JEOL RESONANCE

400 / 500 MHz ECZL NMR spectrometer

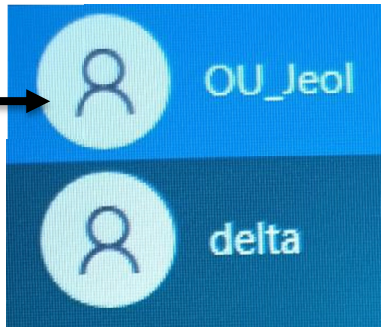
Please be sure to read this instruction manual carefully and fully understand its contents prior to the operation for the proper use of the 400 / 500 MHz JEOL instruments.

Log in your Delta user account:

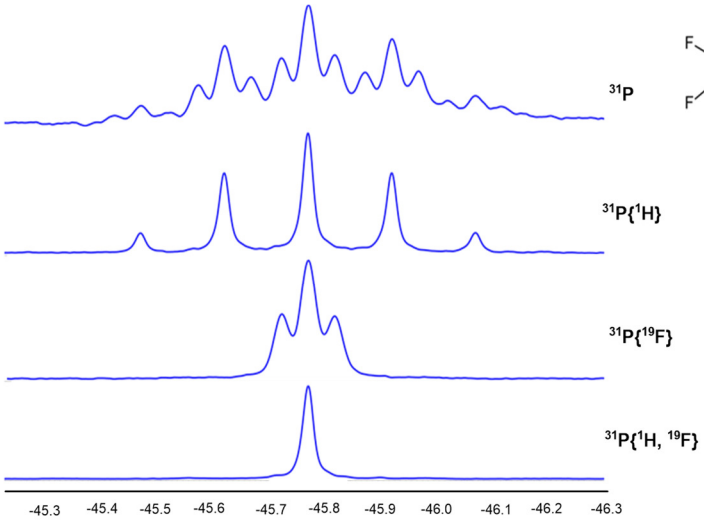
Double click the Delta icon on the desktop screen

NMR workstation computer screen

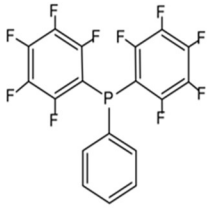
Click here



Enter the password **jeol_23** to log in to your OU_ Jeol NMR account.



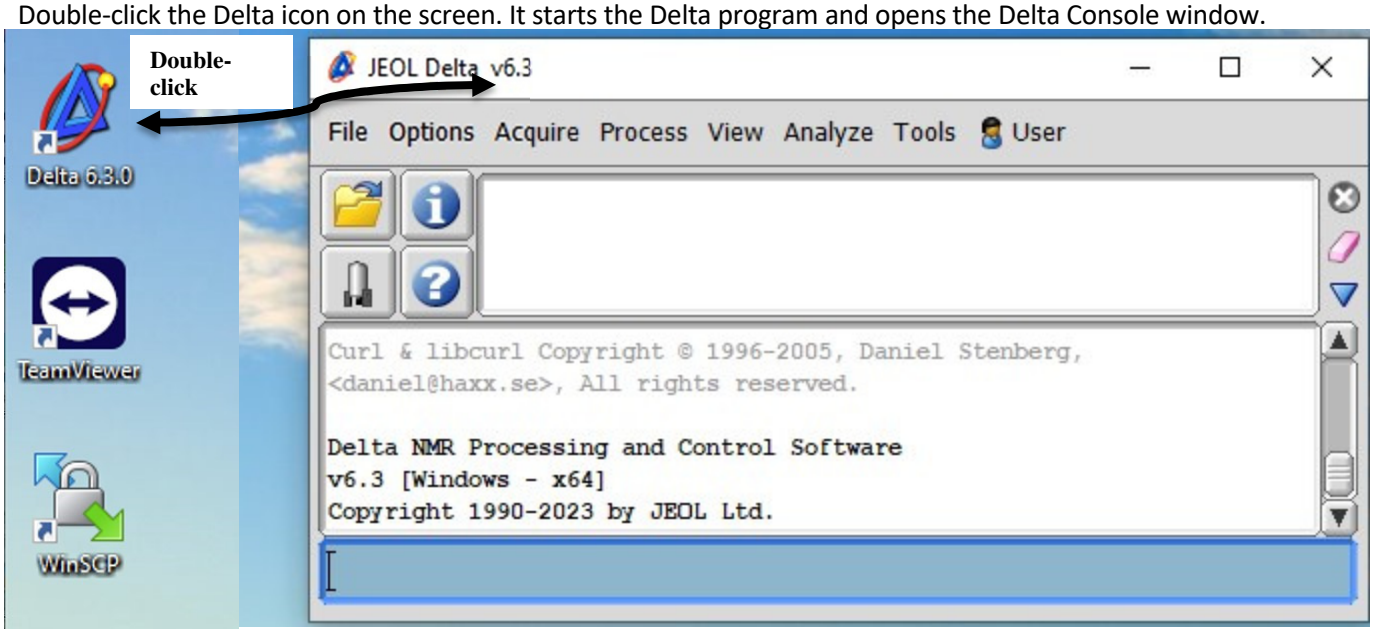
^{31}P NMR spectrum (top)



