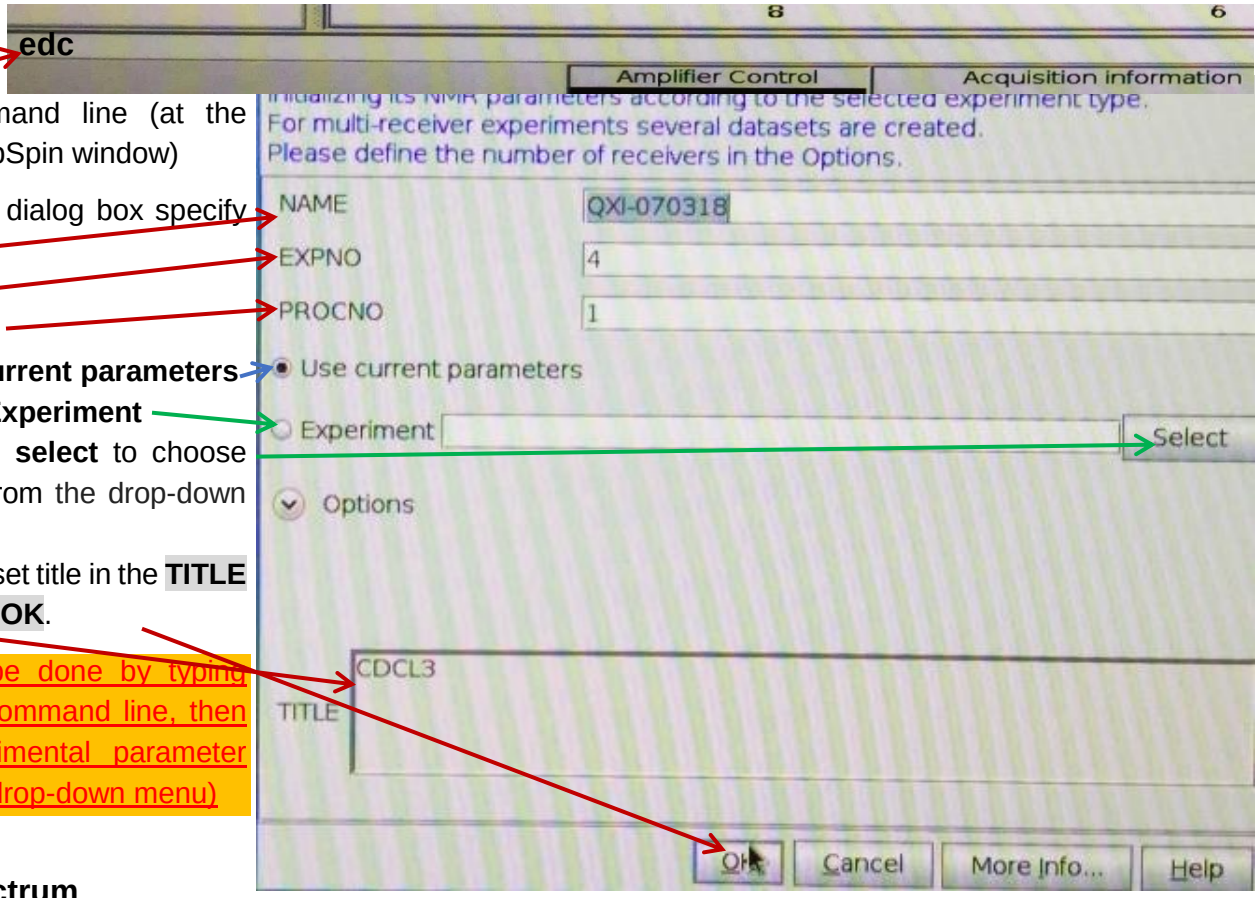
(1). Create file, It can be done by Open an old dataset or Create Dataset. To Open an old dataset, in the **Browser** window to locate your data, right-click a dataset name, then select **Display** from the drop-down menu.

To Create Dataset, clicking on Start in the TopSpin menu bar, then click on Create Dataset





(2). Edit file, type **edc** in the command line (at the bottom of TopSpin window)

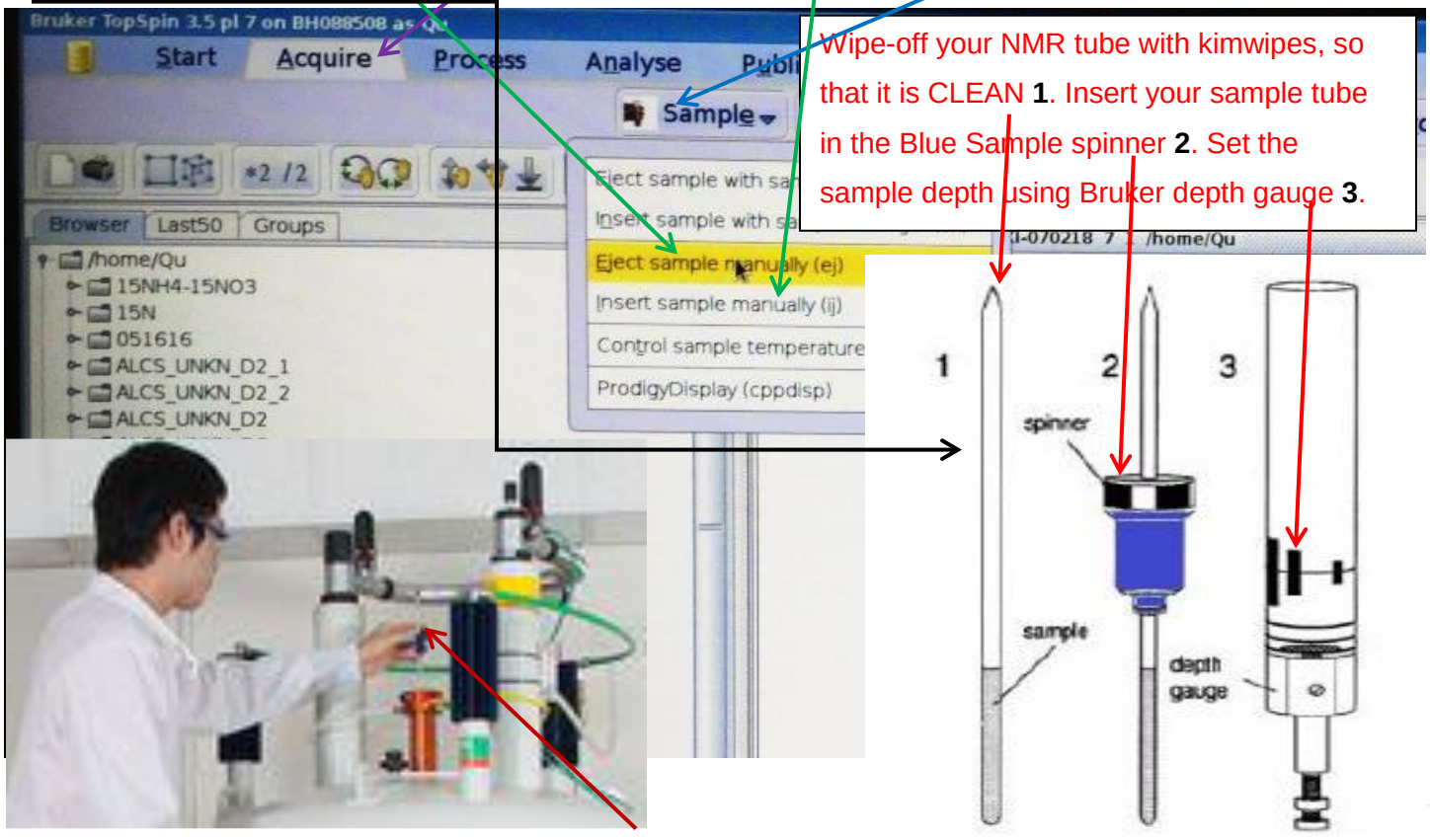


In the popup dialog box specify on **NAME**
EXPNO
PROCNO
select **Use current parameters**
(or click on **Experiment** then click on **select** to choose parameters from the drop-down menu).
type the dataset title in the **TITLE** box. Click on **OK**.

(these can be done by typing **rpar** in the command line, then select experimental parameter set from the drop-down menu)

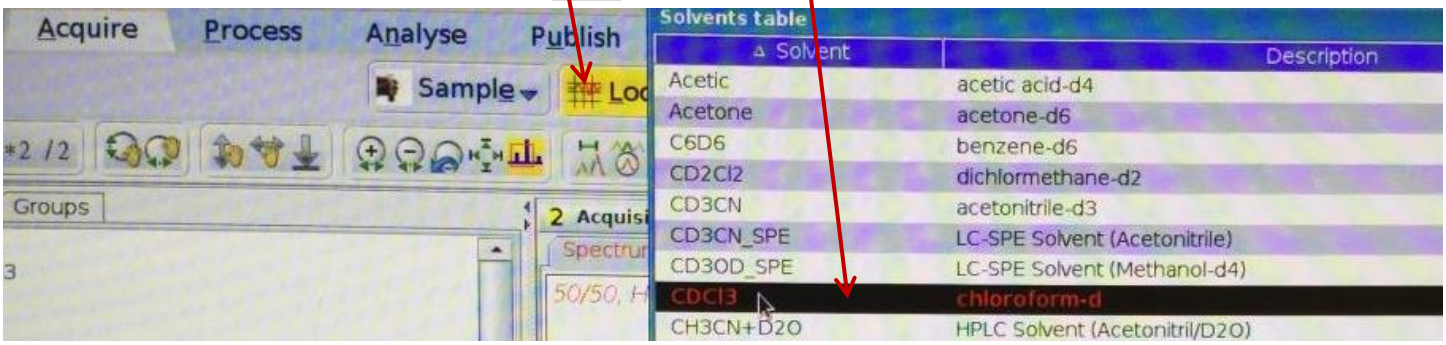
3. Run Spectrum

(1). Exchange Sample Tube, click on **Acquire** in the TopSpin Menu bar, then click on Sample , select **Eject sample manually (ej)** from the drop-down menu, then take sealed $CDCl_3$ tube sample out and place your sample by following **1**-**3**, click on **Insert sample manually (ij).**



! *Make sure your NMR tube is supprted by compressed air before releasing it.*

(2). Acquire Spectrum, a). Lock, click on **Lock**, select the solvent on pop-up table.

