Basic NMR Operation

Guide for the Bruker AV-III 400 MHz NMR Spectrometer
Using ICON NMR in Automation

Dr. Yun Qu update 05/15/2024

Before You Start:

☞ NMR tube must be at least 7 inches in length and must have a top that is neither broken nor cracked. If your tube is shorter than 7 inches, it will not be grabbed fully by the sample changer and may fall and break on the floor or on the top of the magnet. Tubes with chipped tops may be broken by the sample pincers, which grab the tube very firmly close to the top. **Any tube shorter than 7 inches or with a chipped top is not allowed**. Please get the NMR tubes from the Chemistry Stockroom.

☞ NMR tube must not have any label (paper, tape, sticker etc....) attached to it. This will stop the sample pincher from working properly and may result in sample breakage and equipment damage. Tubes can be labeled on the glass or the cap with permanent marker. **Any tube with an attached label submitted to the sample changer is not allowed.**

☞ **The sample changer will not handle sample tubes with screw caps or vacuum valves. They will cause equipment damage**.

Approximate amount of middle-size compound (MW ~ 500) advisable for running following experiments:

^{1}H NMR, ^{19}F NMR, ^{31}P NMR experiments - about 3-5 mg

^{13}C NMR short run experiment (0.5-1 hr)-about 20-50 mg; long run experiment - about 5-10 mg

~0.6 ml of NMR solvent is appropriate for the right solvent level in NMR tube. Unsuitable solvent level can lead to a bad shimming result and horrible looking spectrum.

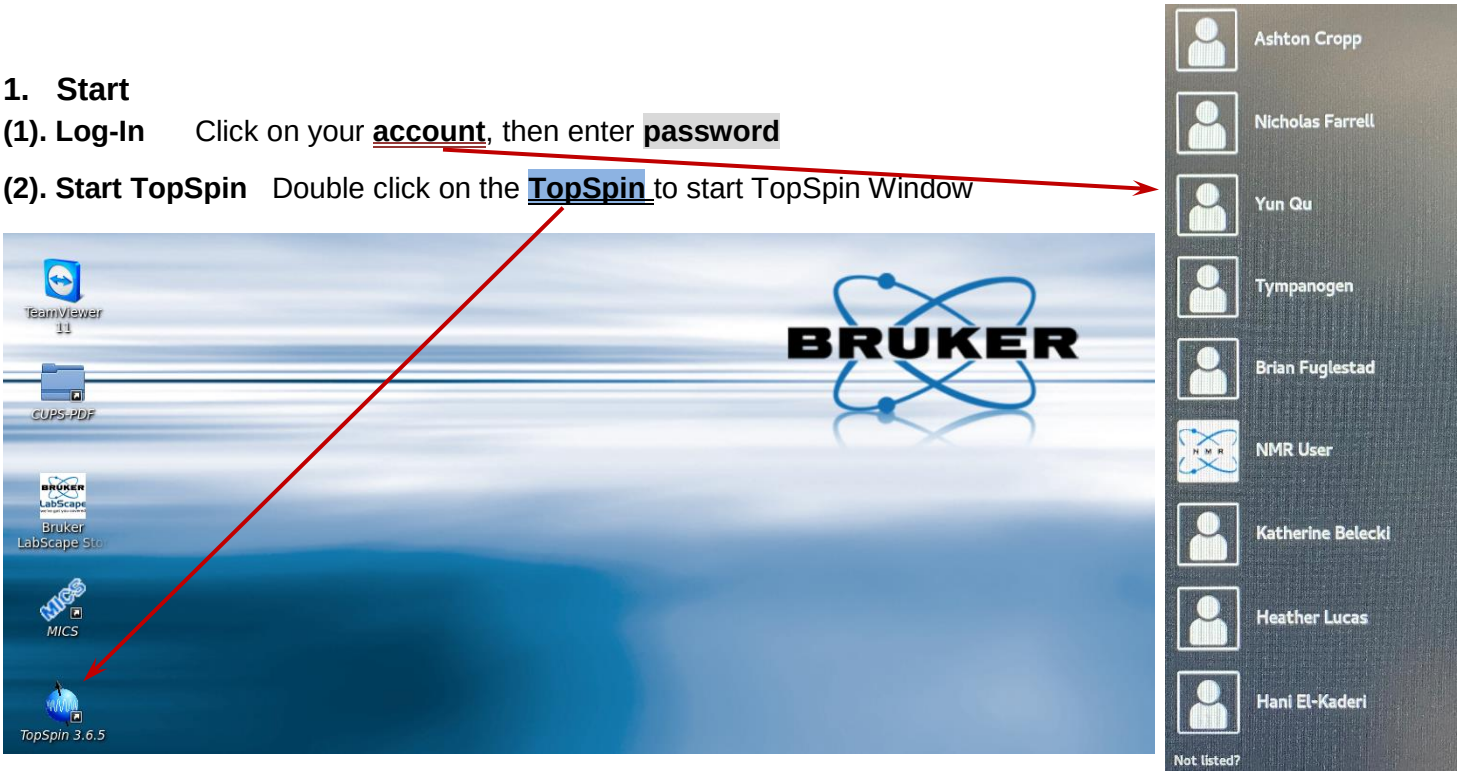
⊘ **Never lean or exert any force on the sample changer or magnet**. This may cause a magnet quench resulting in many weeks of down time and hundreds of thousands of $$ being spent.
Users are asked to report improper sample tubes to the NMR Facility staff immediately in order to avoid needless down time and equipment damage!

☞ The 400 NMR uses the Sample Xpress Automatic Sample Changer, and it is extremely important that you pay attention to the **holder number** containing your sample, and to only remove **your** samples from your **holder number**, from the sample rack. It is possible that someone will have samples in the rack with longer experiments submitted in the Night Queue. You must be sure to not disturb samples left by other users. **Labeling your NMR tubes (**on the glass or the cap with permanent marker.**) is TRONGLY recommended**.

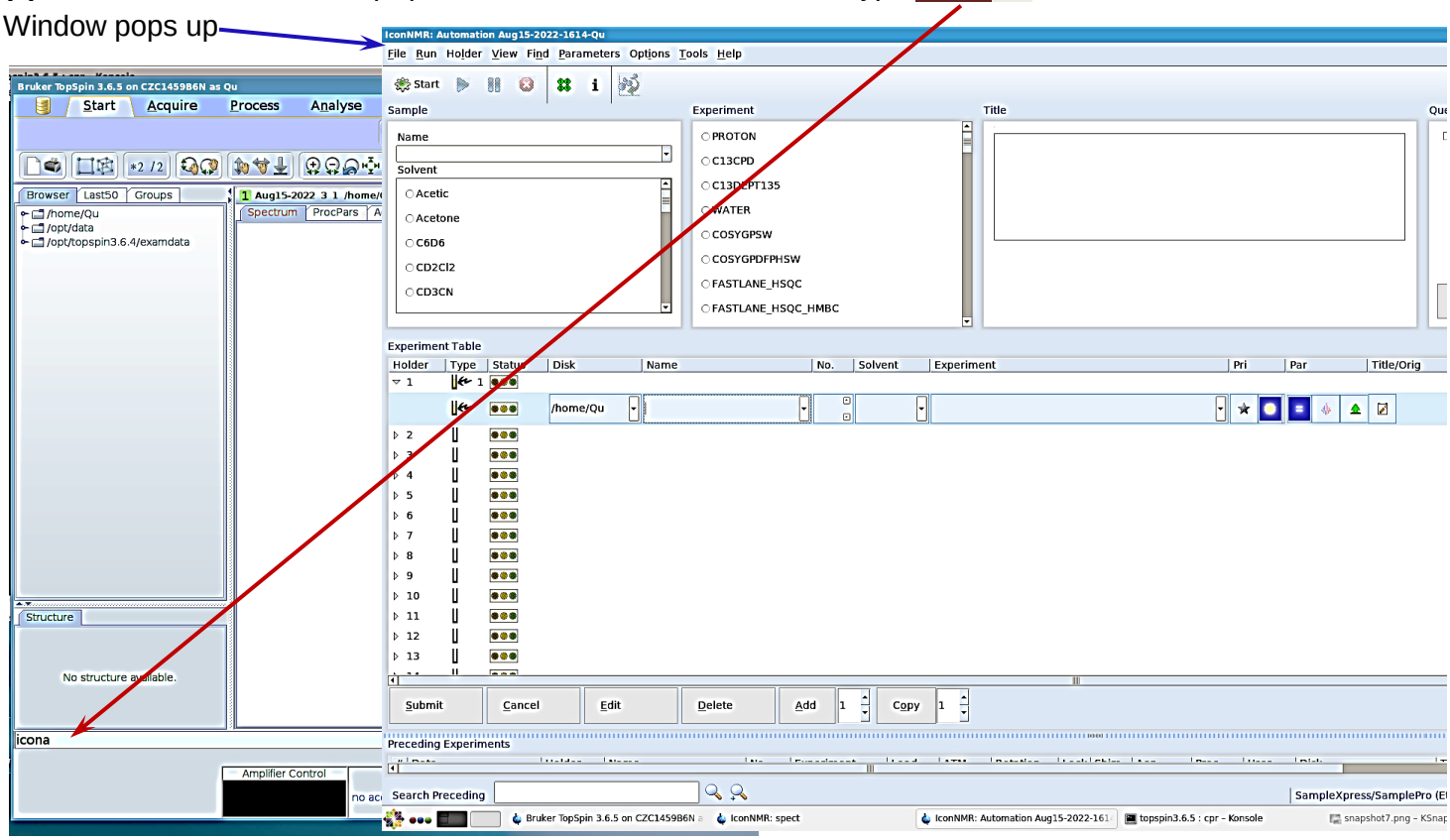
1. Start

(1). Log-In Click on your **account**, then enter **password**

(2). Start TopSpin Double click on the **TopSpin** to start TopSpin Window

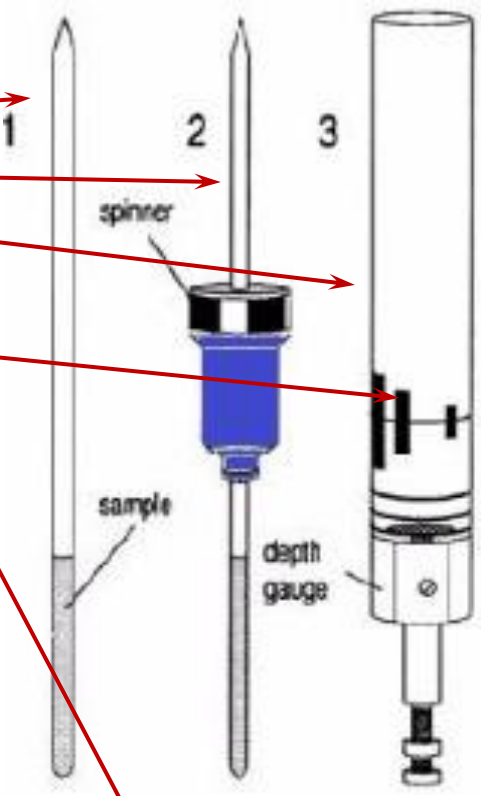


(3). Start ICON NMR On TopSpin Window**,** in the command line, type **icona** to start ICON NMR, ICONNMR Window pops up