Decoupling from ^{1}H, ^{19}F and simultaneous decoupling from ^{1}H and ^{19}F

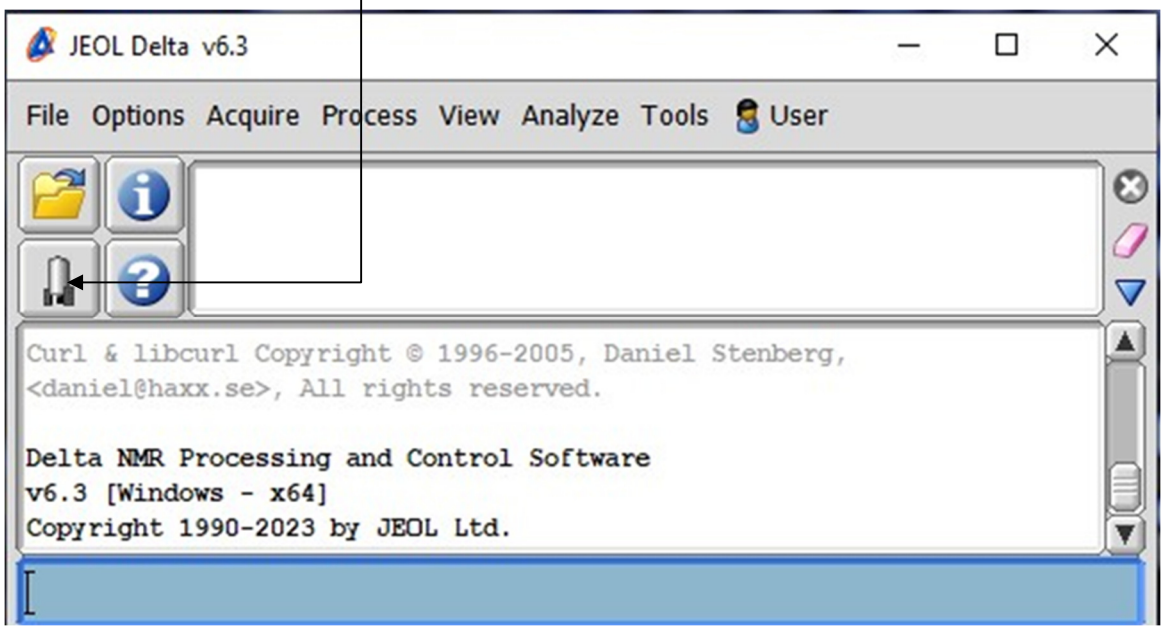
Starting the Delta Program

Double-click the Delta icon on the screen. It starts the Delta program and opens the Delta Console window.