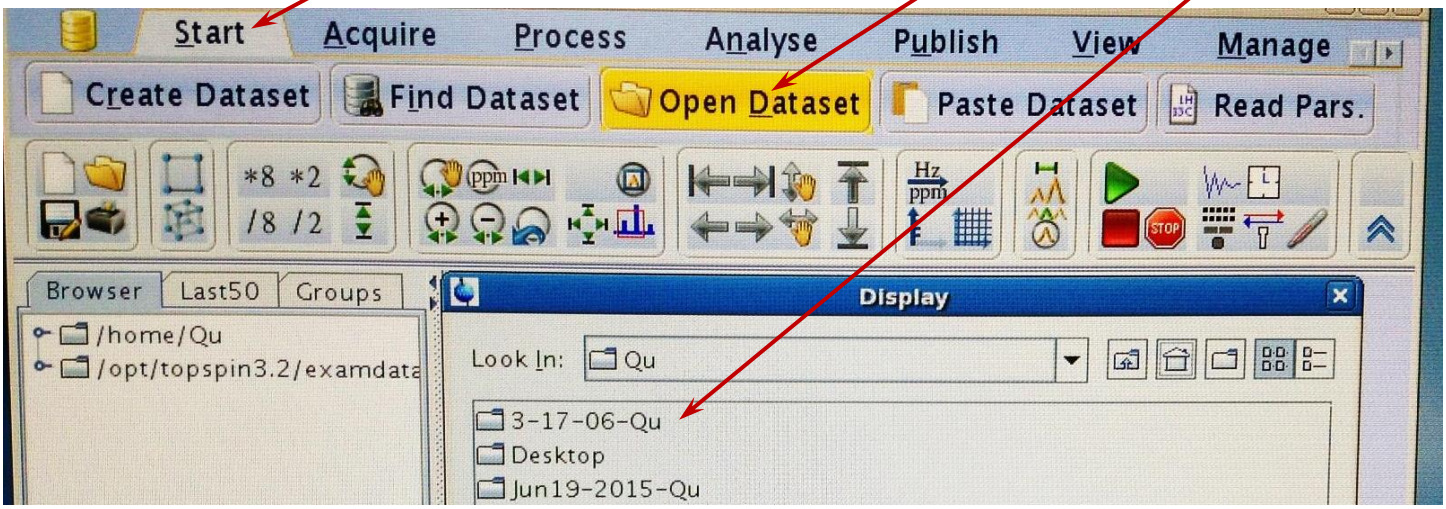


b). Tune Probe, click on Tune **c). Sample Spin,** click on Spin (only spin the sample for 1D NMR, no spin on multidimensional NMR) **d). Shim,** click on Shim to perform topshim **e).** click on Prosol then click on Gain **f). Run Spectrum,** to record spectrum, click on Go

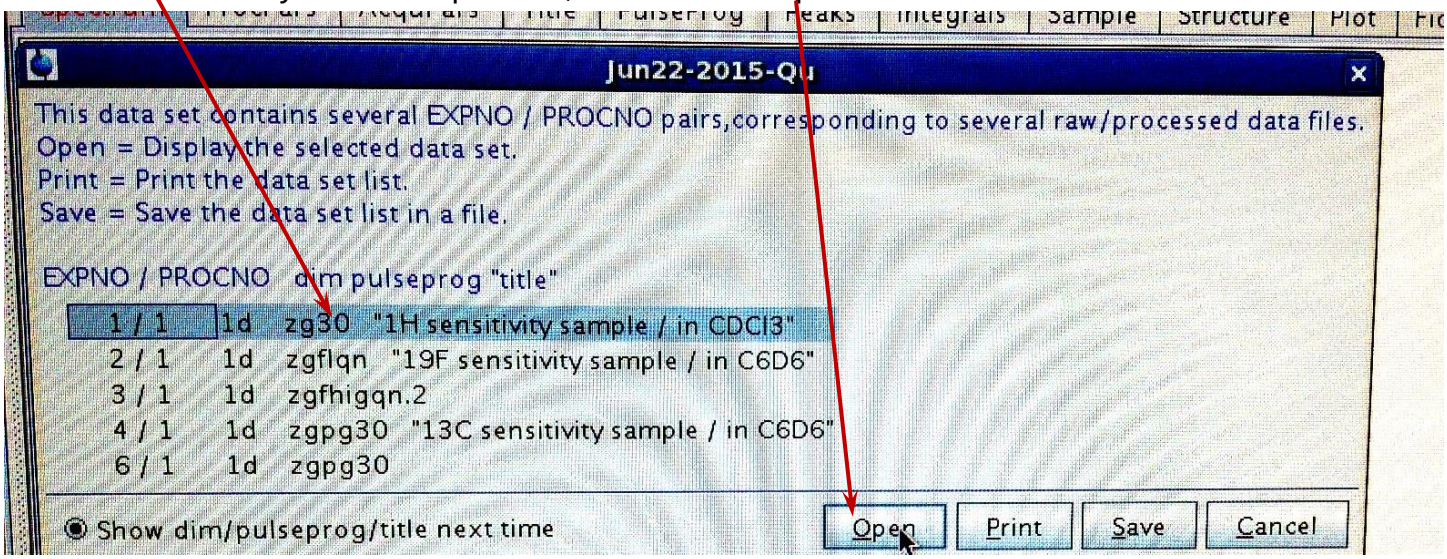
4. Data Processing

(1). Open Dataset, Dataset can be opened by **a)** or **b)**.

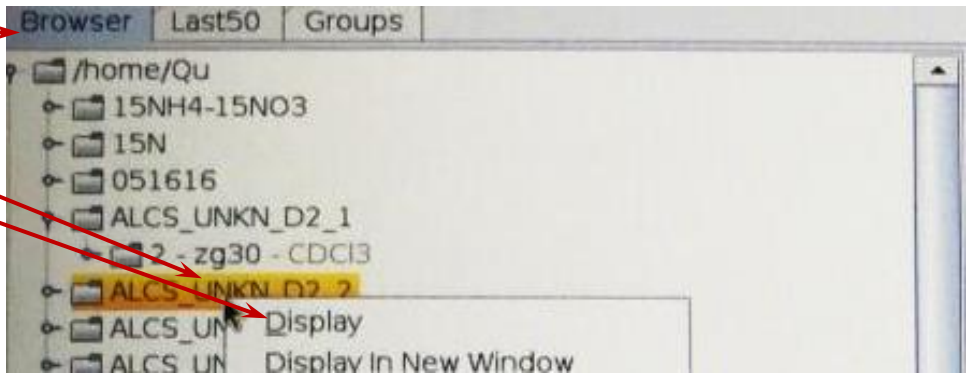
a). Clicking on the **Start** in the TopSpin Menu bar, then click on **Open Dataset**, your dir/files will appear



select dir/file which you want to process, then click on **Open**



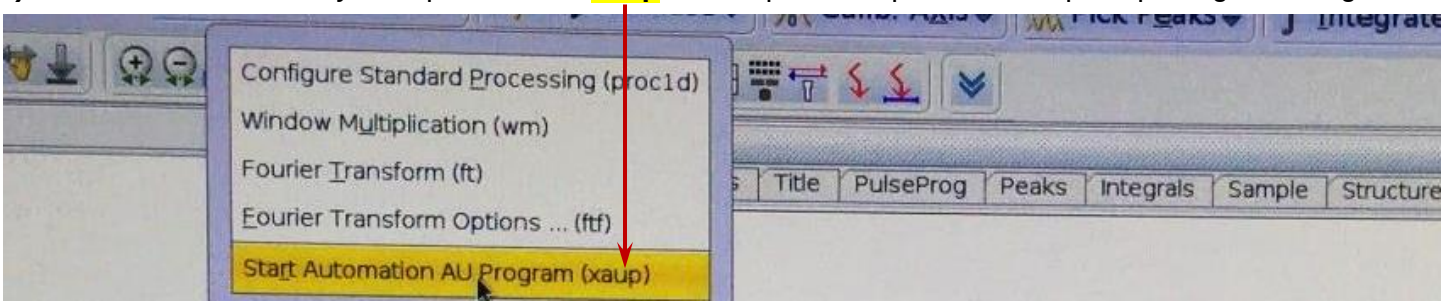
b). click on the **Browser**, locate your data and right-click on a dataset name, and choose **Display** from the drop down menu