2. Setup Experiment

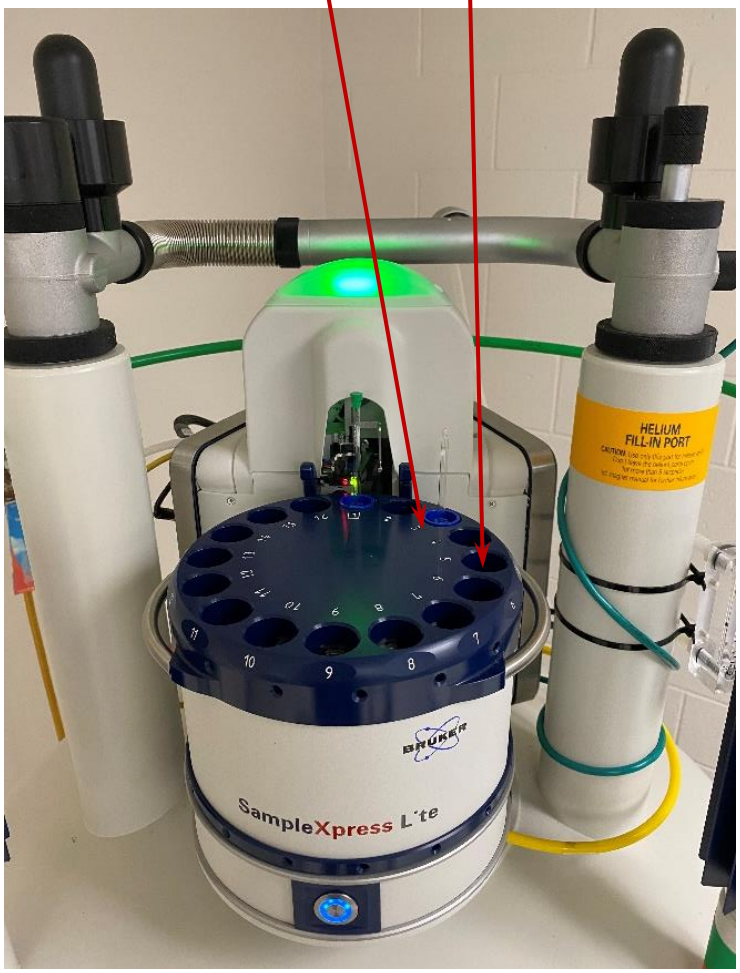
(1). Sample tube/s



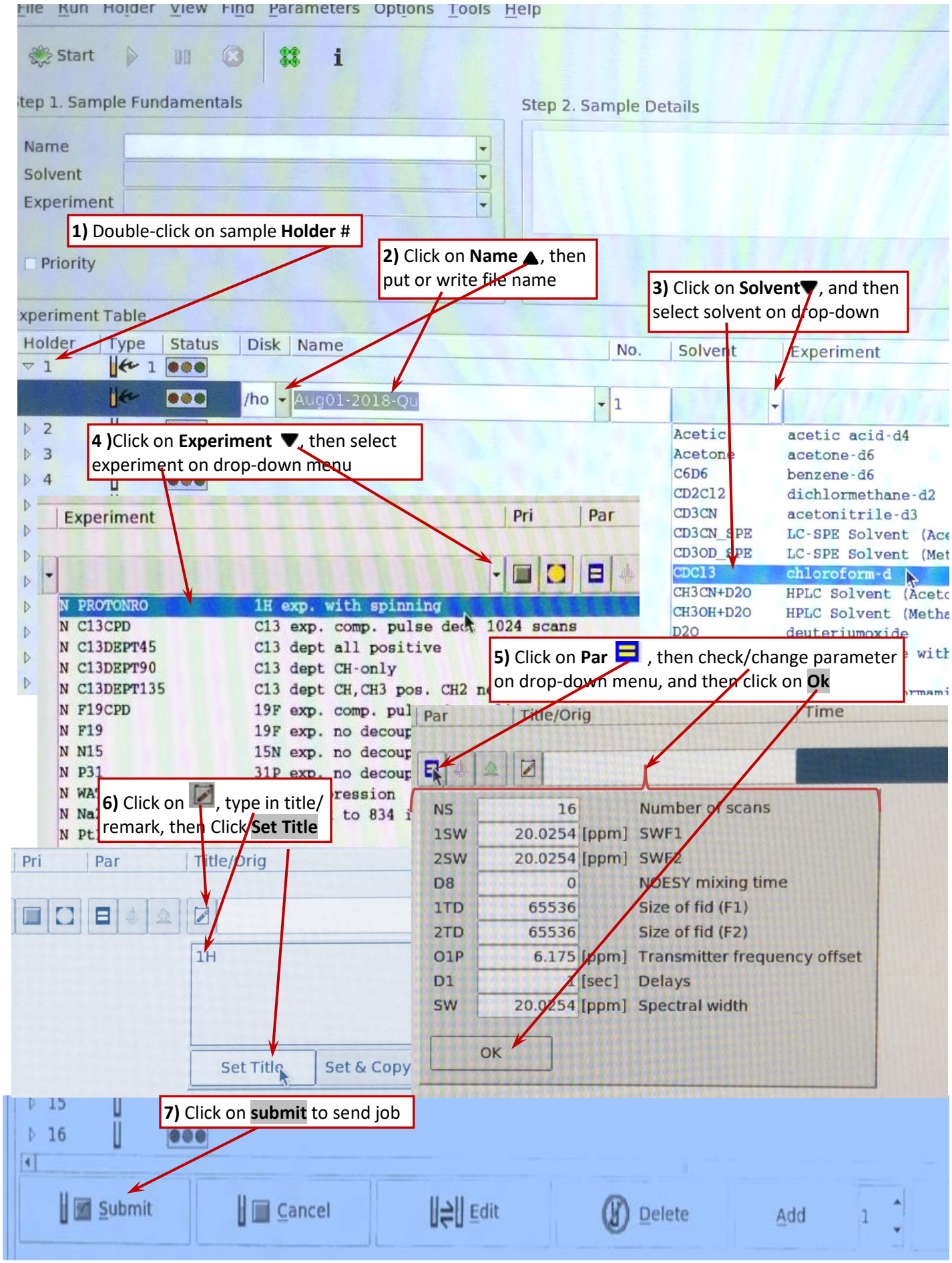
1) Wipe-off NMR tube **1** with kimwipes
2). Insert sample tube in the **spinner** **2**
3). Place the sample with the spinner into Bruker **depth gauge** (add more sample liquid or solvent to the NMR sample tube if the liquid is below the center bar, mark as 3/5/8mm as the liquid in tube must be above the center bar).

! The position of Bruker depth gauge has been calibrated; changing the depth /position will affect the NMR spectrum quality and can break the NMR sample tube in the NMR magnet that will damage the NMR probe! Not only cause to shut down the NMR instrument, but also cost $$$ to repair it!

4). Place sample tube/tubes in the holder position at the SampleXpress Lite Sample Changer **carefully** (place all samples once at the beginning).

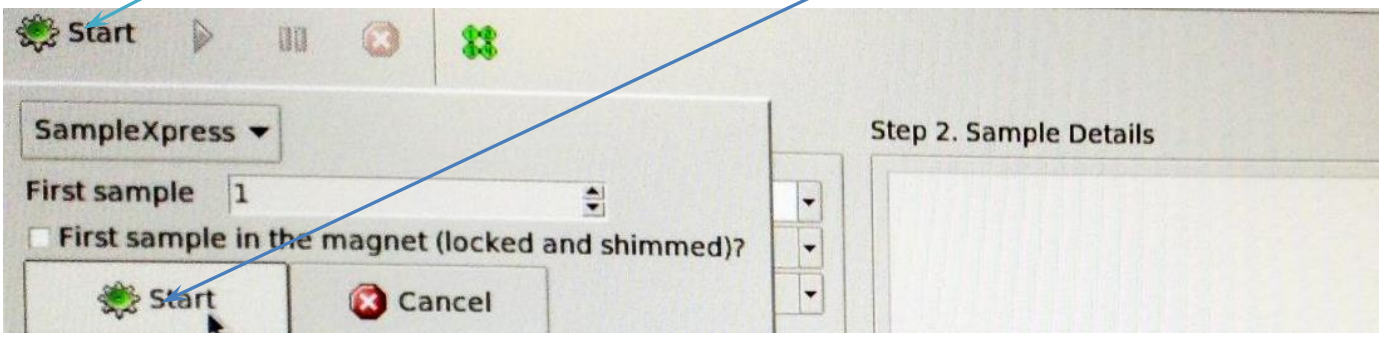


(2). Setup ICON NMR, steps 1-7



3. Start NMR experiment

On the first sample click on **Start** on the left top corner button**,** and then click on **Start** on popup box. The status indicator will turn **green** (**do not interrupt the running when the Green light is on, will cause many problems!**). (You can also start experiment after all samples/experiments are setup, and click on the sample # that you want to start first)



Summarize the perform Icon NMR:

1). *Double-Click* in the row of **Holder** #. The row will "open up".

2). *Click* on the **Name**, then put /write file name.

3). *Click* on the **Solvent**, a pull-down menu will appear, then select a solvent from the pulldown Menu.

4). *Click* on the **Experiment**, select the experiment from the pulldown Menu.

5). *Click* on the icon, a dialog box popup, enter the new value to change basic parameters, then *click* [OK].

NS is the number of scans; D1 is the relaxation delay

SW is the spectral width in PPM; O1P is the center of the spectrum

6). Click on , the dialog box popup, add "title or remark", then Click [**Set Title**] when everything is entered.

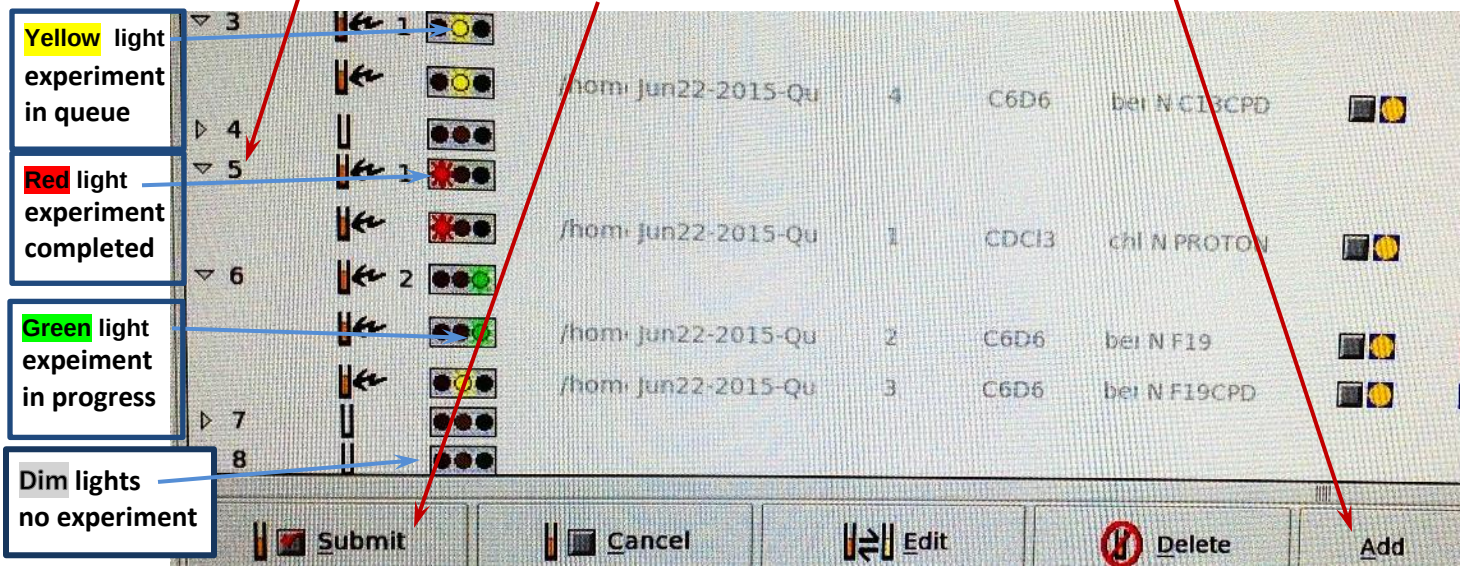
7). Double-Check that everything in the row is correct, confirm that sample is properly placed in the correctly numbered holder in the sample-change. Then click the Submit , the status indicator will turn yellow.

8). Click on **Start** on the left top corner button, then click on **Start** on popup box. The status indicator will turn **green**. Once **green**, SampleXpress Lite Sample Changer will insert the sample, tune the probe, lock on the solvent, shim, and proceed with your experiment. **Do not interrupt the running when the Green light is on, will cause many problems!**

Start when the first sample is setup or skip step 8 now to setup all samples/experiments, and then click on the sample # you want to start first, then do step 8.

*** Add another experiment for the same sample:**

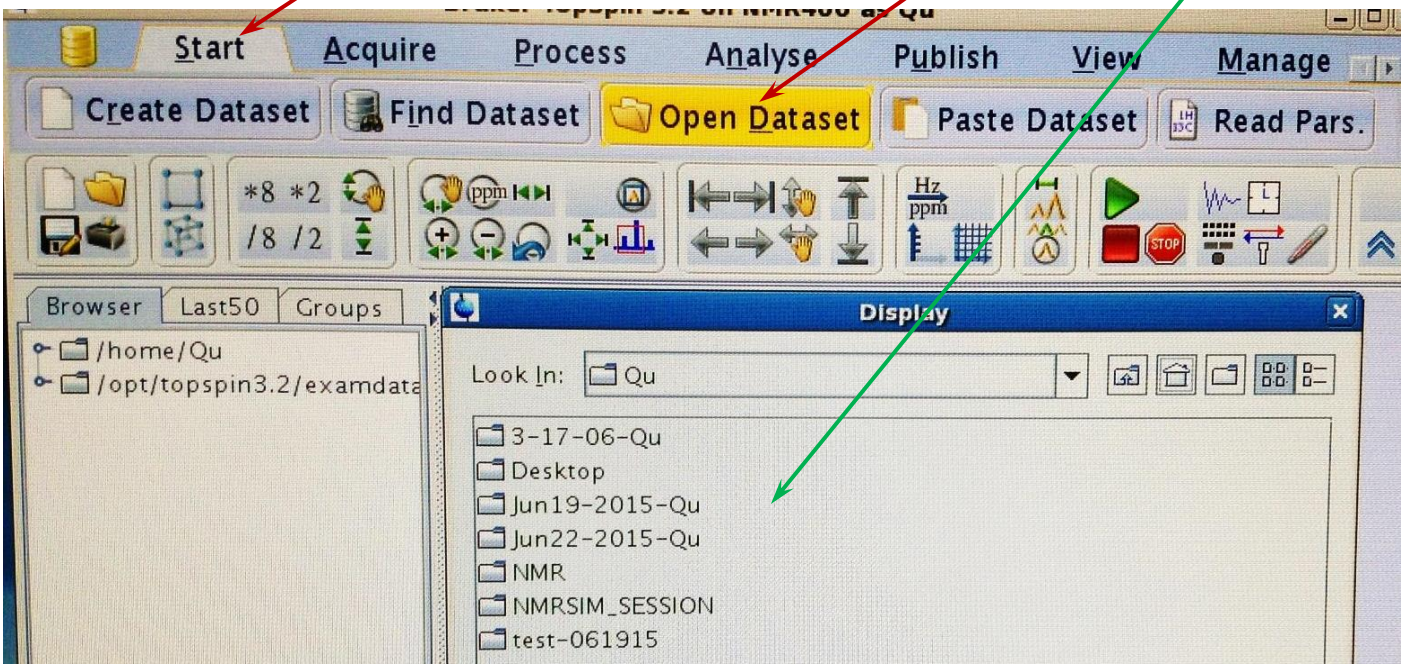
Select the row # containing a previously defined experiment, and then *click* the **Add** button. Go through step 2-7 procedures above, and then *click* **Submit**. (experiments are starting/running by submissions order)