Connecting to the Spectrometer

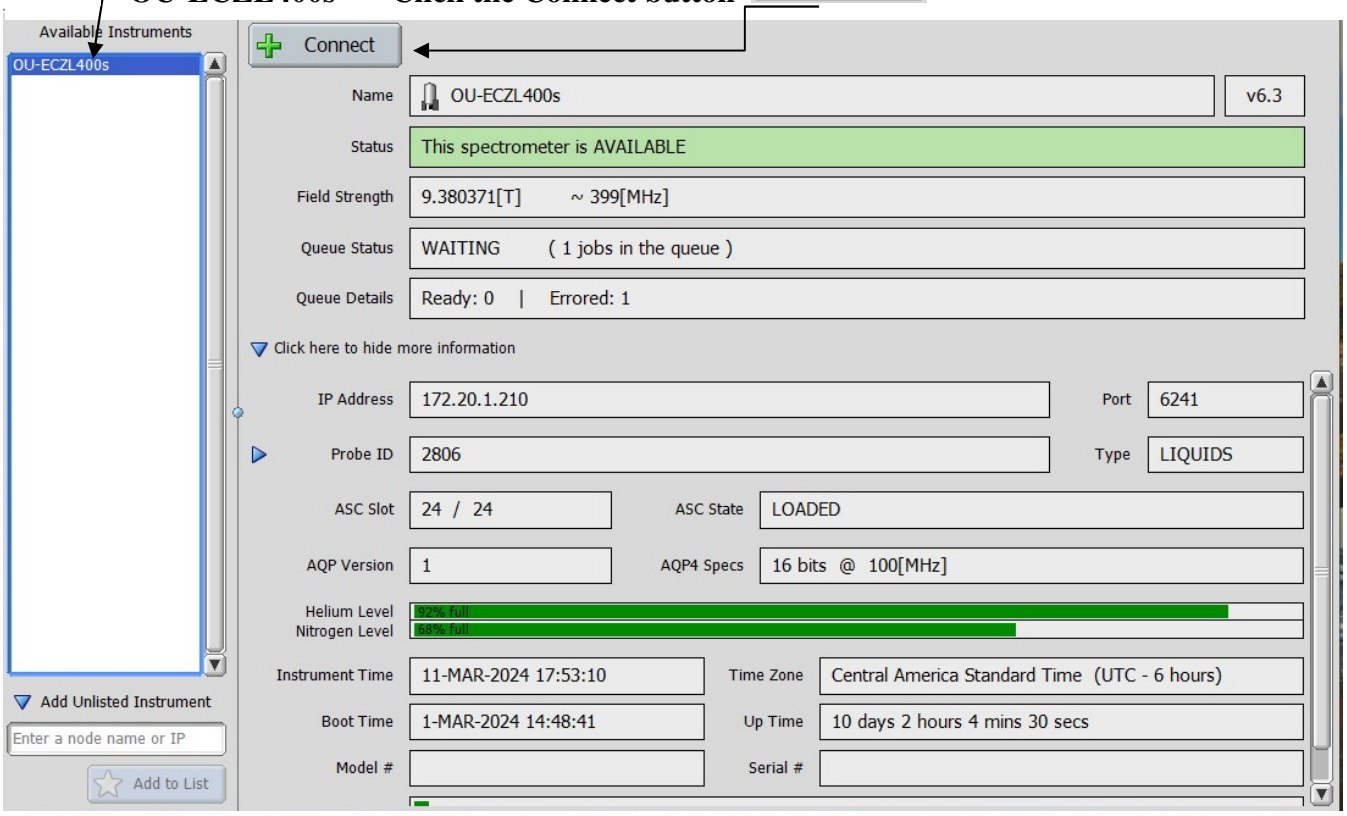
Click the magnet icon in the Delta Console window.



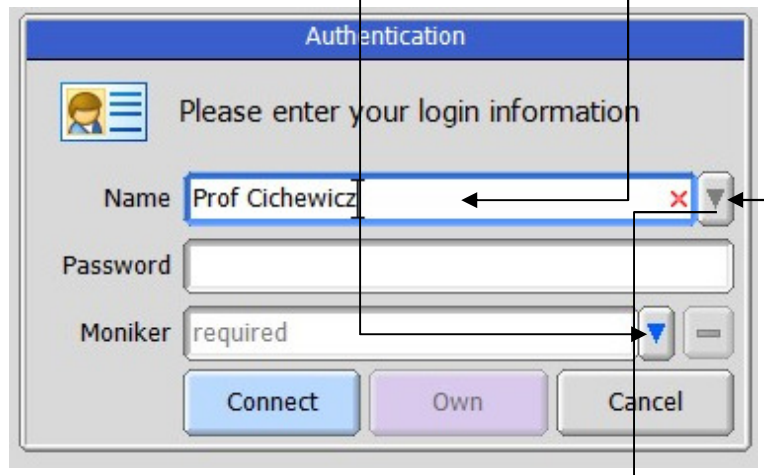
Click here

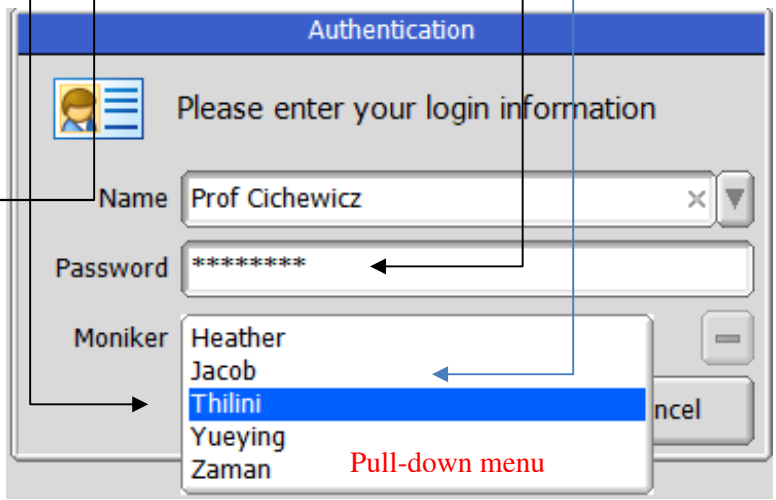
The Spectrometer Control window appears and shows a name of available instrument.