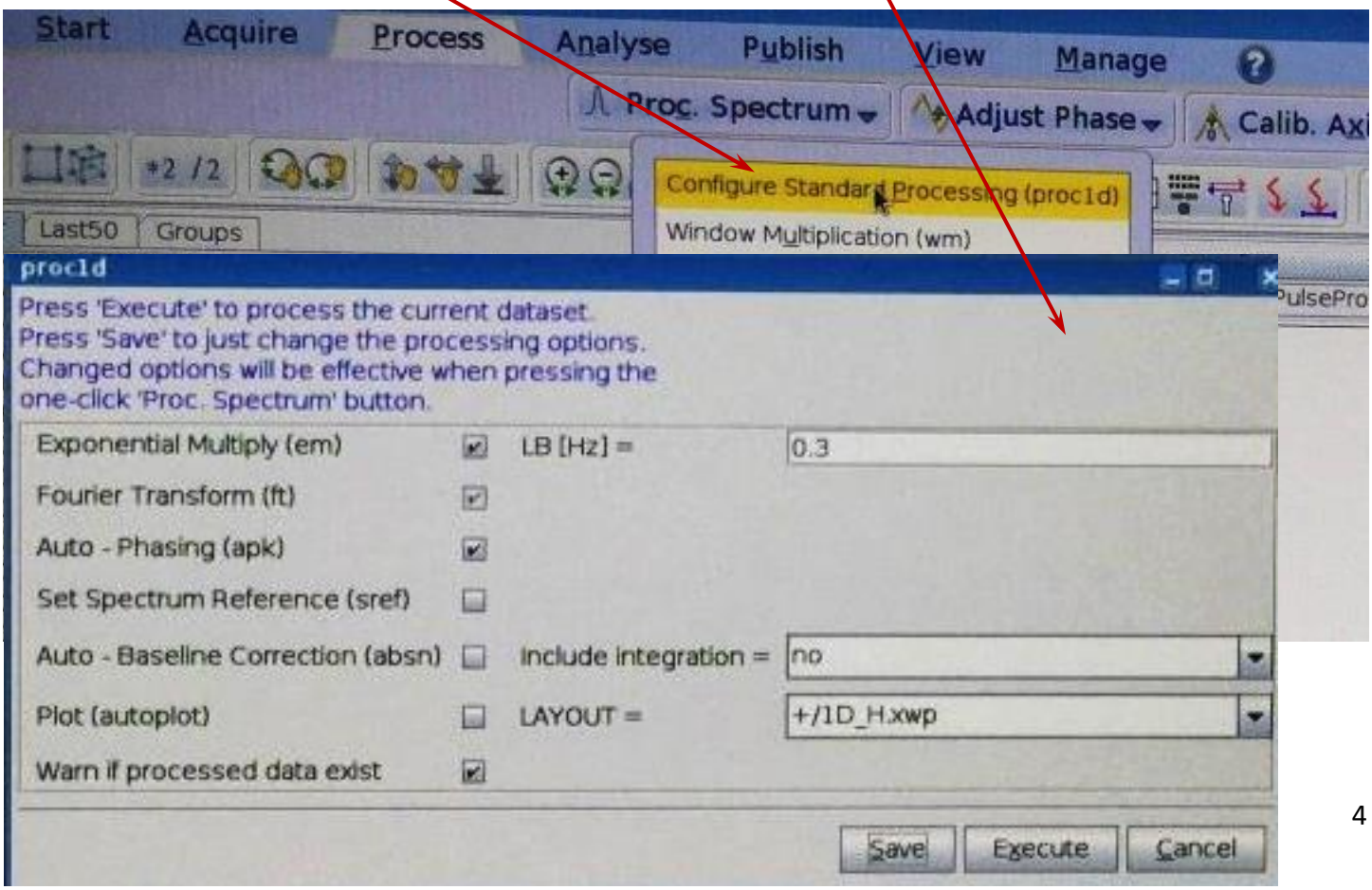
(2). Process Spectrum,

Click on the **Process** in the TopSpin Menu bar, then click on **Proc. Spectrum** there are a few options to choose from the drop-down menu

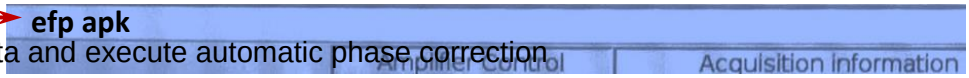
a). Auto Process, for easy and quick, Select **xaup** to auto process spectrum with peak picking and integration.



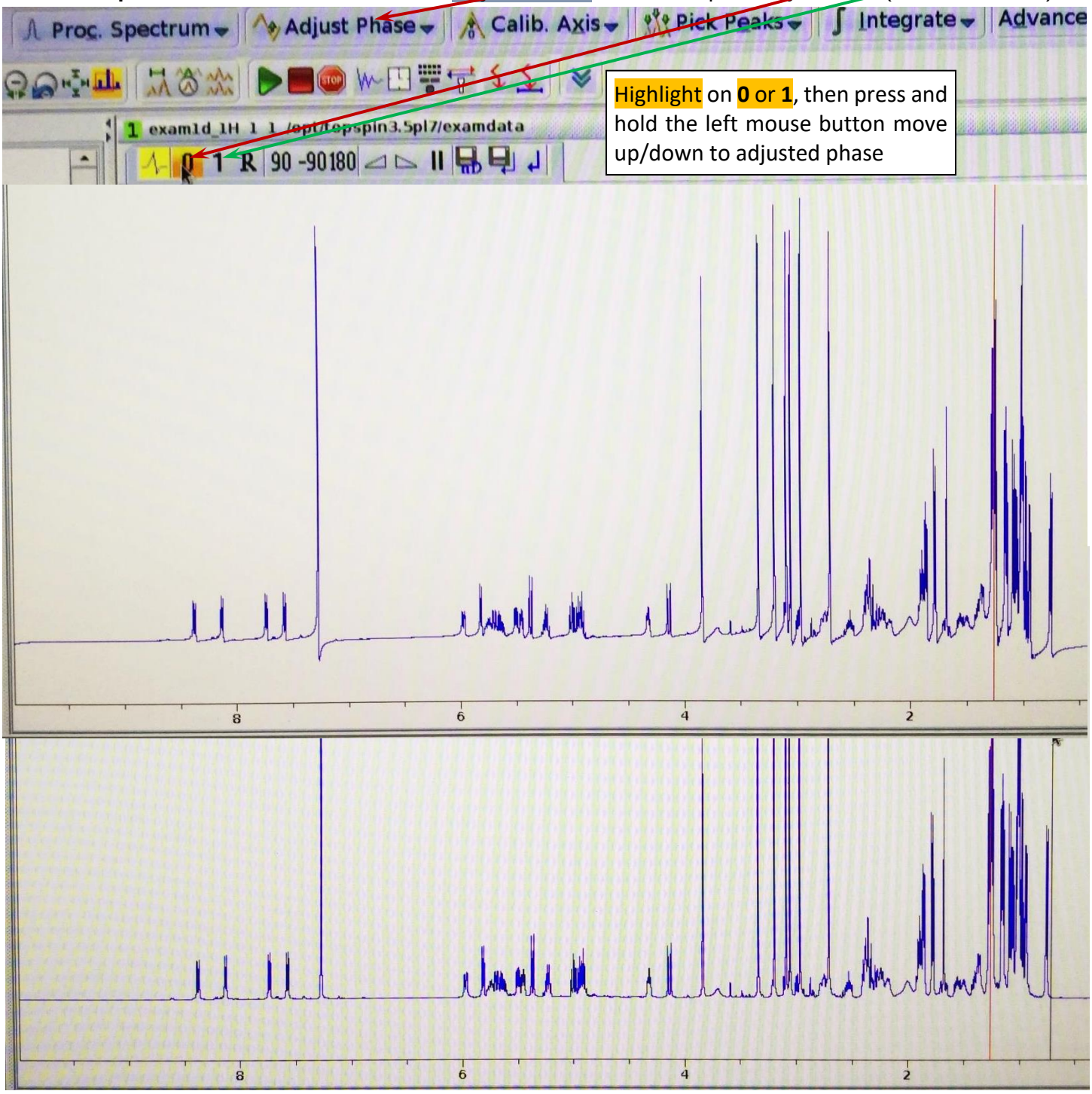
b). Standard Process, Select **proc 1d** (2d, 3d), then fill in drop-down table to process spectrum



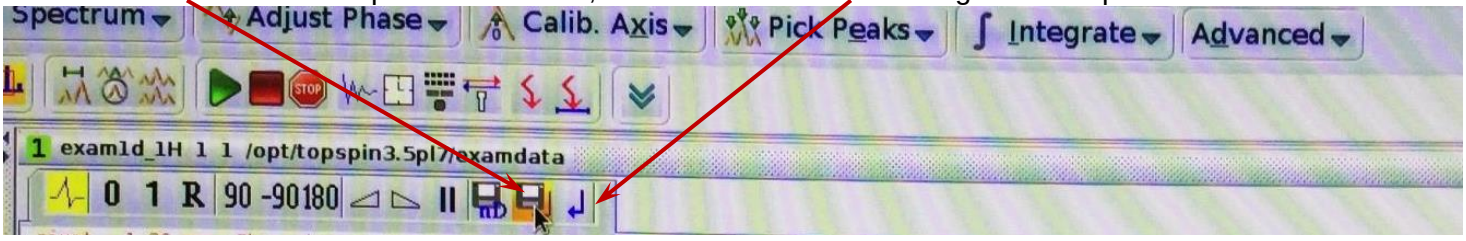
c). Manual Process, type **efp apk** in the command line to process data and execute automatic phase correction