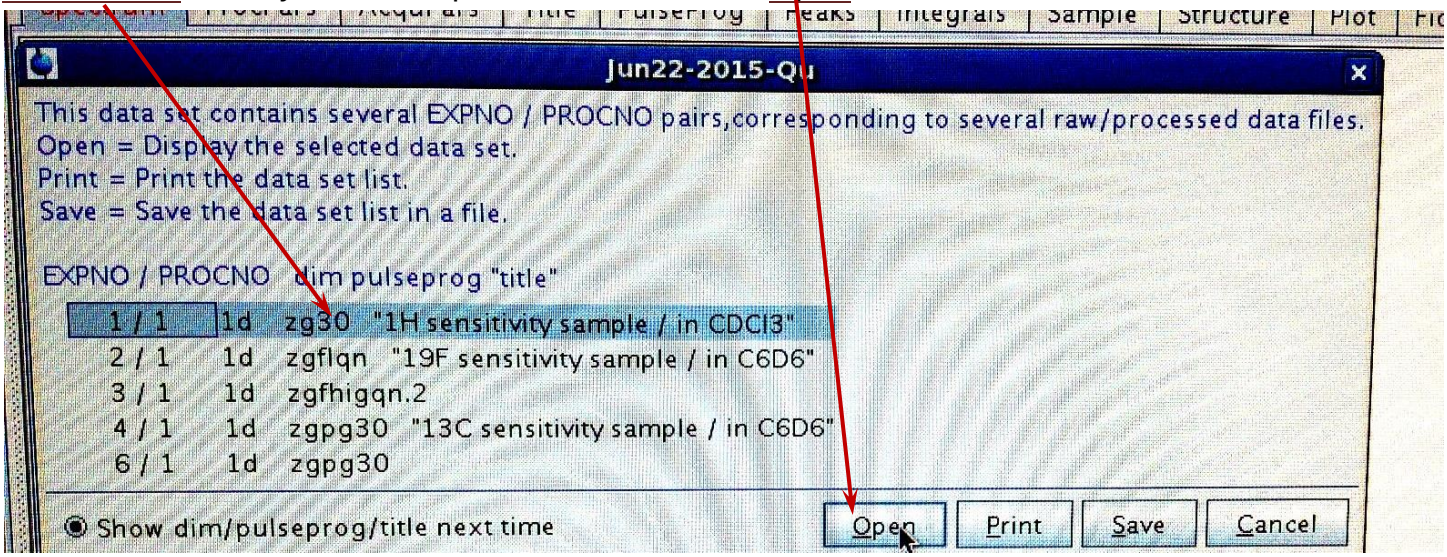
4. Data Processing

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

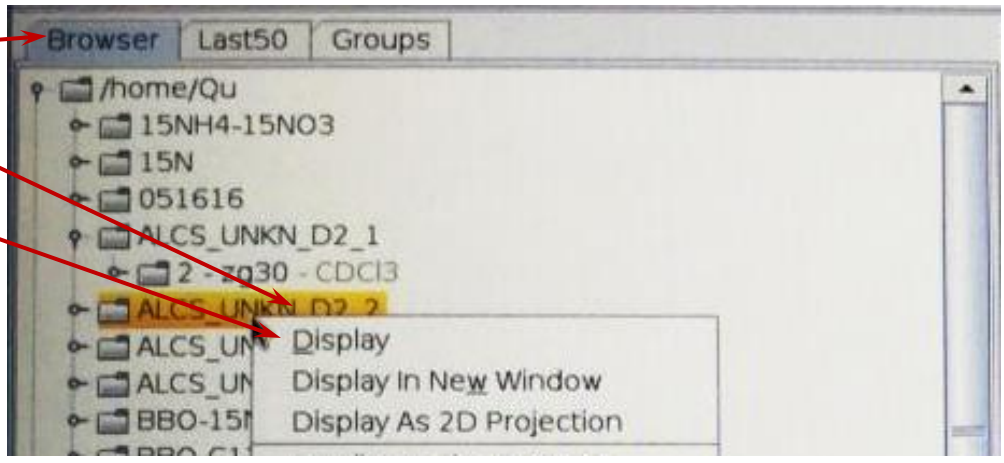
a). Clicking on the **Start** in the TopSpin Menu bar, then click on **Open Dataset**, your dir/files will appear



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



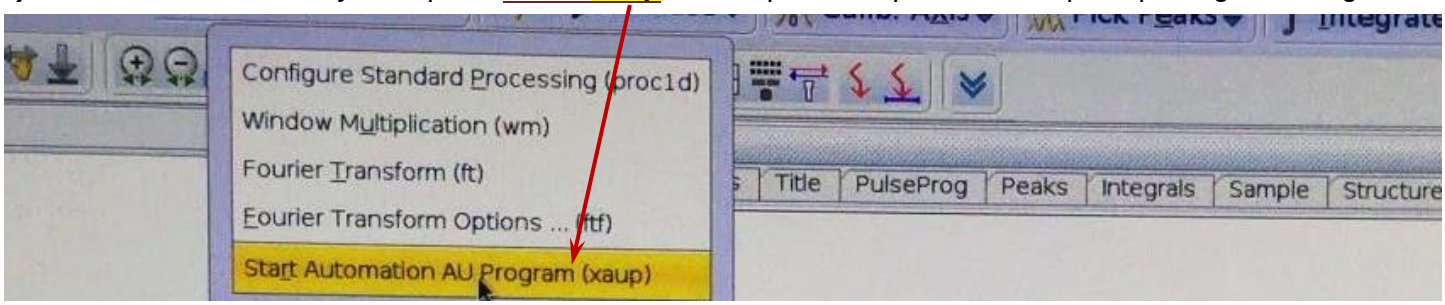
b). click on the **Browser**, locate your data and right-click on a dataset name, and choose **Display** from the drop down menu



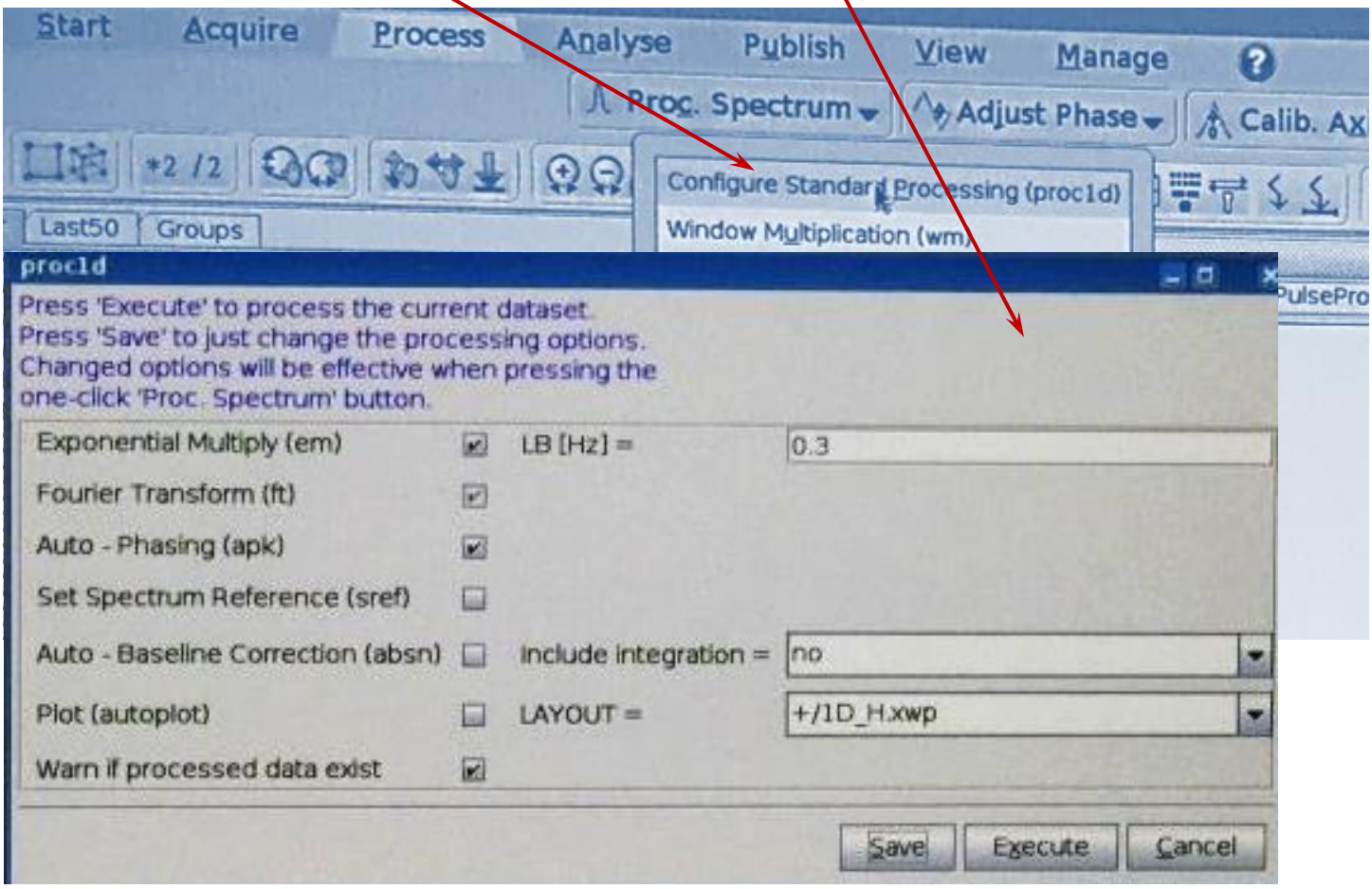
(2). Process Spectrum,

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

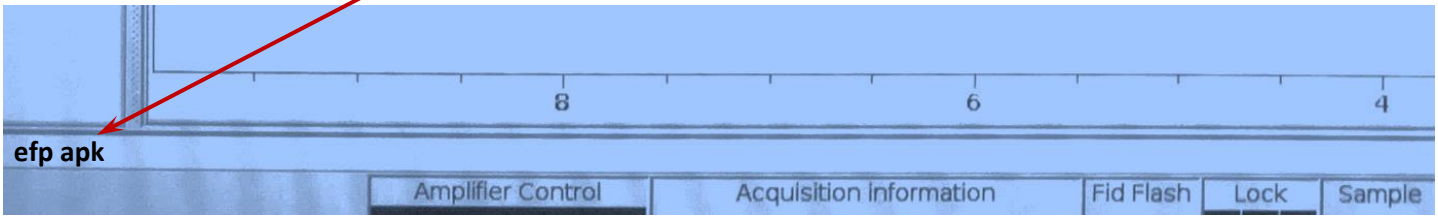
a). Auto Process, for easy and quick, Select **xaup** to auto process spectrum with peak picking and integration.



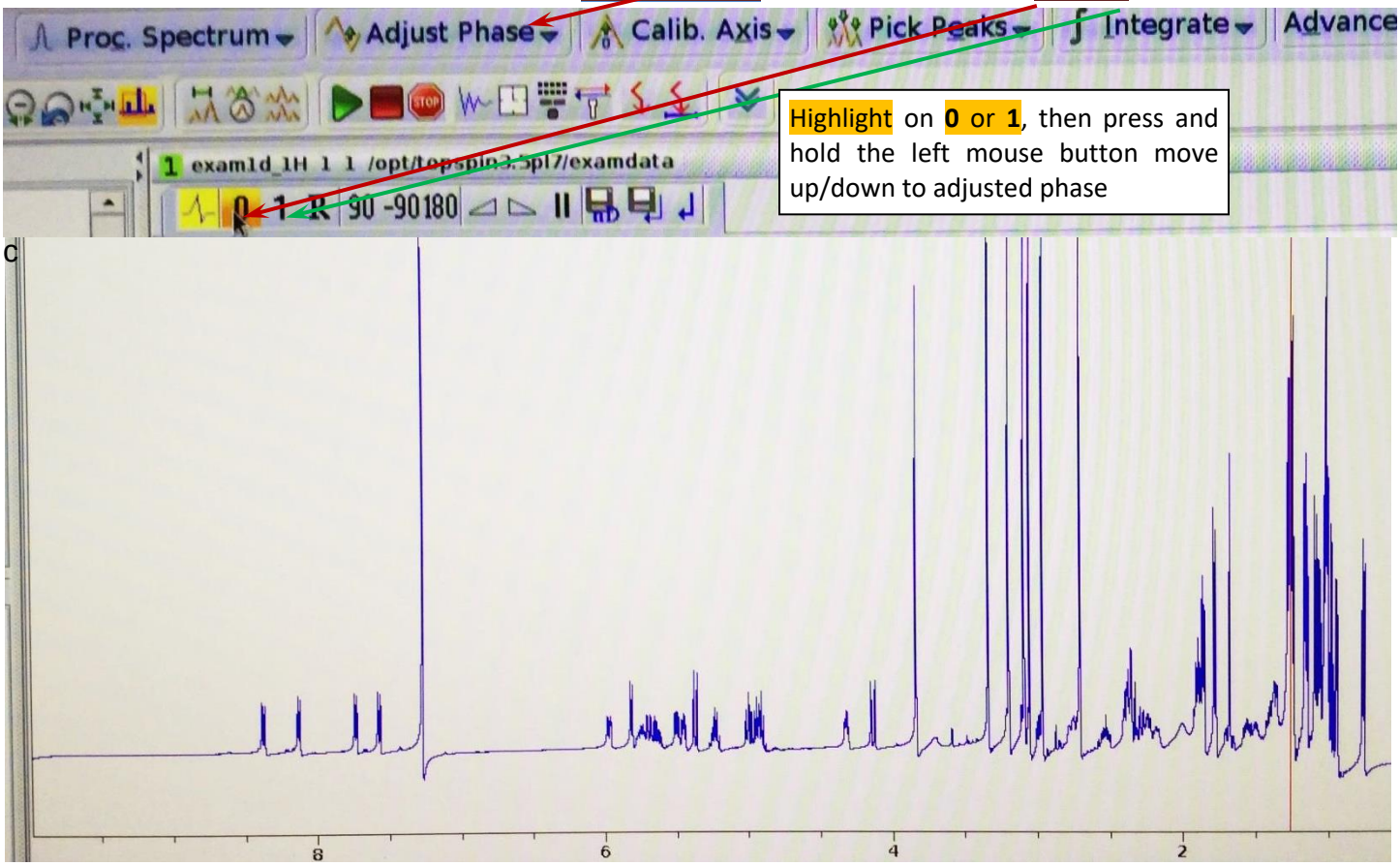
b). Standard Process, Select **proc 1d** (2d, 3d), then fill in drop-down table to process spectrum



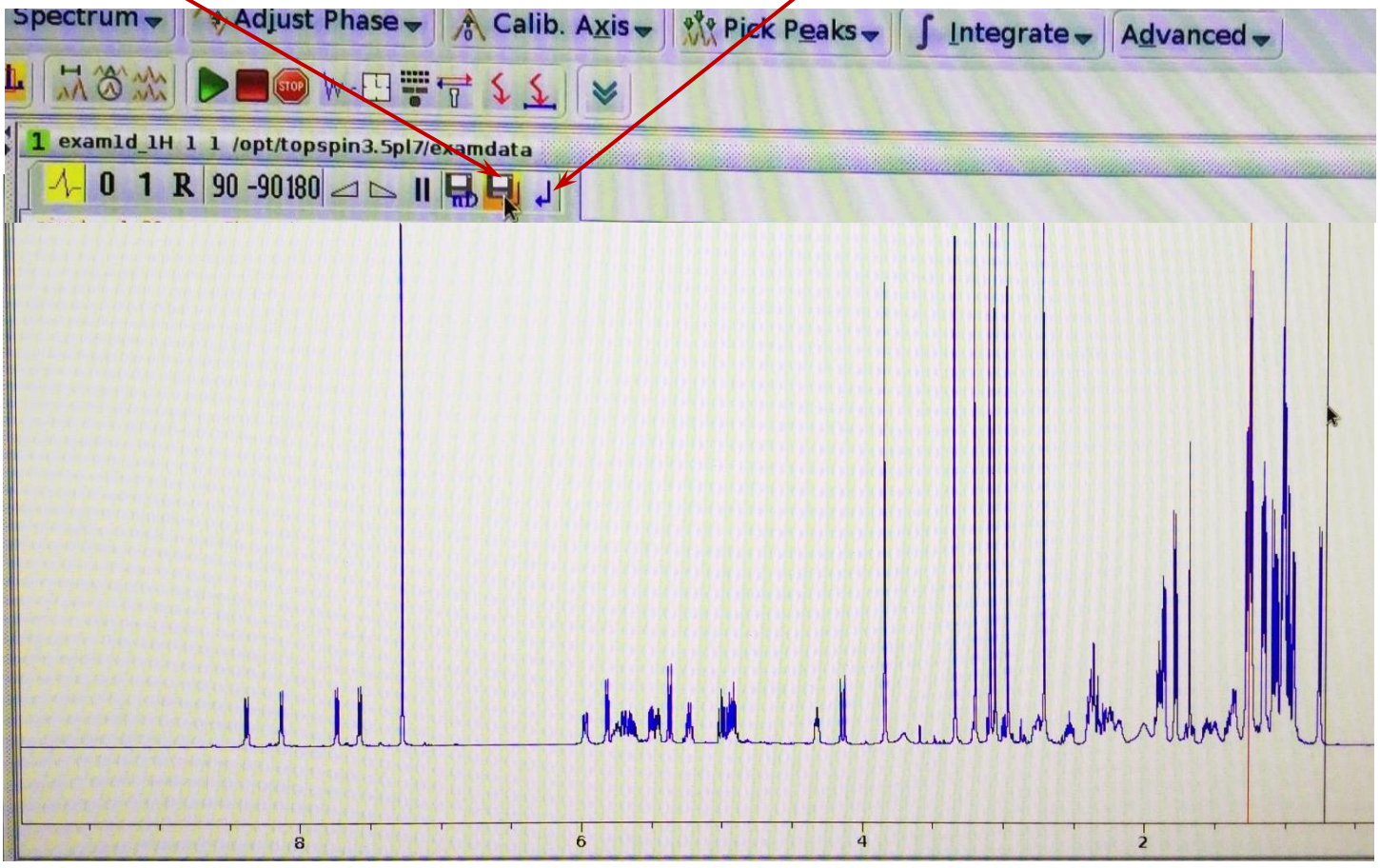
c). Manual Process, type **efp apk** in the command line to process data and execute automatic phase correction



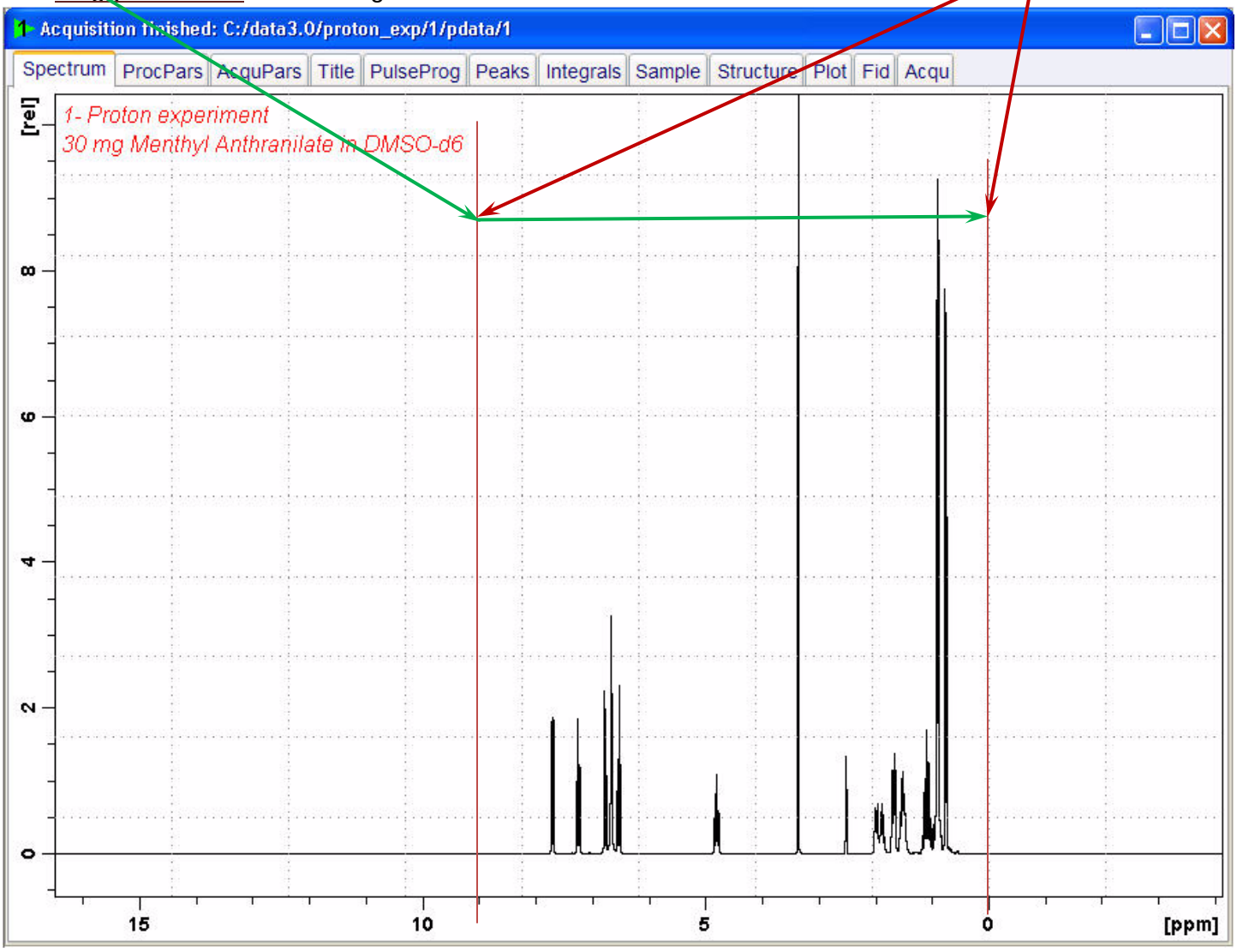
(3). Phase Correction. The simplest procedure is automatic phase correction which is implemented using the command **apk**. For more corrections, click on **Adjust Phase** for manual phase by **0** and **1** (0 and first order)



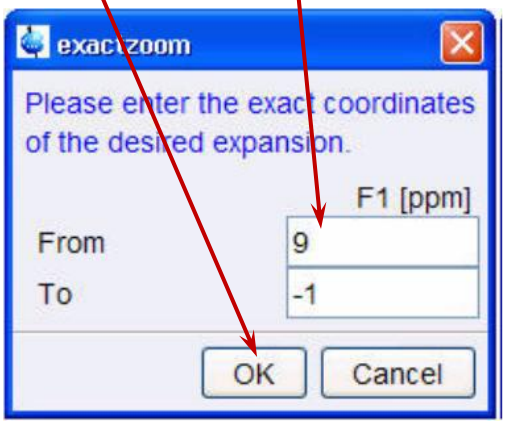
Click on disc icon to save phase correction, then click on return icon to get out the phase section.

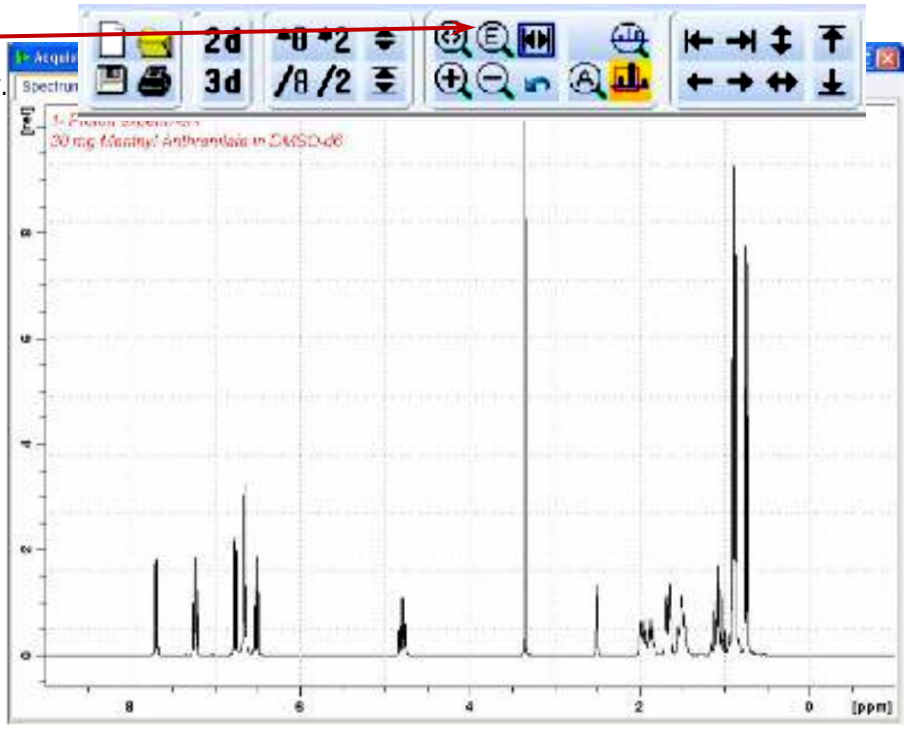


(4). Optimizing the Spectral Width. To display a specific region of your spectrum hold the left mouse button and drag your mouse over the region of interest.