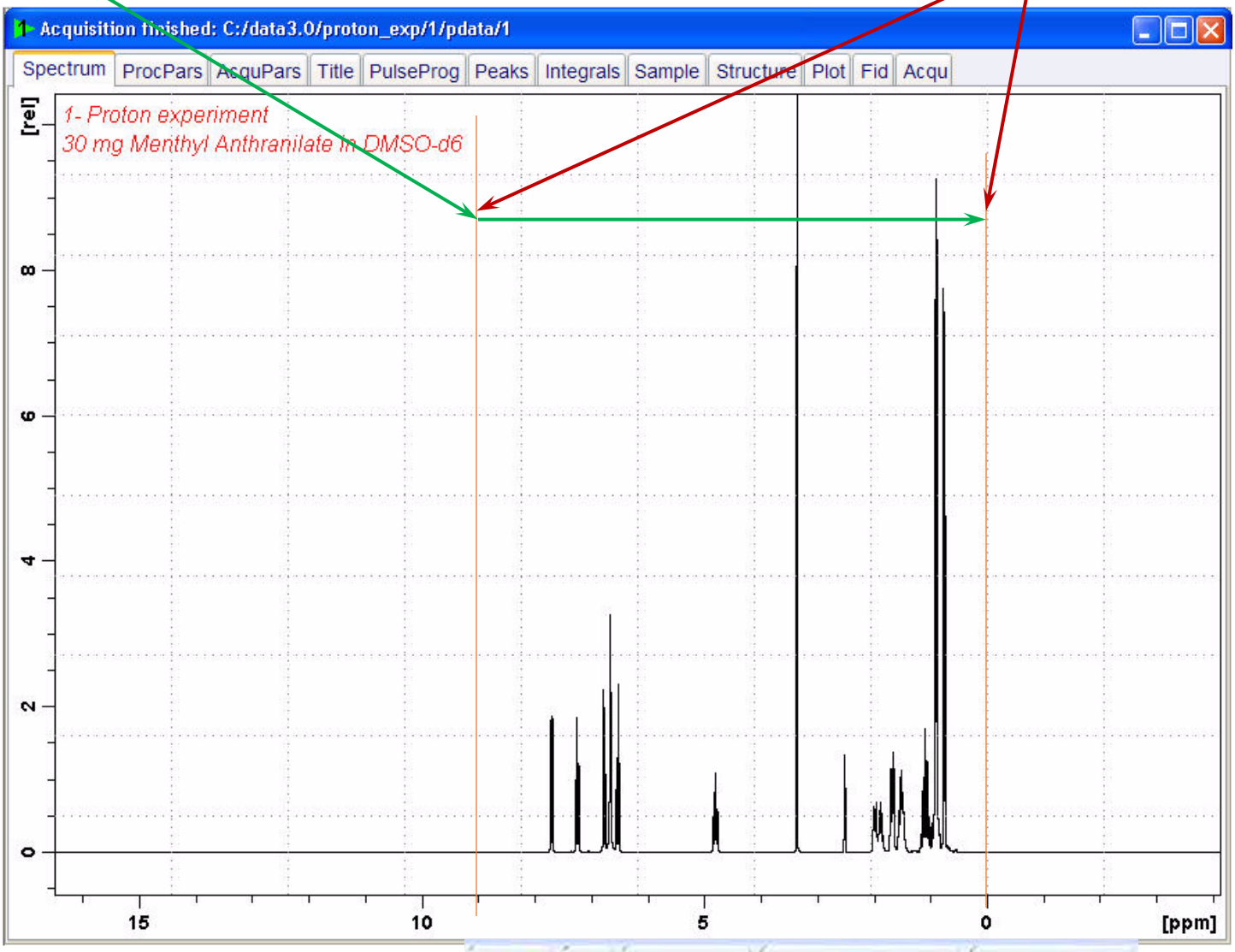
(3). Phase Correction. The simplest procedure is automatic phase correction which is implemented using the command **apk**. For more corrections, click on **Adjust Phase** for manual phase by **0** and **1** (0 and first order)



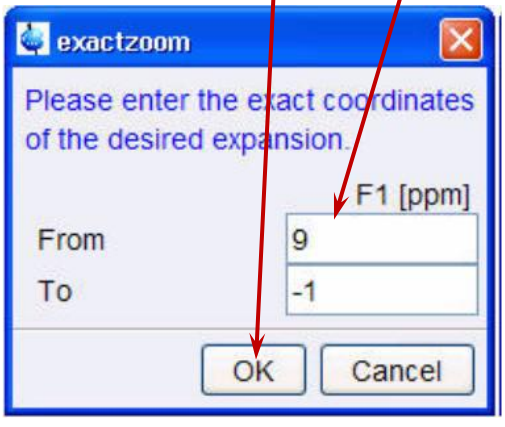
Click on disc icon to save phase correction, then click on return icon to get out the phase section.

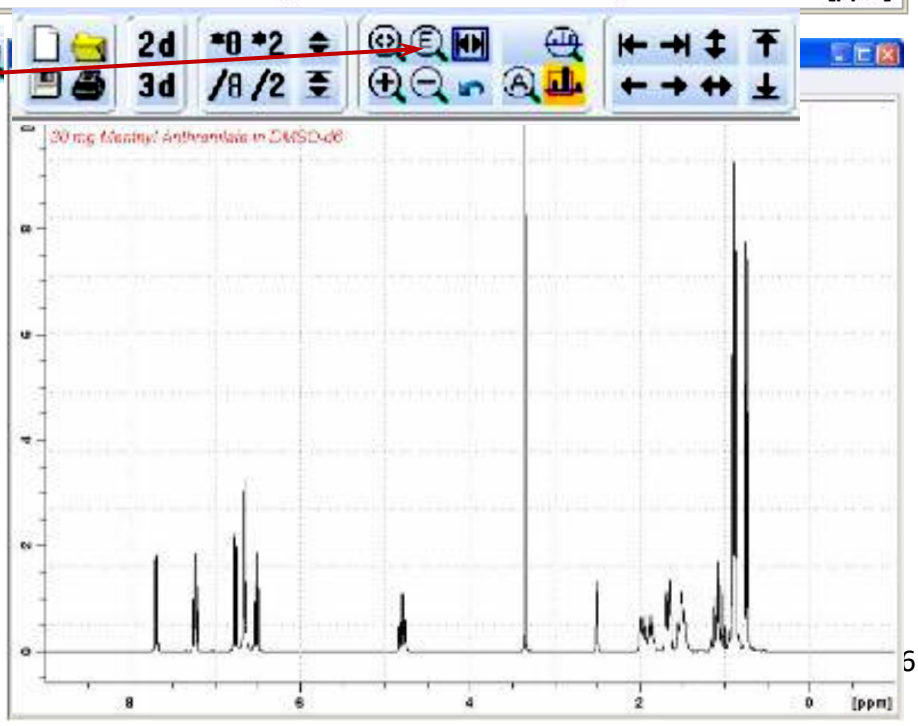


(4). Optimizing the Spectral Width. To display a specific region of your spectrum hold the left mouse button and drag your mouse over the region of interest.

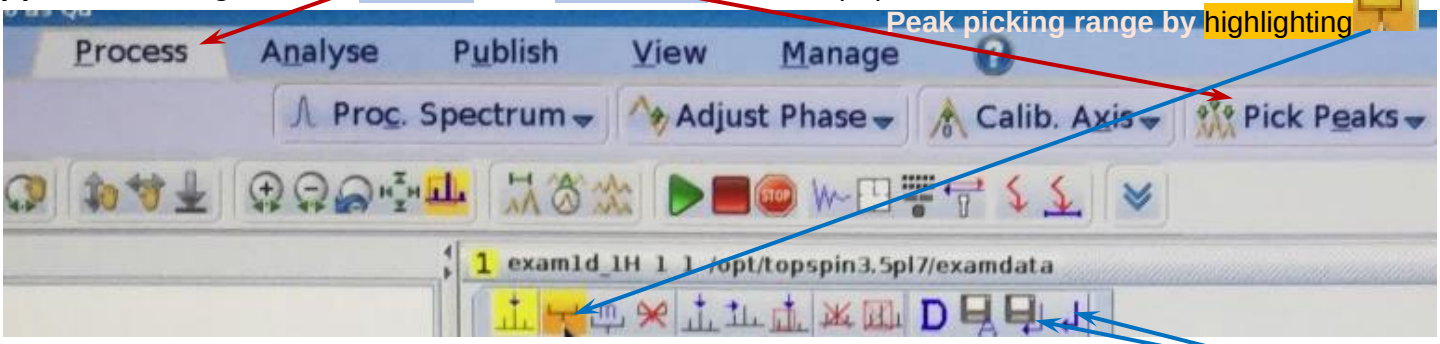


For exact chemical shift range, click  and specify the range you wish to display. For example, to display the region of the spectral window from 9 to -1 ppm, put 9 and -1 in the **From** and **To** fields, then click OK.