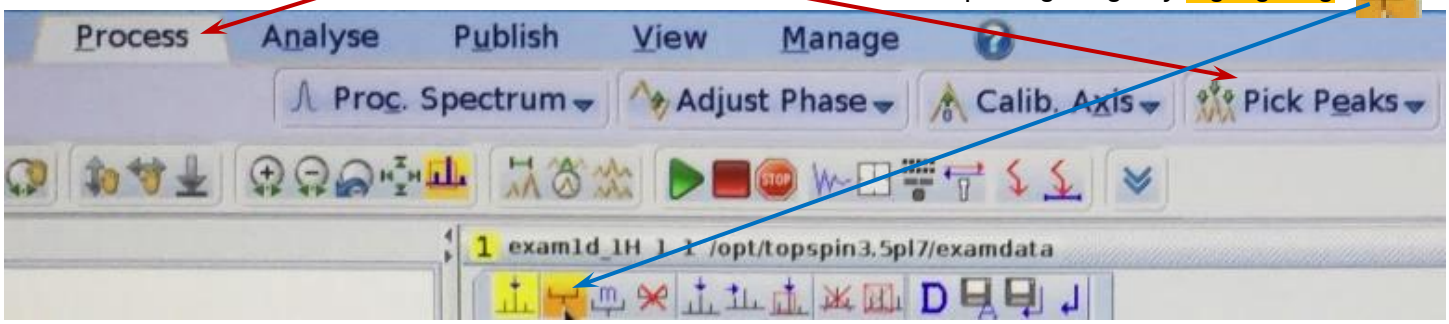


For exact chemical shift range, click  and specify the range you wish to display. For example, to display the region of the Spectral window from 9 to -1 ppm, put 9 and -1 in the **From** and **To** fields, then click OK.

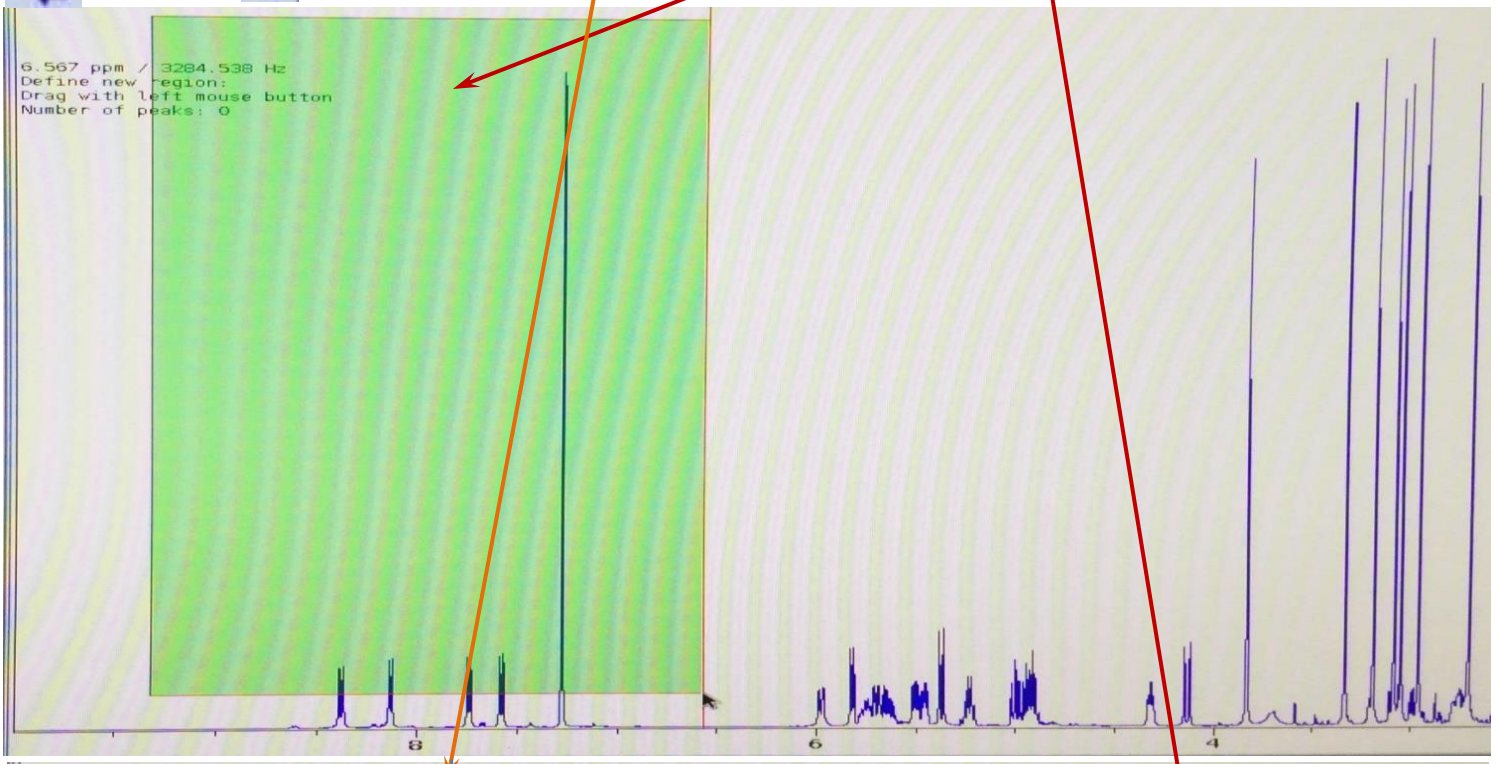


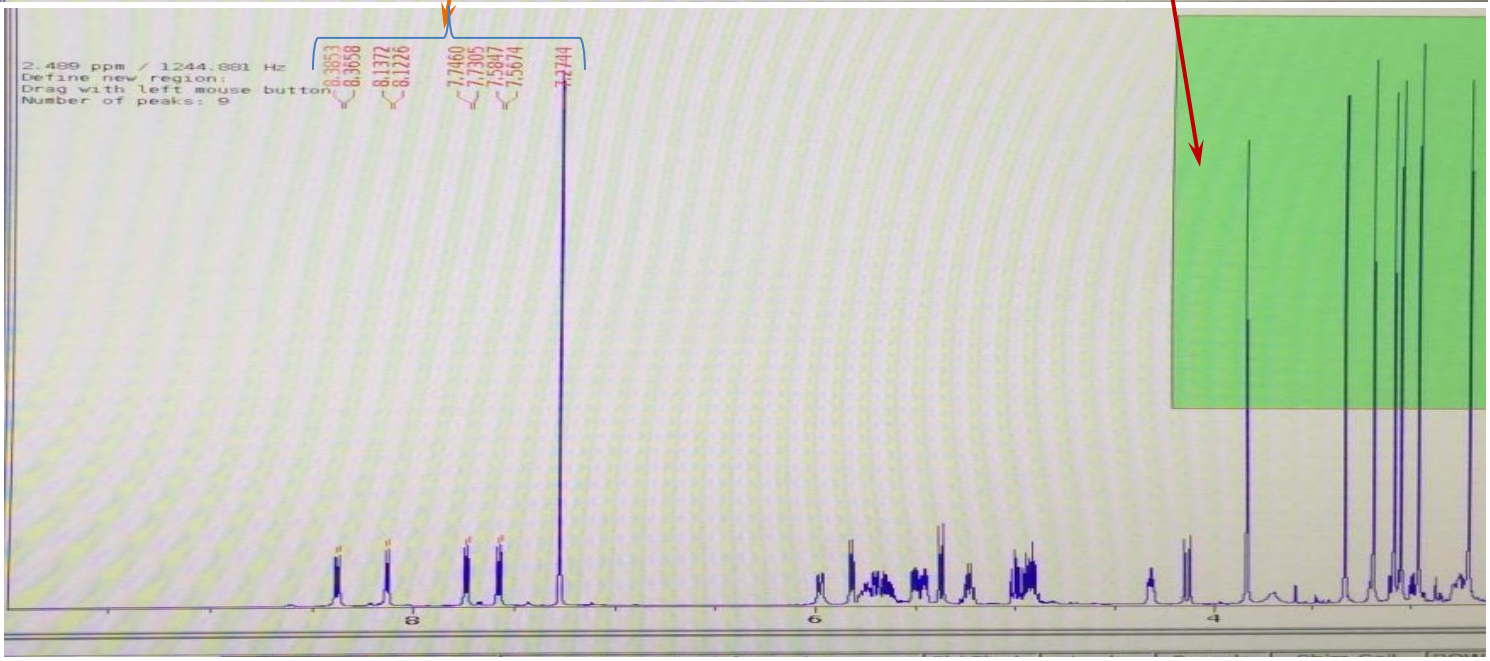


(5). Peak Picking. Click on **Process,** then **Pick Peaks** in the TopSpin Menu bar. Click on to define range
Peak picking range by highlighting



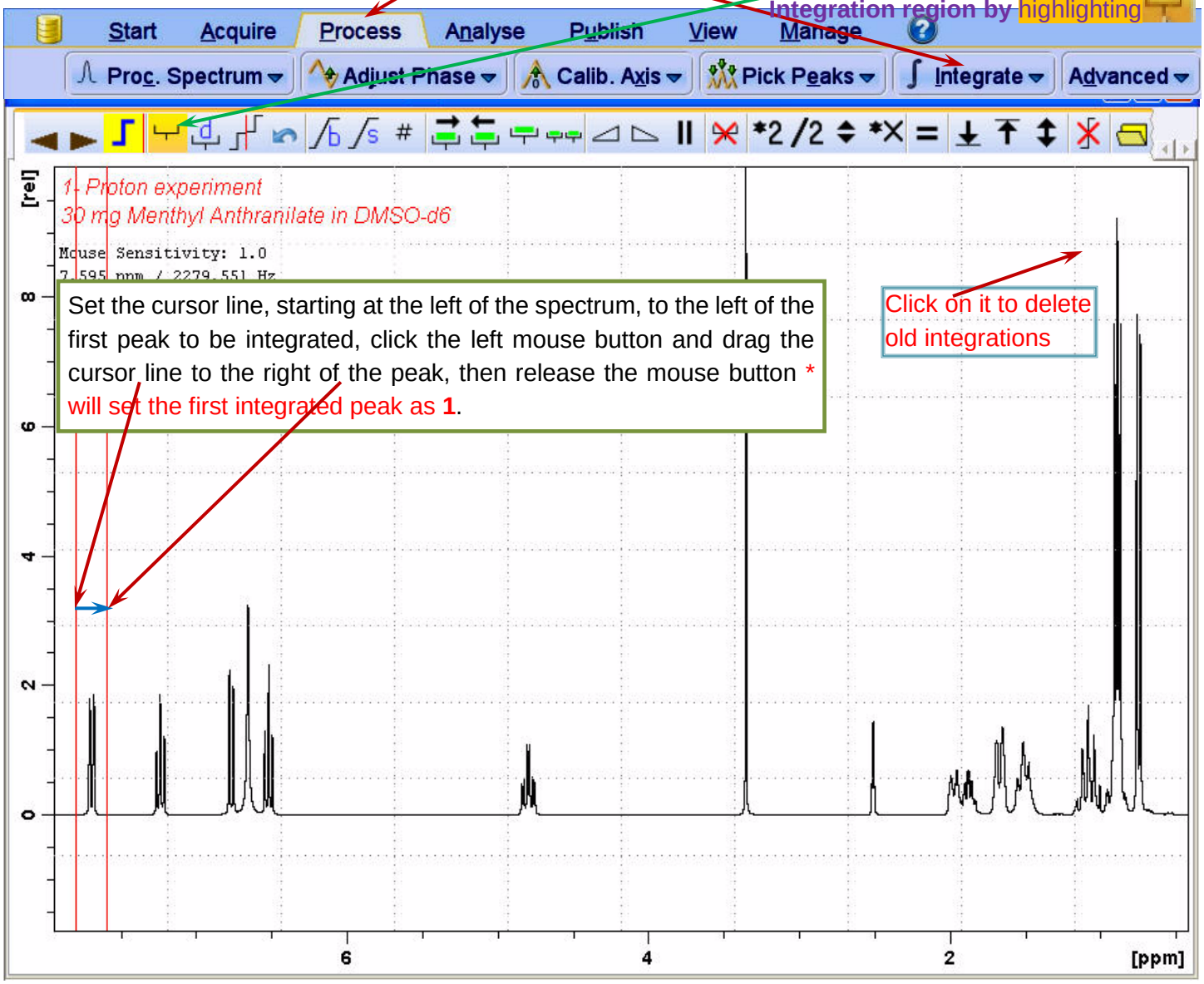
Press and hold the left mouse button over the peaks of interest. A green highlight will form, release the left mouse button, all peaks in green area were picked. Repeat this process to pick all peaks of interest. Then click to save and to get out the mode

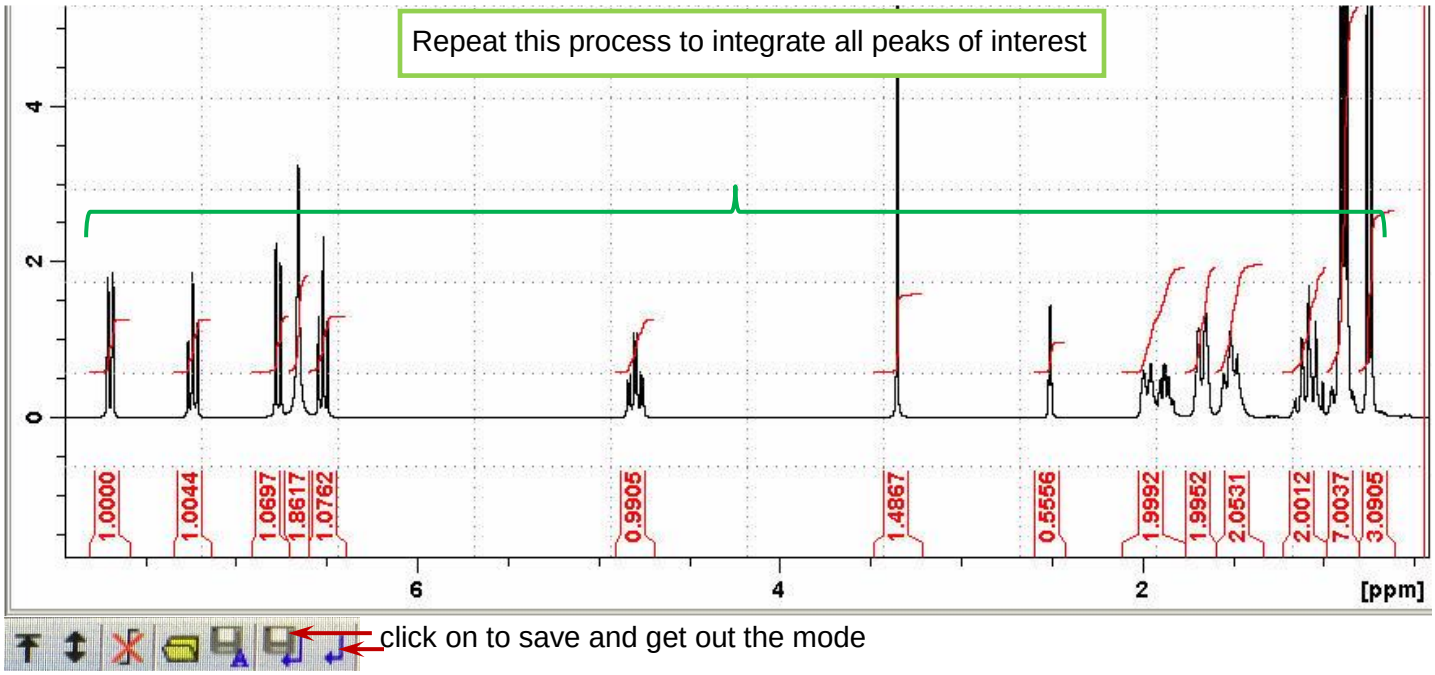




(6). Manual Integration. Click on **Process**, then Click on **Integrate** and to define integration region.