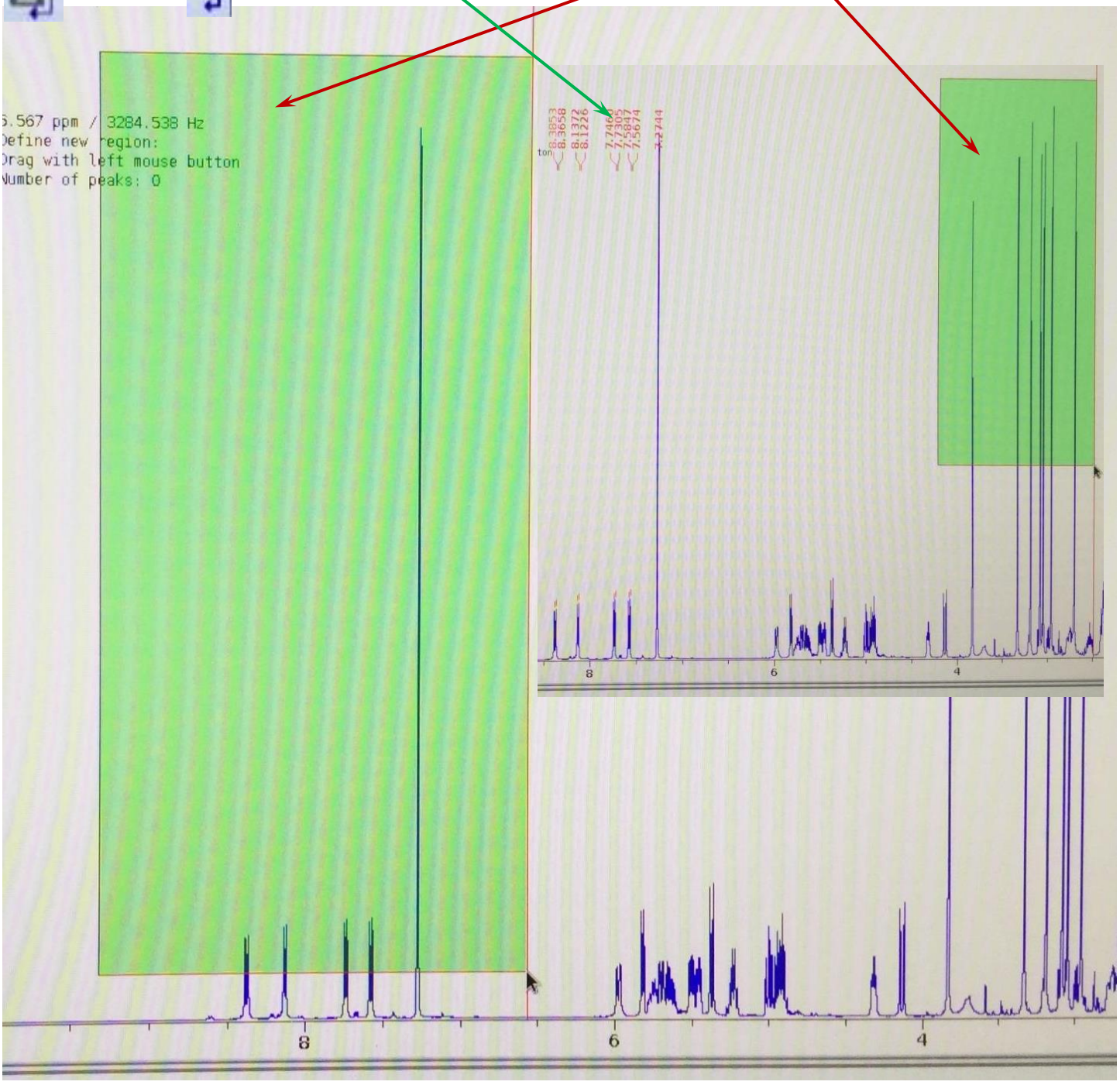




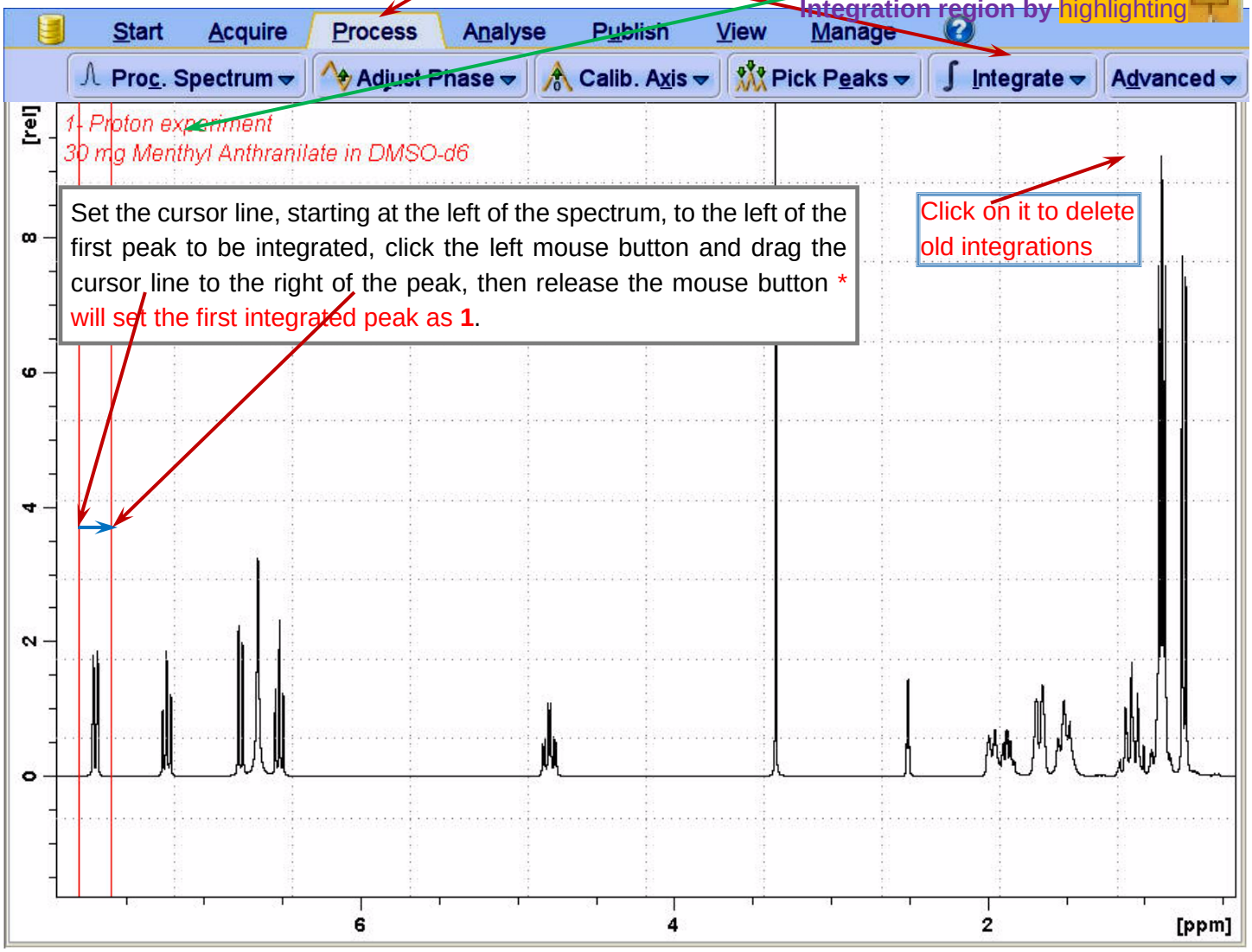
(5). Peak Picking. Click on **Process,** then **Pick Peaks** in the TopSpin Menu bar. Click on to define range

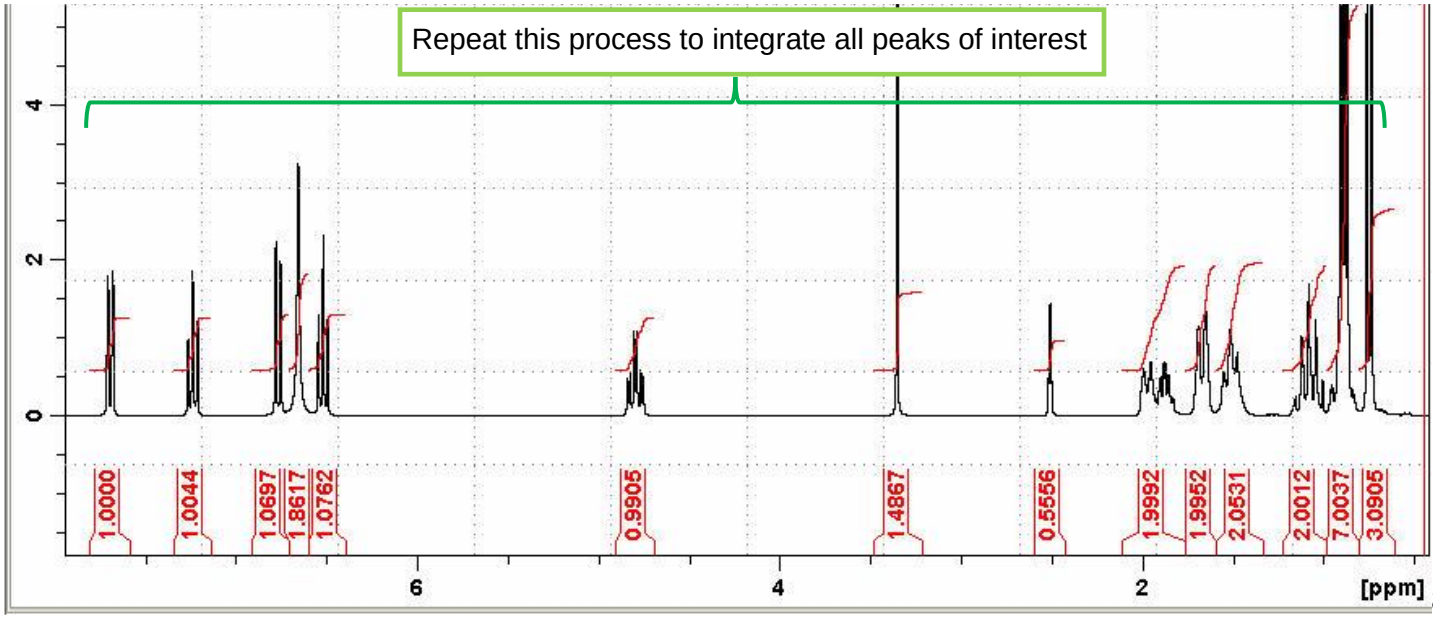


Press and hold the left mouse button over the peaks of interest. A green highlight will form, release the left mouse button, all peaks in green area were picked. Repeat this process to pick all peaks of interest. Then click to save and to get out the mode