Integration region by highlighting

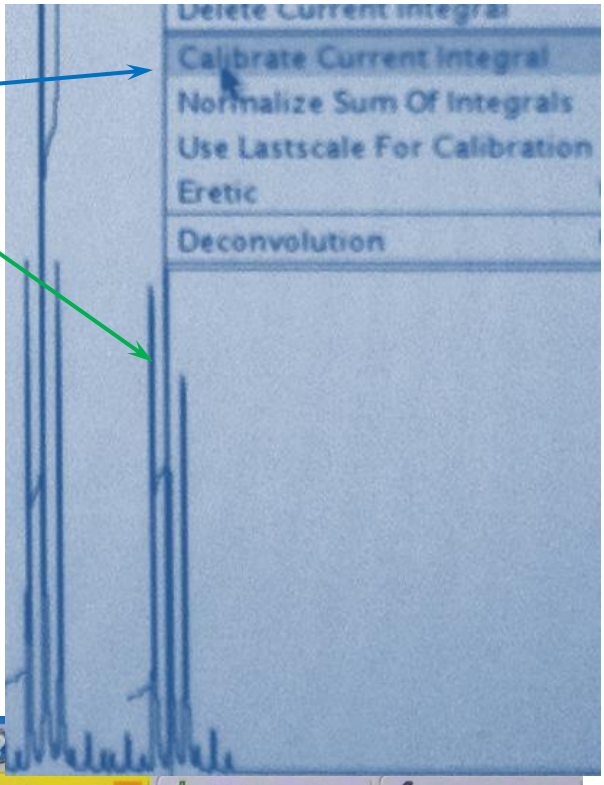


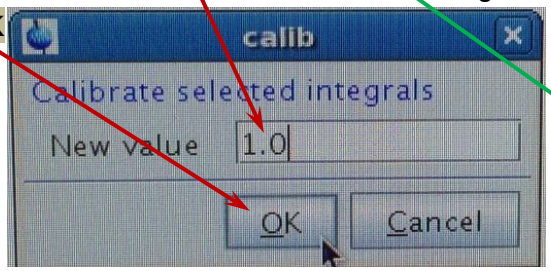


click on to save and get out the mode

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**



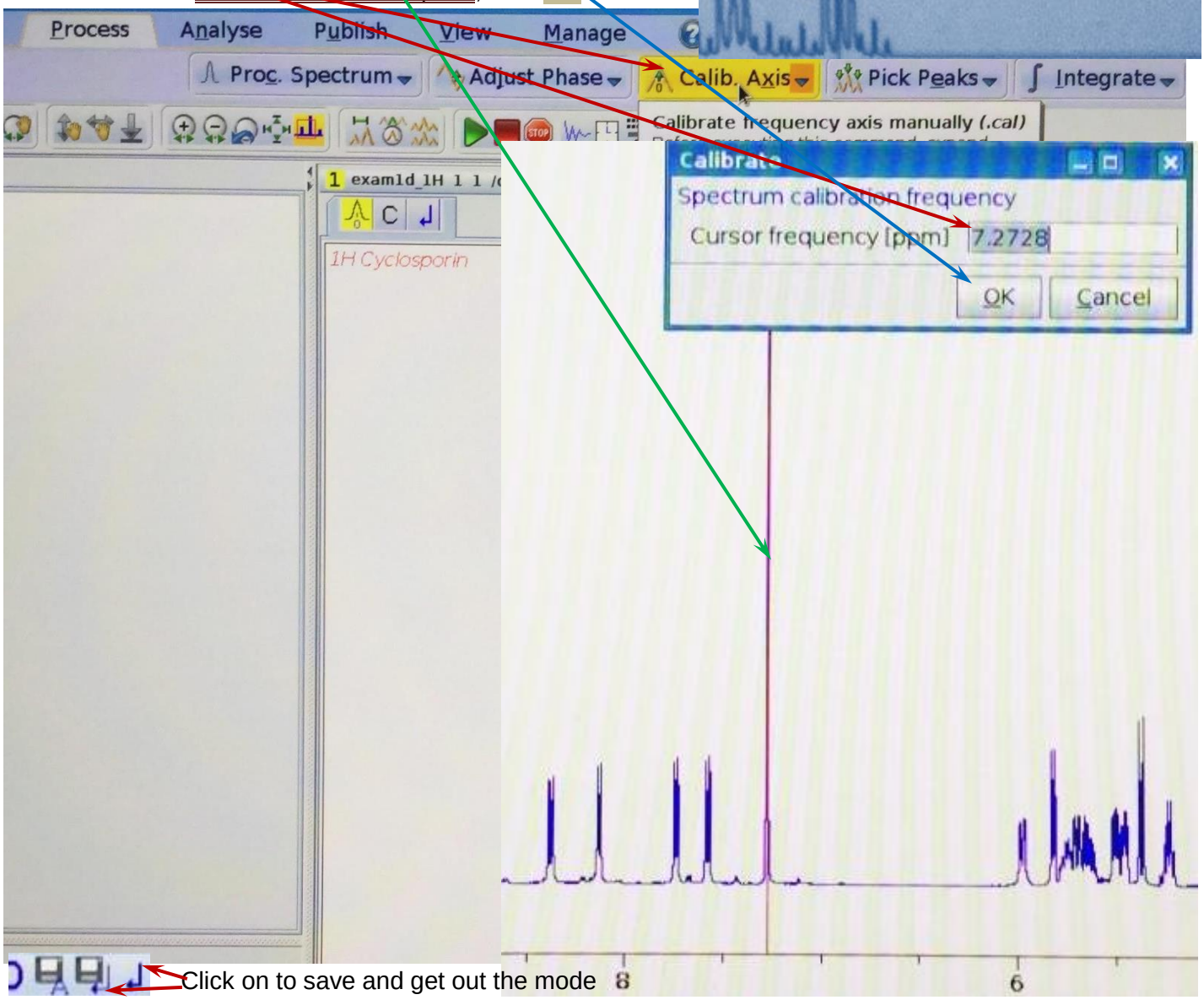


Click on  then click to save and out the mode

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

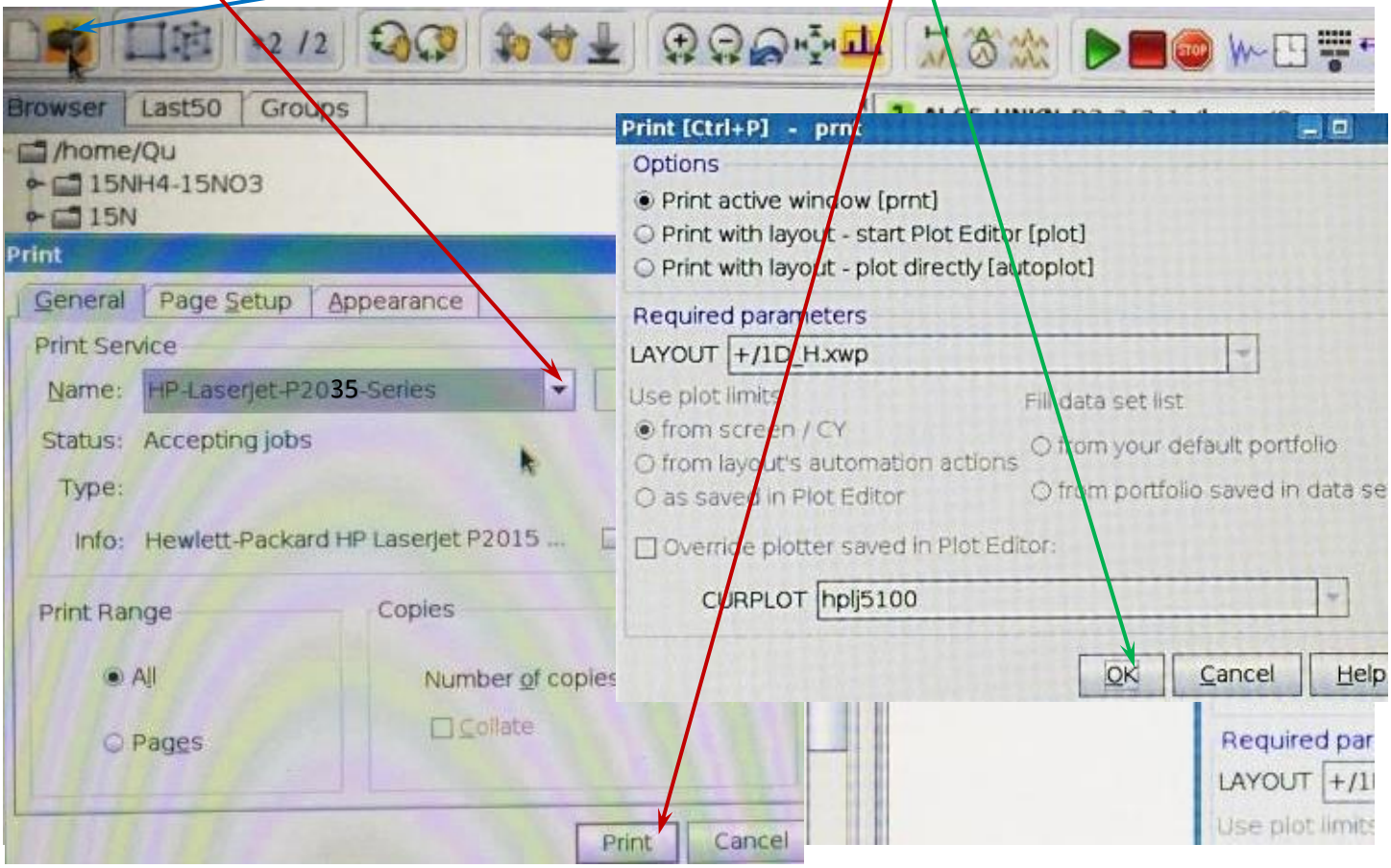
Option for set peak chemical shift reference