(6). Manual Integration. Click on **Process**, then Click on **Integrate** and to define integration region. Integration region by highlighting

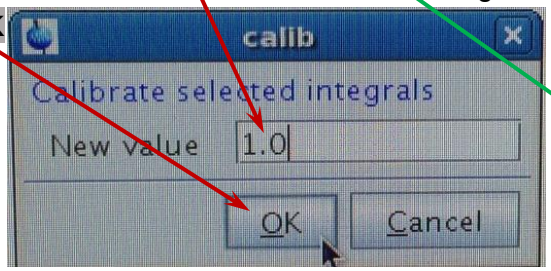


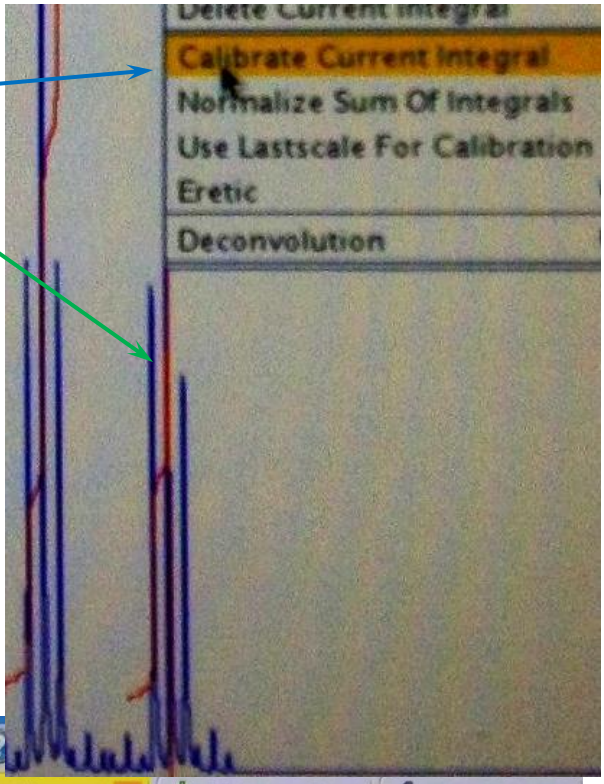



click on to save and get out the mode

Option for set integral reference

Place the cursor within the integral label peak and press the right mouse button. Select **calibrate Current Integral** on popup menu. Enter the desired value of the selected integral in popup box, then **OK**



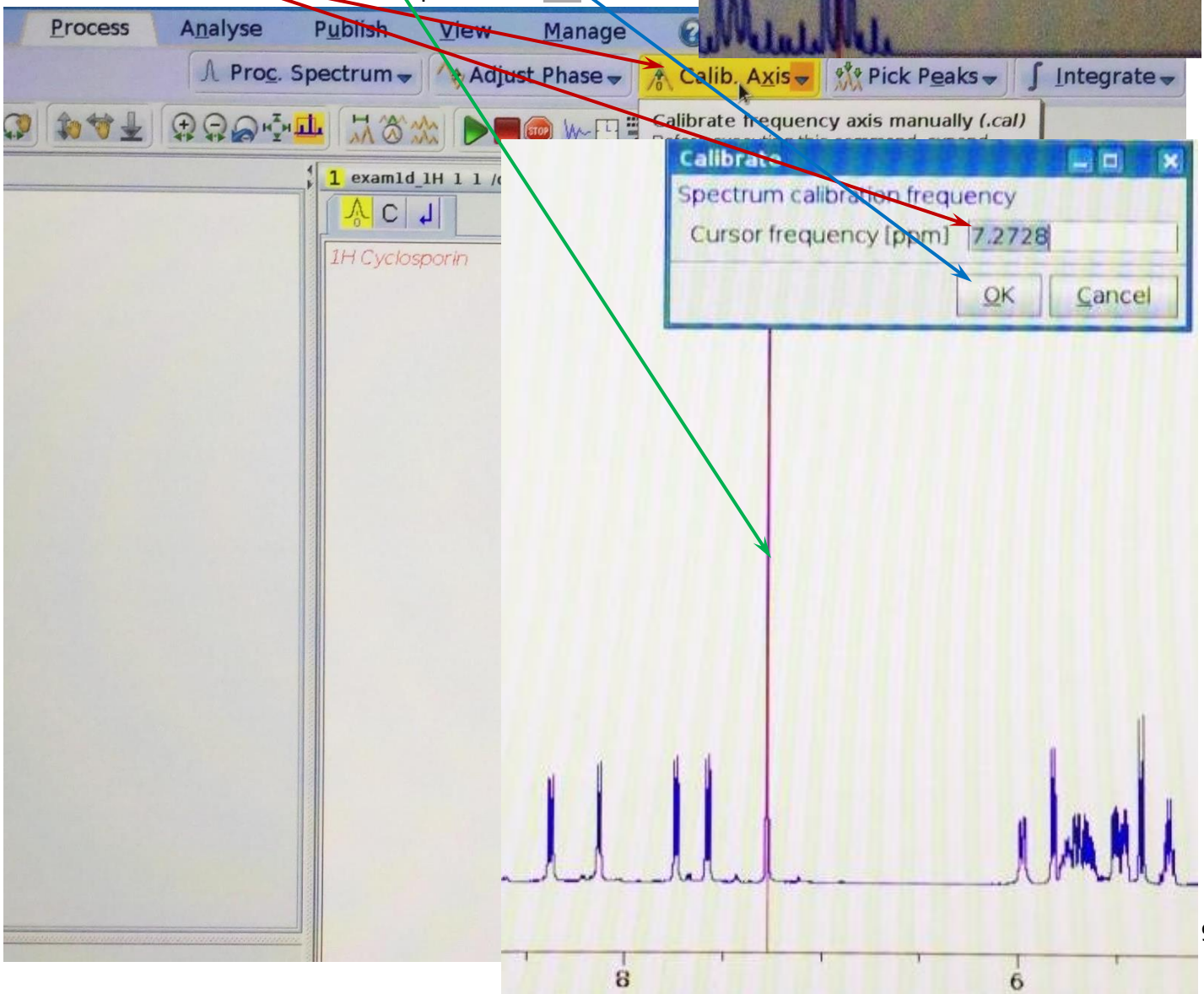


Click on  then click to save and out the mode

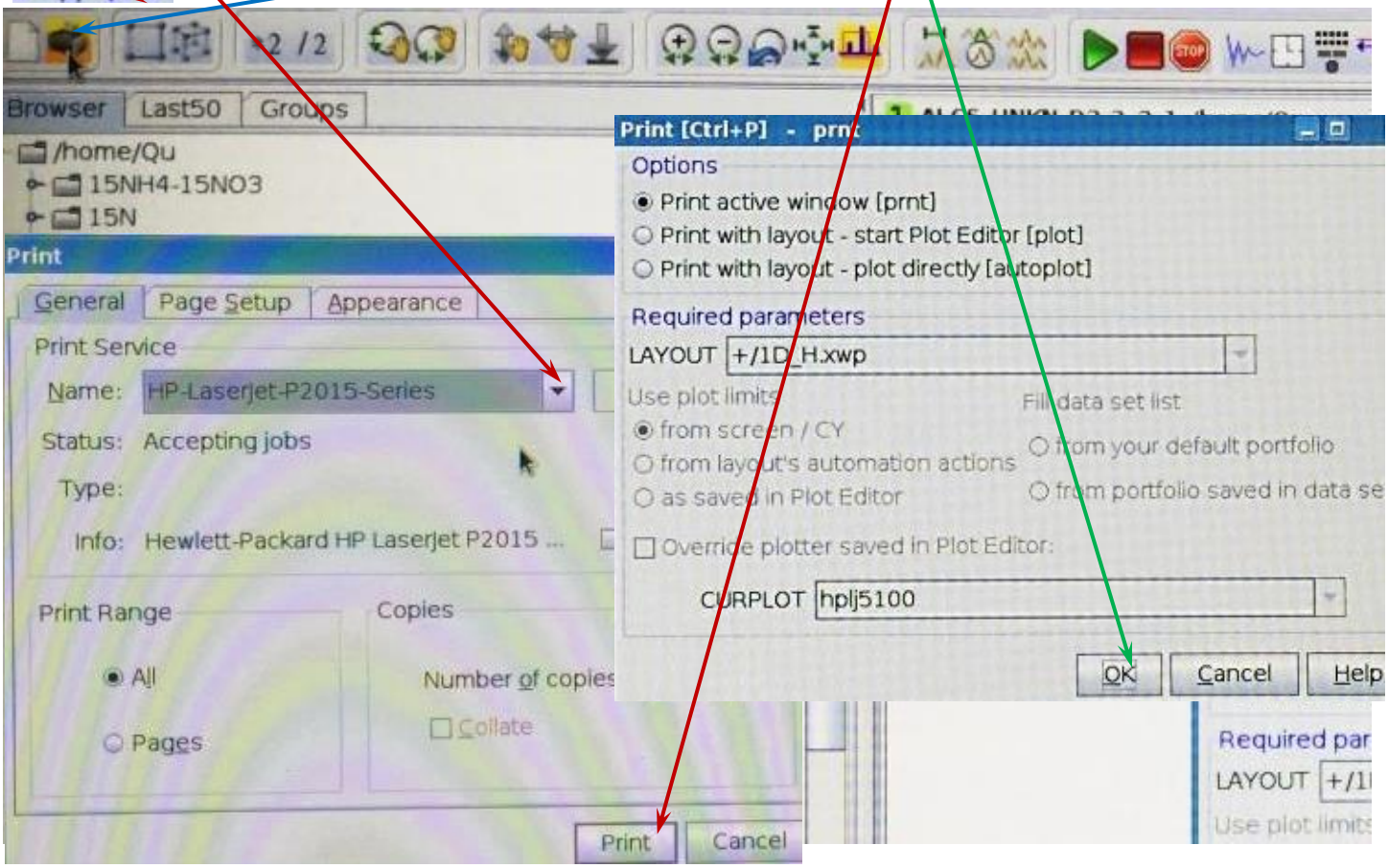
***chemical shift auto set to lock solvent**