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

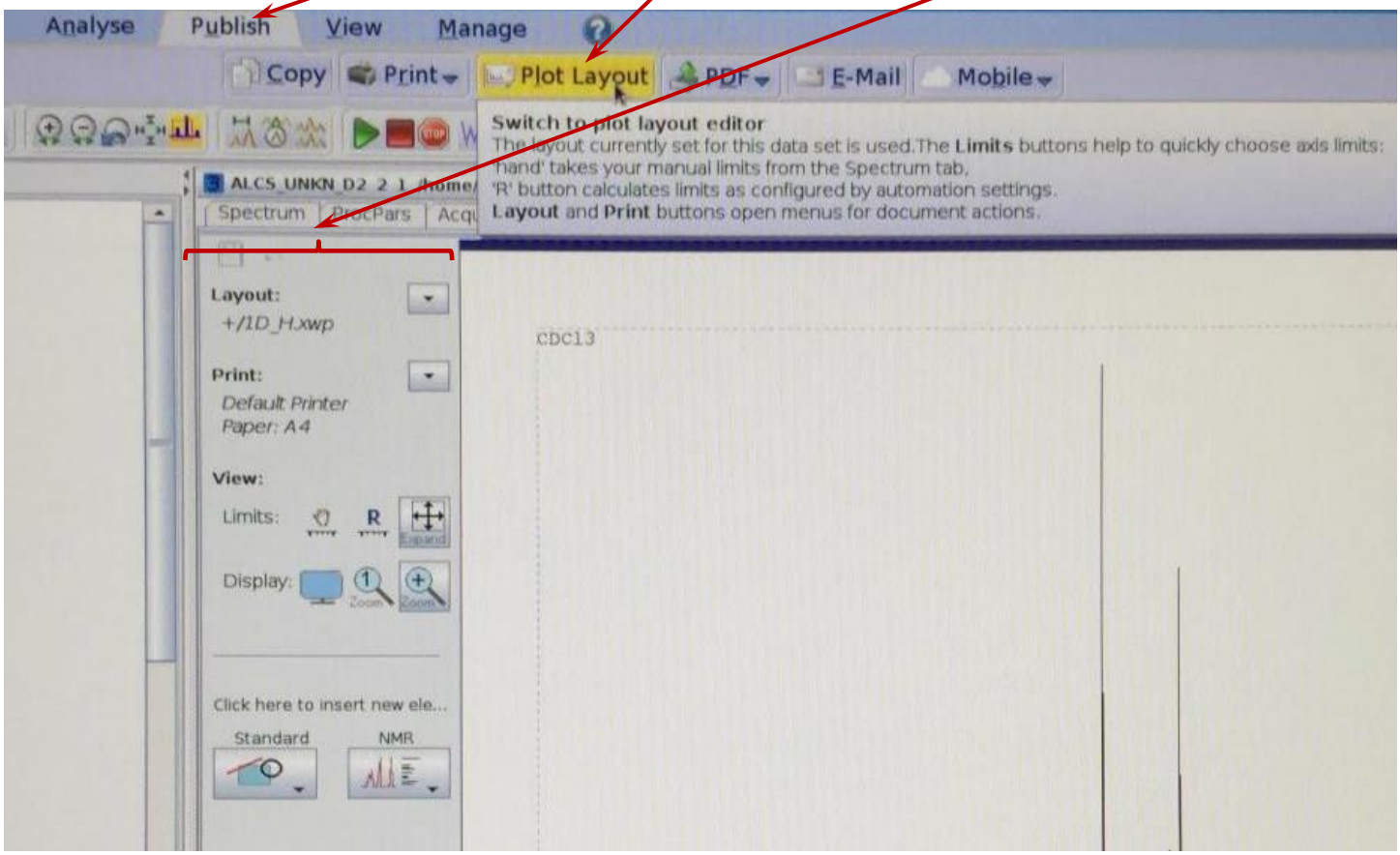


Click on to save and get out the mode

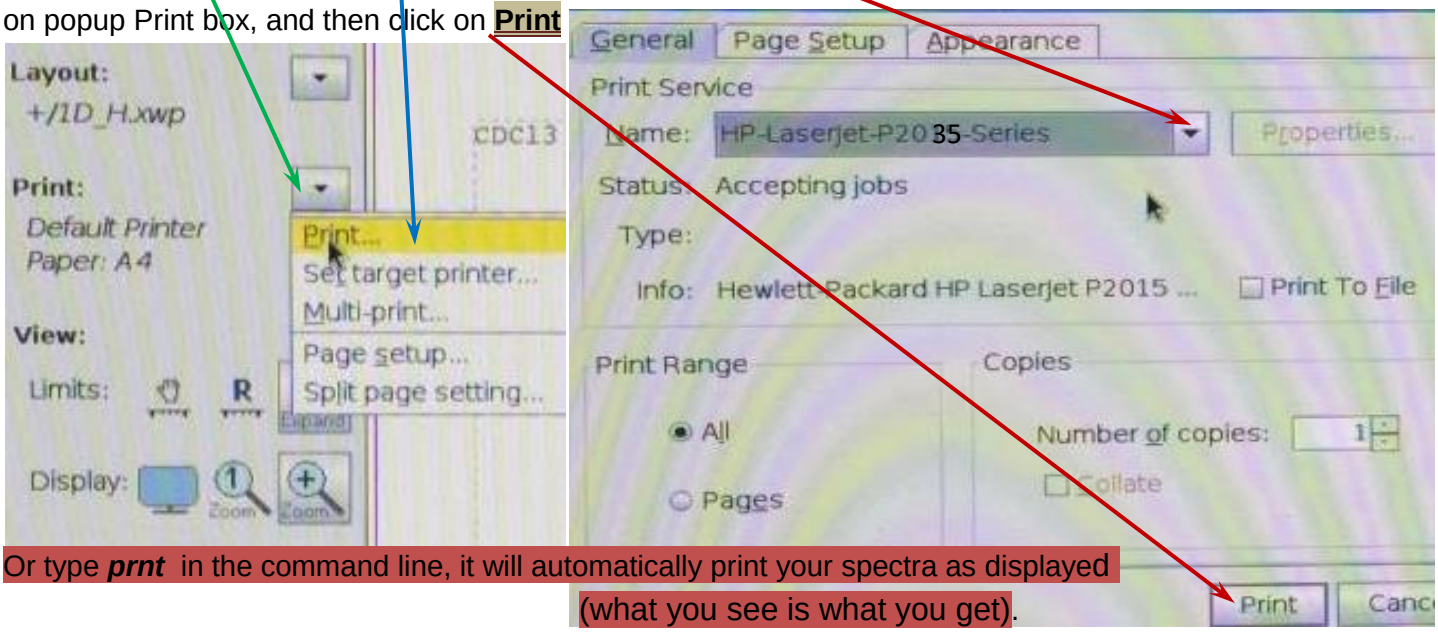
(7). Plotting **a). Quick print**, click on  printer icon, then click **ok** on drop-down box, another Print box popup, click on ▼ to select **HP-Laserjet-P2035** in **Name**, then click on **Print**



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



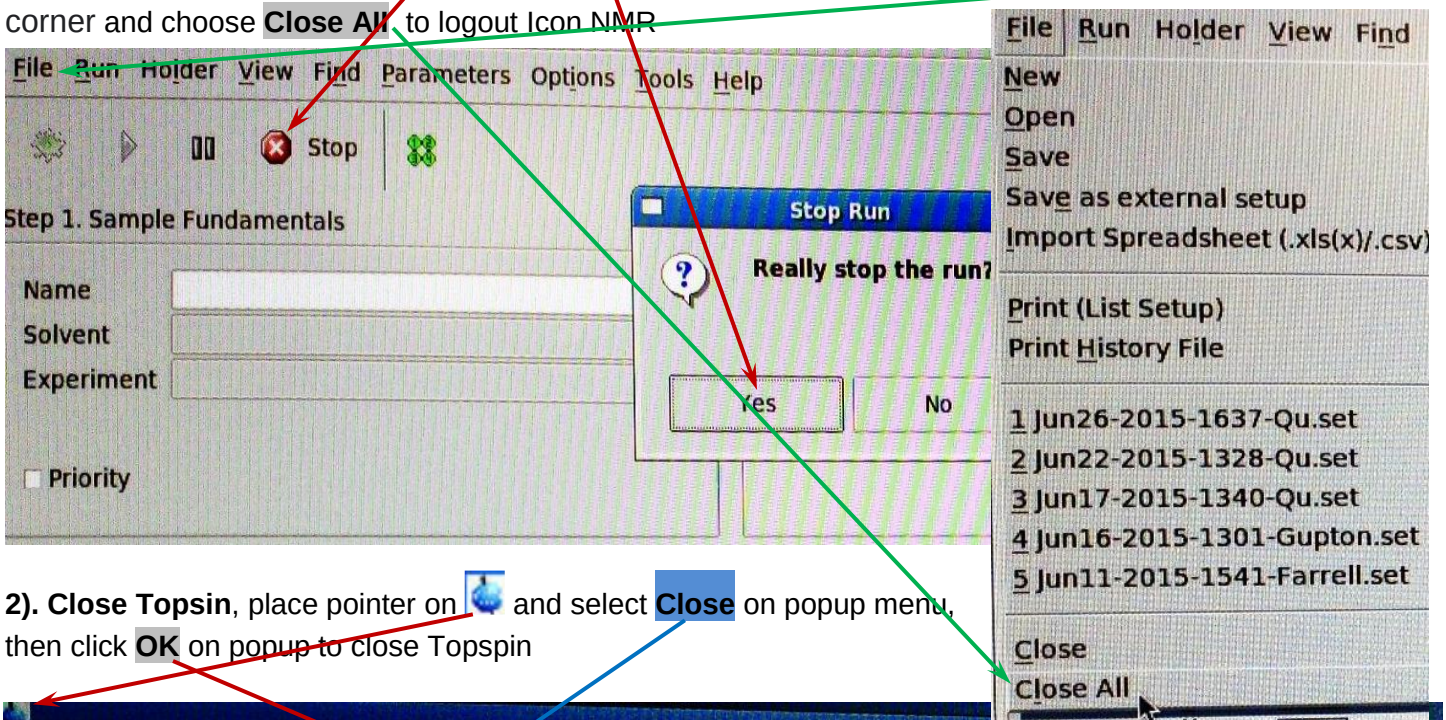
Print, click on ▼ and select **Print** on drop-down list, click on ▼ to select **HP-Laserjet-P2035-Series** in **Name** on popup Print box, and then click on **Print**



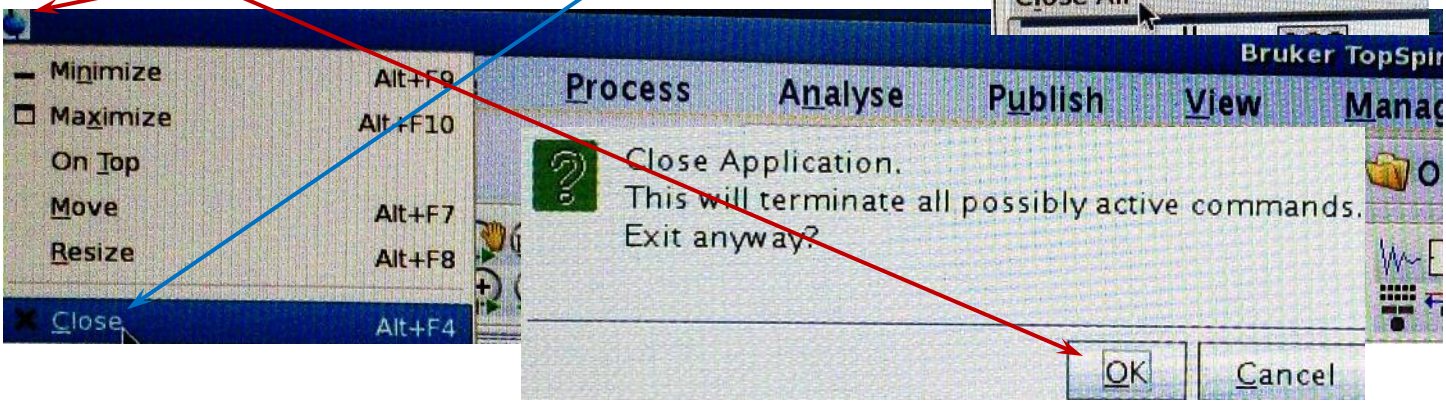
Or type ***prnt*** in the command line, it will automatically print your spectra as displayed (what you see is what you get).

5. Close Icon NMR &TopSpin (must close Icon NMR first)

1). Close Icon NMR, Click Stop, select **yes** on popup, then place pointer on **File** the top/upper-left corner and choose **Close All** to logout Icon NMR



2). Close Topsin, place pointer on and select **Close** on popup menu, then click **OK** on popup to close Topspin



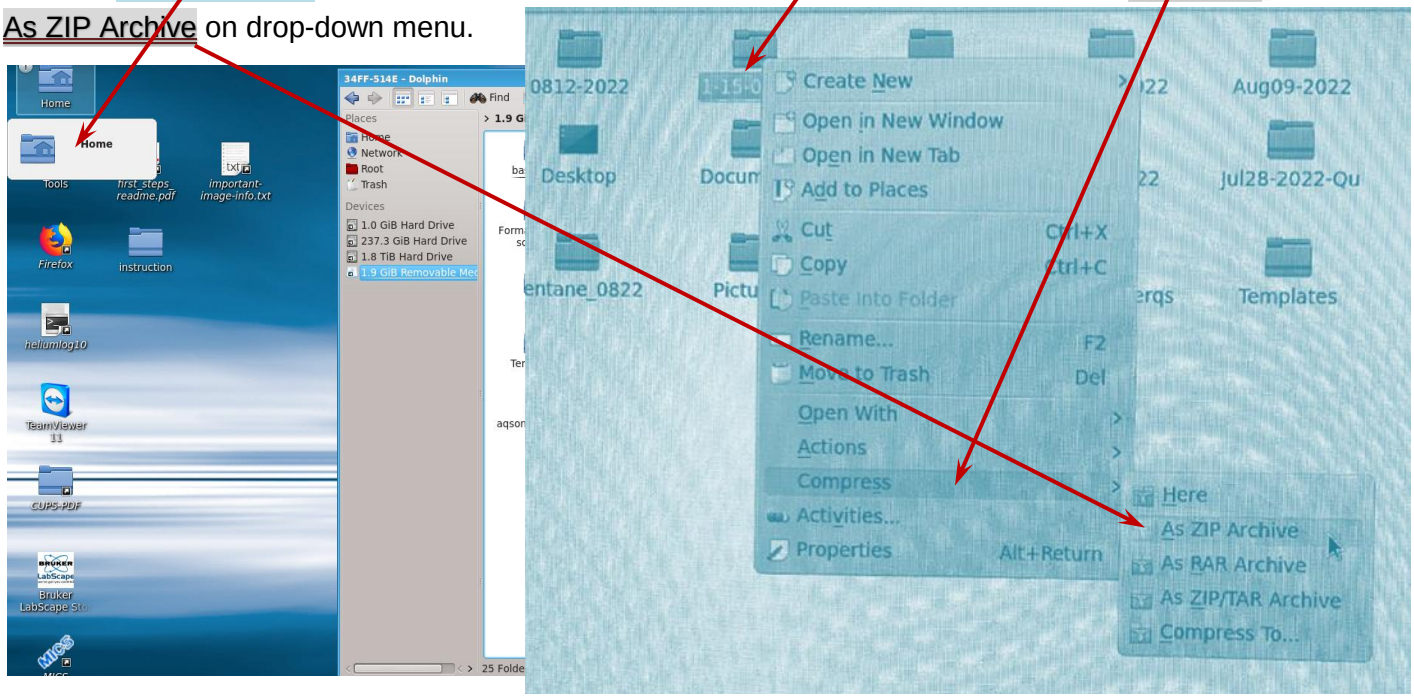
6. Finish.