Option for set peak chemical shift reference

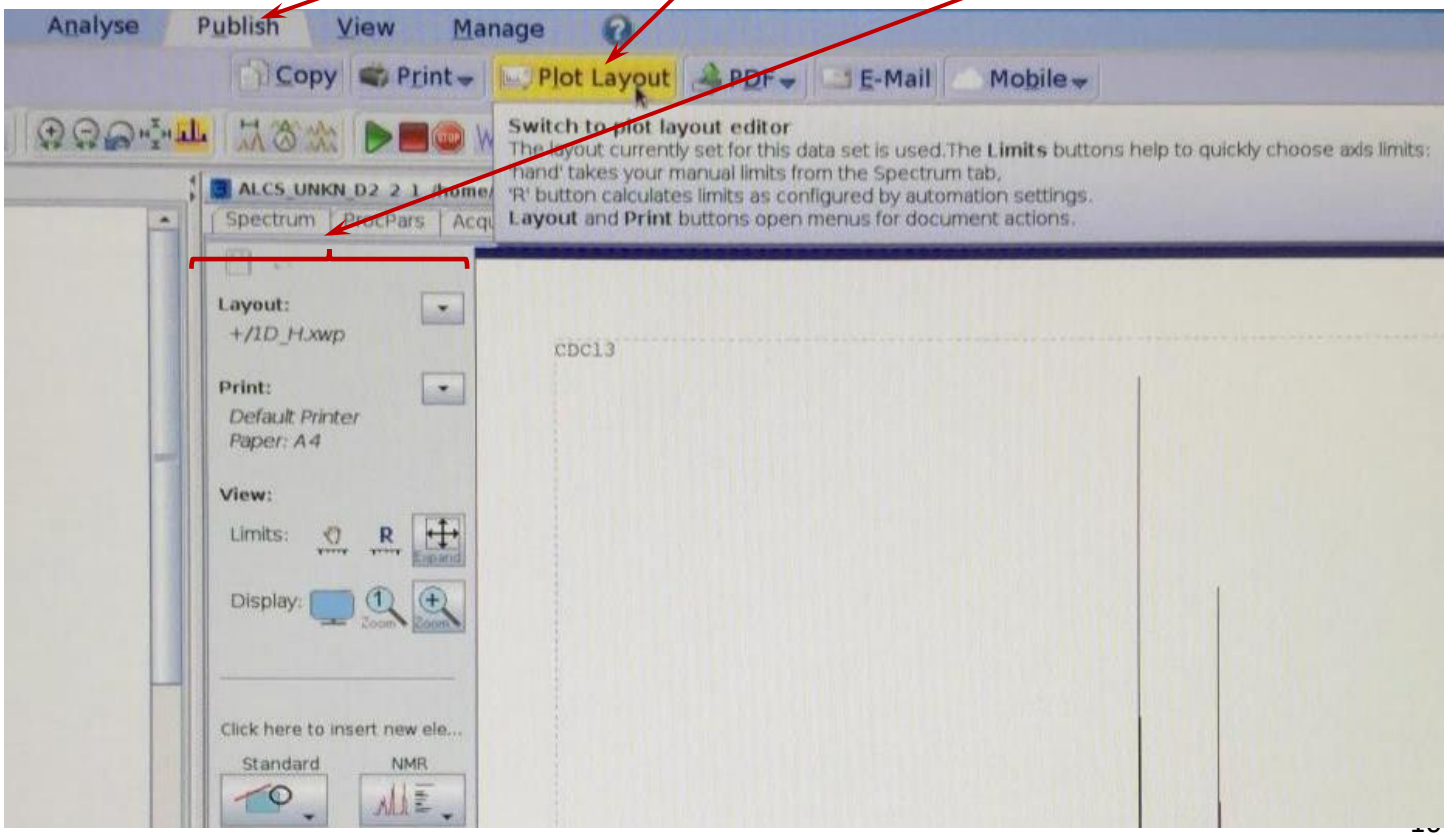
Click **Calib. Axis**, then click the peak to reference.
Enter the desired chemical shift of the peak, click **OK**.



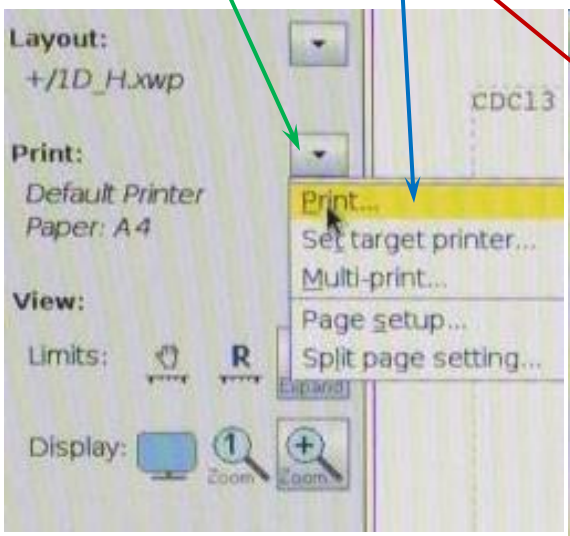
(7). Plotting **a). Quick print**, click on printer icon, then click **ok** on drop-down box, another Print box p[illegible] to select **HP-Laserjet-P2015** in **Name**, then click on **Print**

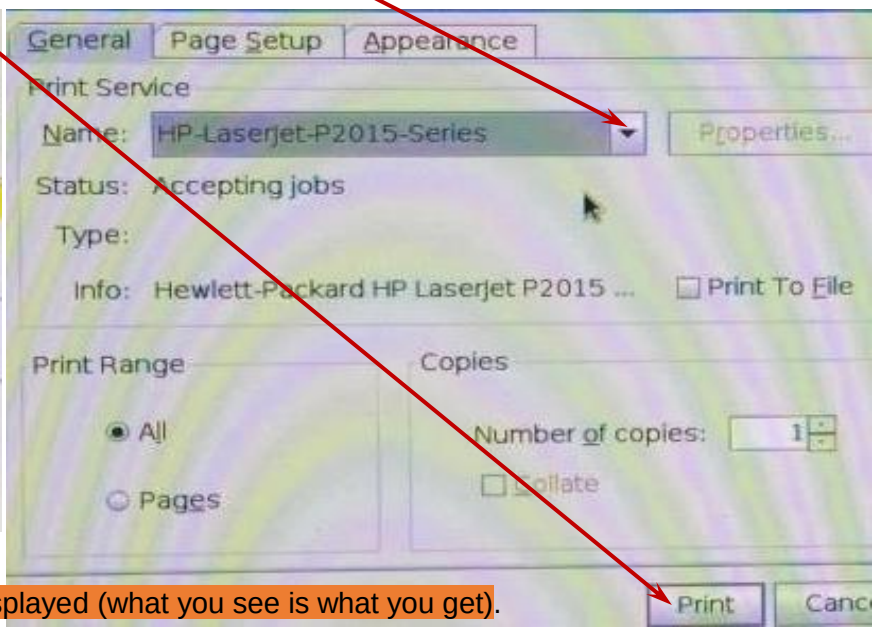


b). Plot Editor, click on **Publish**, then click on **Plot Layout**. Editing by a panel at the left (active/select menu)



Print, click on ▼ and select **Print…** on drop-down list, click on ▼ to select **HP-Laserjet-P2015-Series** in **Name** on popup Print box, then click on **Print**

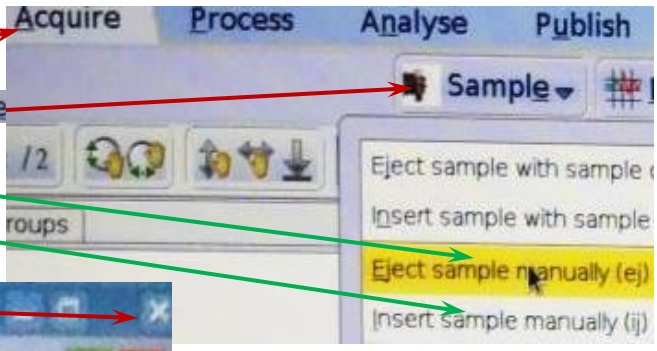




or type ***prnt*** in the command line, it will automatically print your spectra as displayed (what you see is what you get).