After experiments are done, **there are only red and Dim lights, no green and yellow lights showing on ICON NMR,** then remove all sample tubes from the SampleXpress Sample Changer, take sample tubes out from blue spinner, and place blue spinner in the box.

7. Copy/Transfer Files

(1) Email Send/Transfer Files by Email Attachment

1). Zip Files There are a few files in one spectrum. It is easier to make a Zip file, send it by email attachment. Click on Home directory on desktop, then select and right click on the files, then select Compress → As ZIP Archive on drop-down menu.

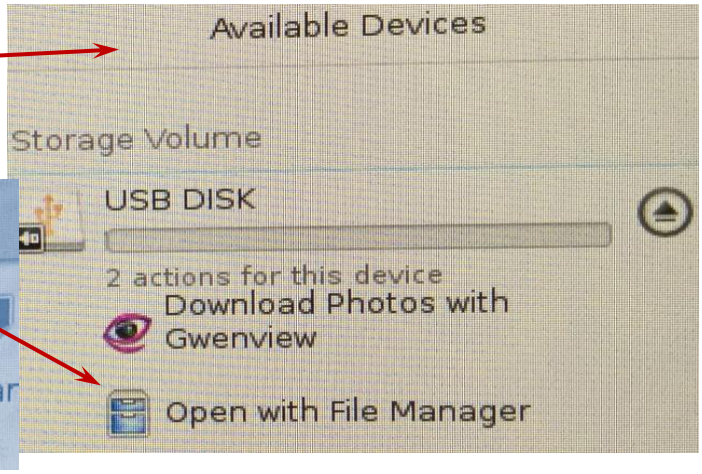


2). Email File Click on 

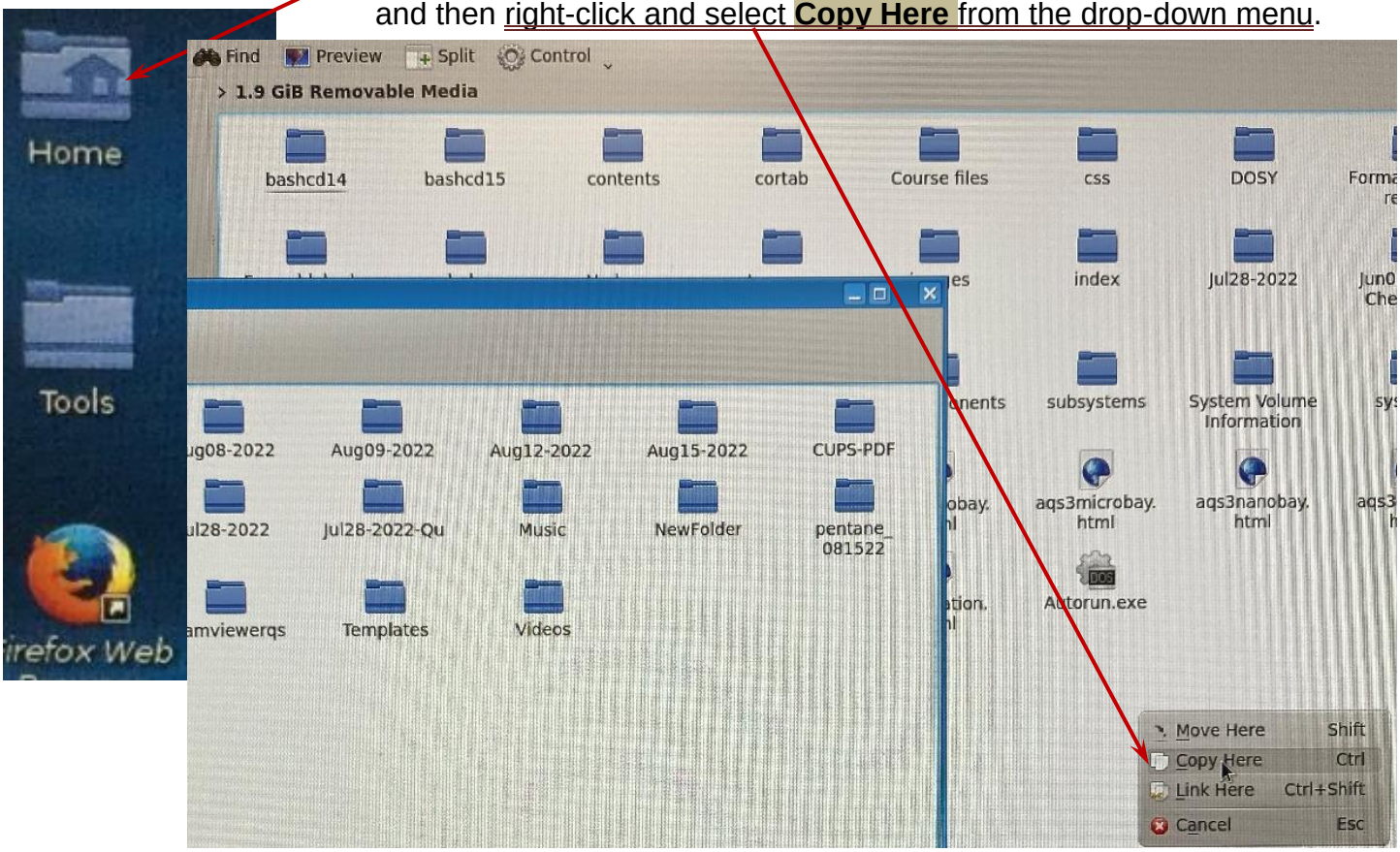
to open internet browser, then get and login your email account, send the zip as attachment.

(2) USB Flash Drive Copy/Transfer Files to USB Flash Drive

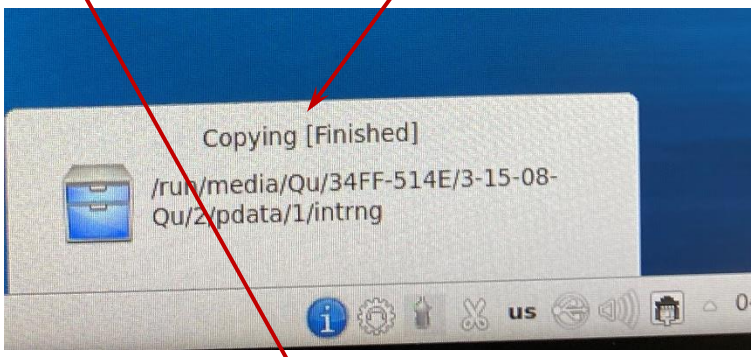
1). Open USB Insert your USB flash drive to PC, Available Devices window pop up, click on Open with File Manager on drop-down menu to open the USB, the USB drive open

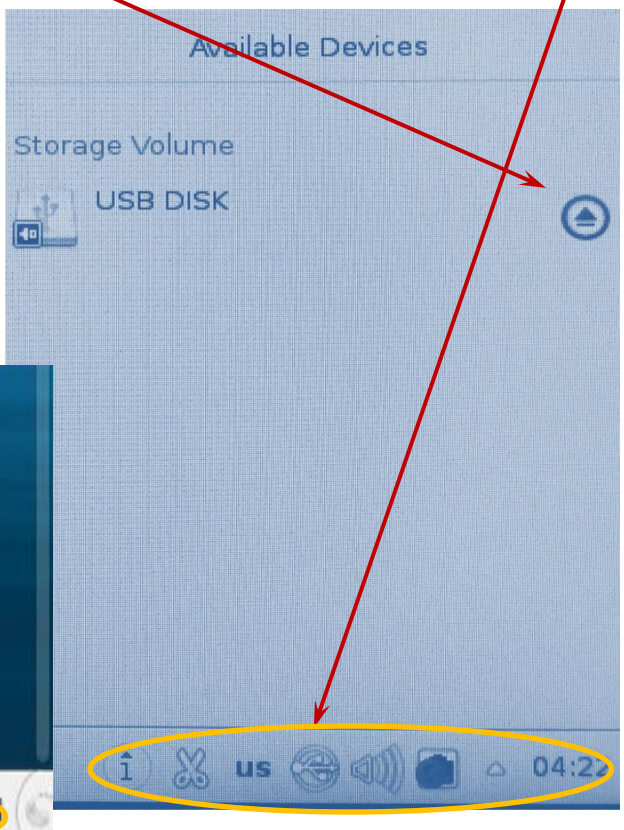


2). Copy Files Click **Home folder** on desktop, then select the file/files and drag to USB DISK window, and then right-click and select **Copy Here** from the drop-down menu.



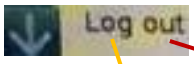
(3). Close USB when Copying [Finished], click on the arrow on the Available Devices window, the **disappear**, the USB drive can be removed/unplug.



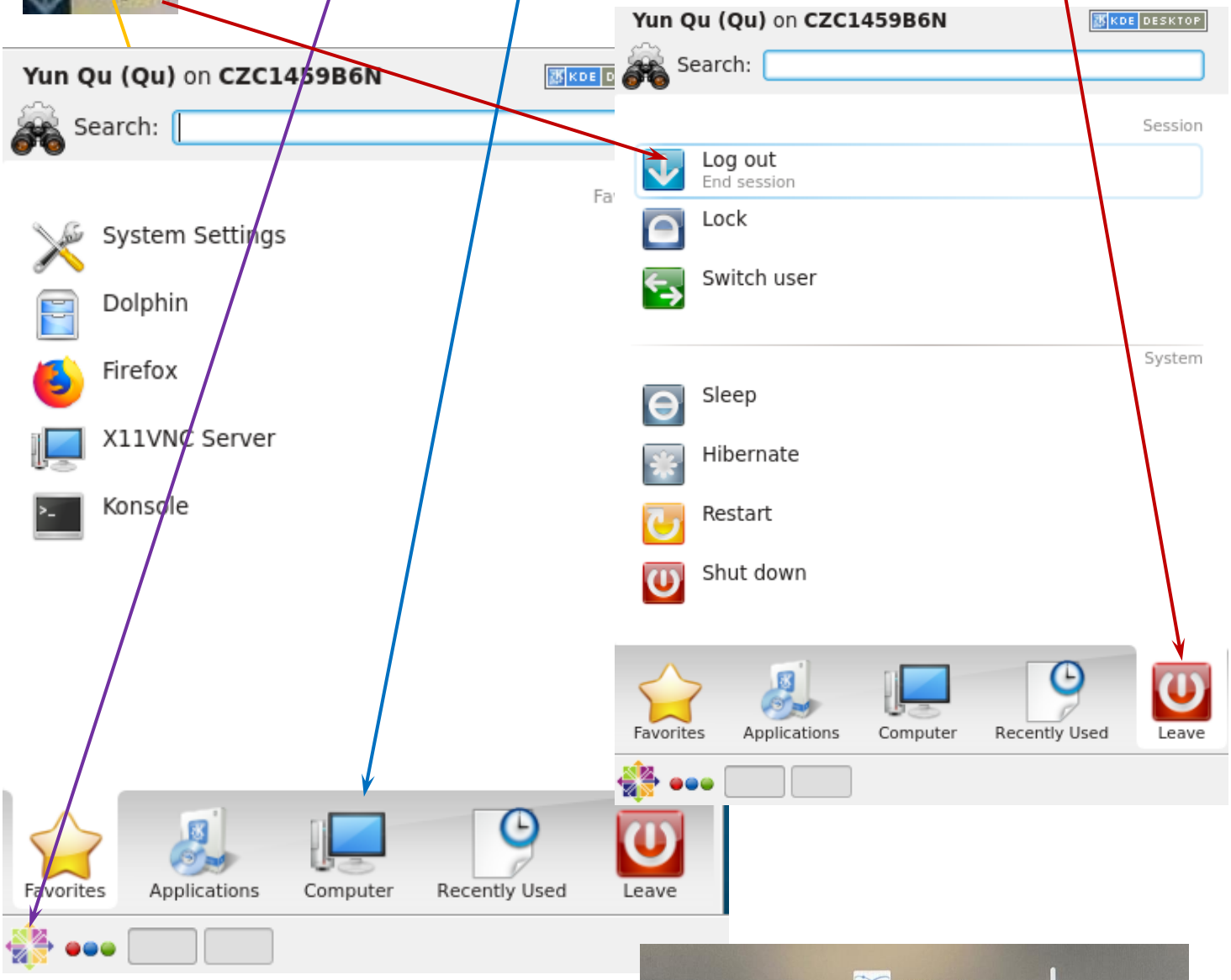


8. Logout **logout your account** when you finish all!

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 (logout).