5. Close TopSpin

(1). Exchange Sample Tube, click **Acquire**, and select **eject sample manually (ej)** on drop-down menu take sample tube out and place the sealed CDCl3 sample tube back in, then click on **Insert sample manually (ij)**.

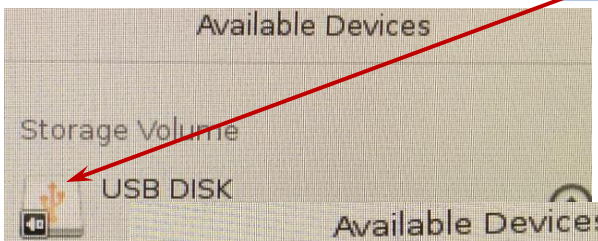


(2). Close, Click X on the top right corner to close TopSpin window

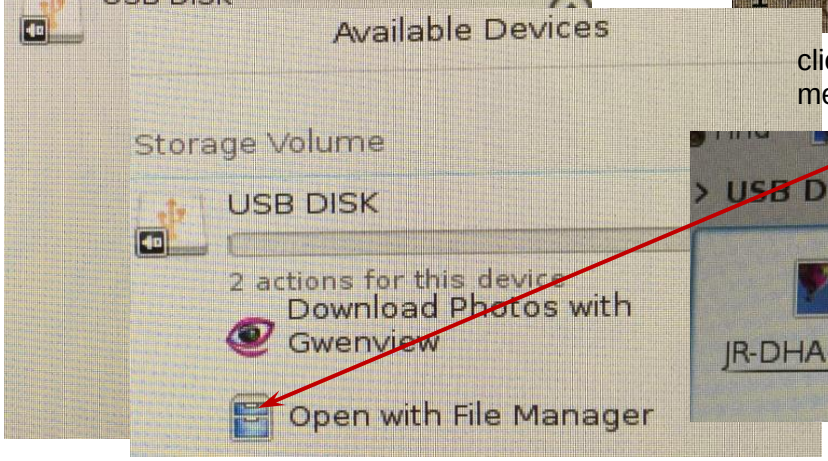
6. Copy/Transfer Files to USB Flash Drive

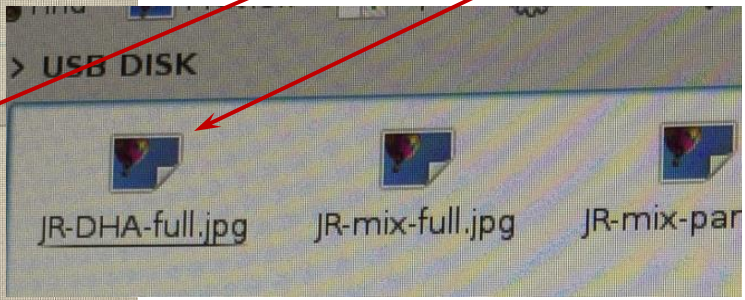
(1). Open USB, insert your USB flash drive to PC,

icon appearing on bottom right corner, click on it, Available Devices window pop up, click on **USB DISK**



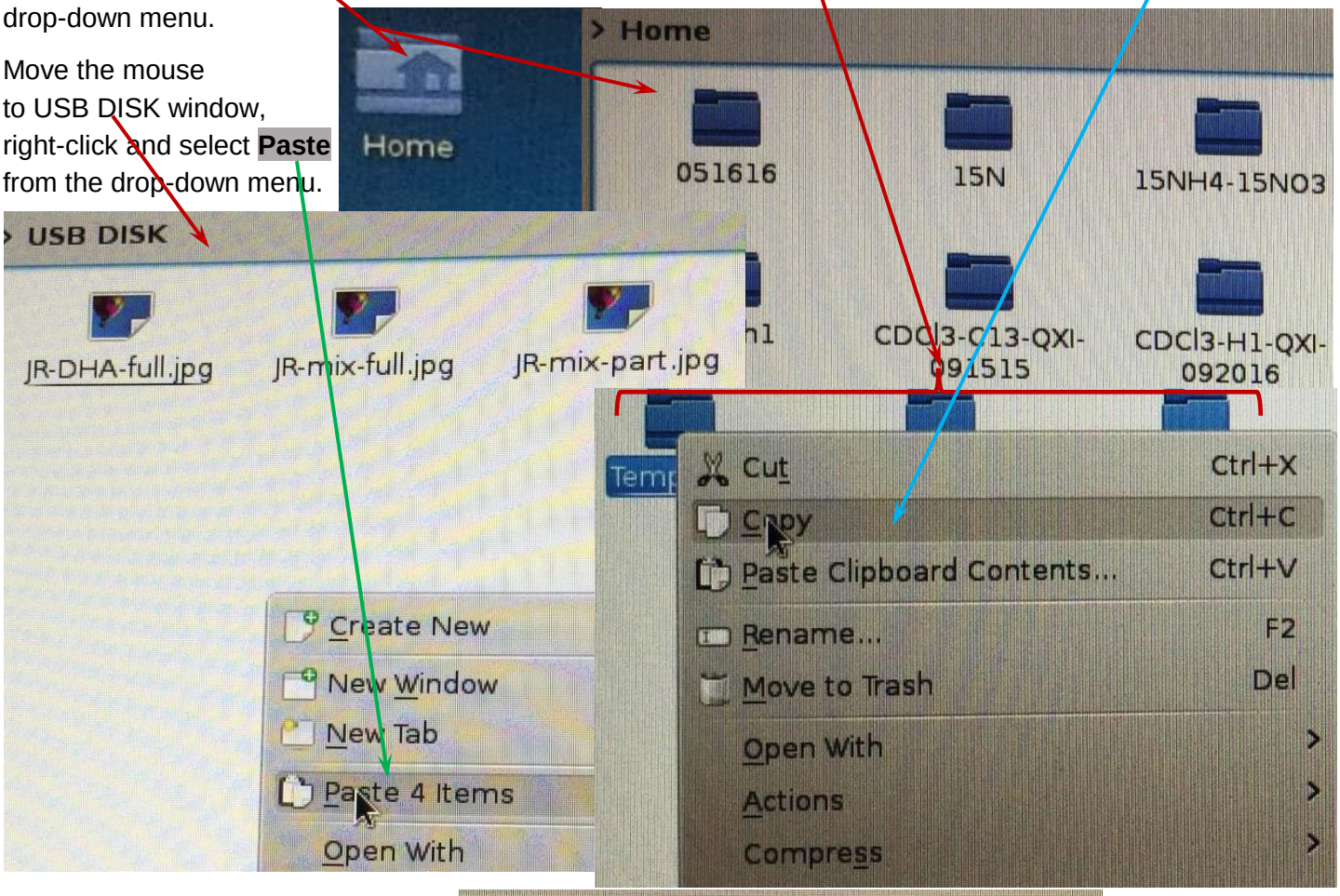
click on **Open with File Manager** on drop-down menu to open the USB, the USB drive open





(2). Copy Files, click **Home folder** on desktop, then select the file/files, right-click and select **Copy** from the drop-down menu.

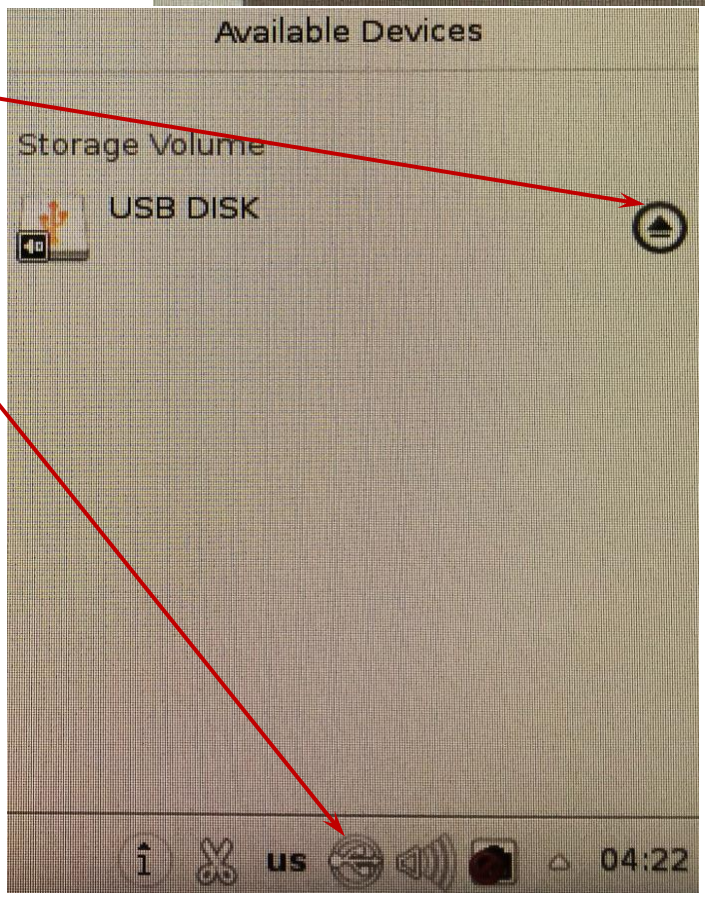
Move the mouse to USB DISK window, right-click and select **Paste** from the drop-down menu.



(3). Unplug USB,

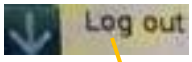
After copy files, click on  to unmount the USB flash drive,

After  icon disappear, the USB drive can be removed.



7. Logout

Click on bottom left corner  , more icons appear on the bottom bar, place pointer on 

, then select 

on popup icons on the left column to logout your account. The screen shows as