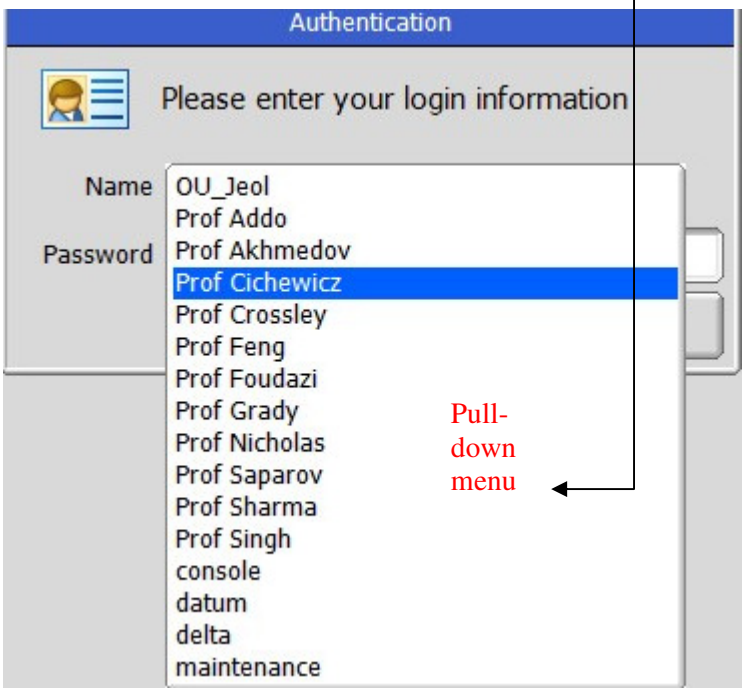
OU-ECZL400s **Click the Connect button**

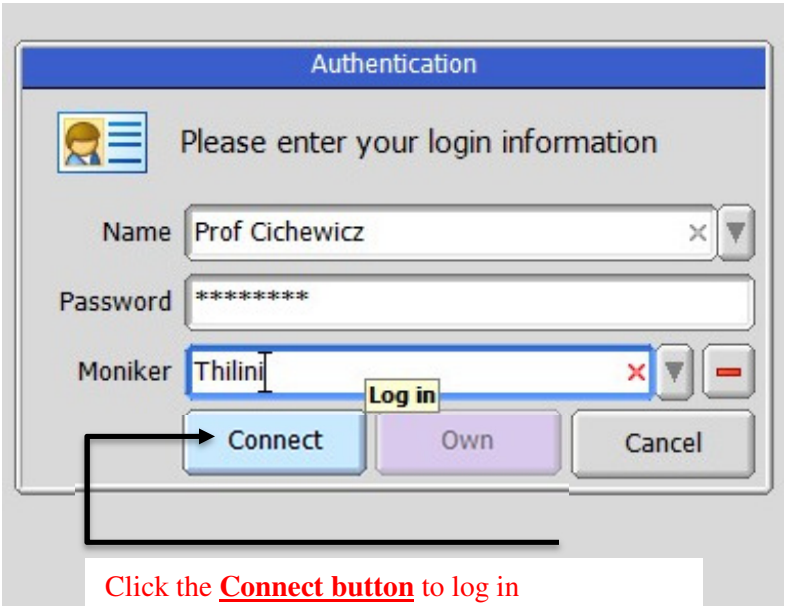


The Authentication dialog window appears. To log into your supervisor's NMR account, you need to follow these steps: (a) type your **supervisor name** or click the arrow on the Authentication window to open the **pull down menu** and select your supervisor name; (b) type your **password** (c) Click the arrow under Moniker to select your **name** on the **pull-down menu**









Loading sample (s)

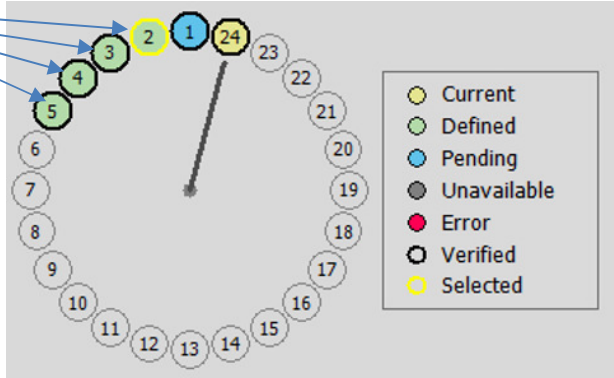
Carefully insert the sample holder(s) into the empty slots (2, 3, 4, and 5) of the automatic sample changer. Do not use slot numbers 1 and 24.

The standard line shape test sample (0.1% $CHCl_3$ in acetone-d_6) should always be in slot #1. It will be used for 3D gradient shimming every 12 hours. Slot #24 is always occupied by an empty NMR tube.

Automatic sample changer (24 slots)

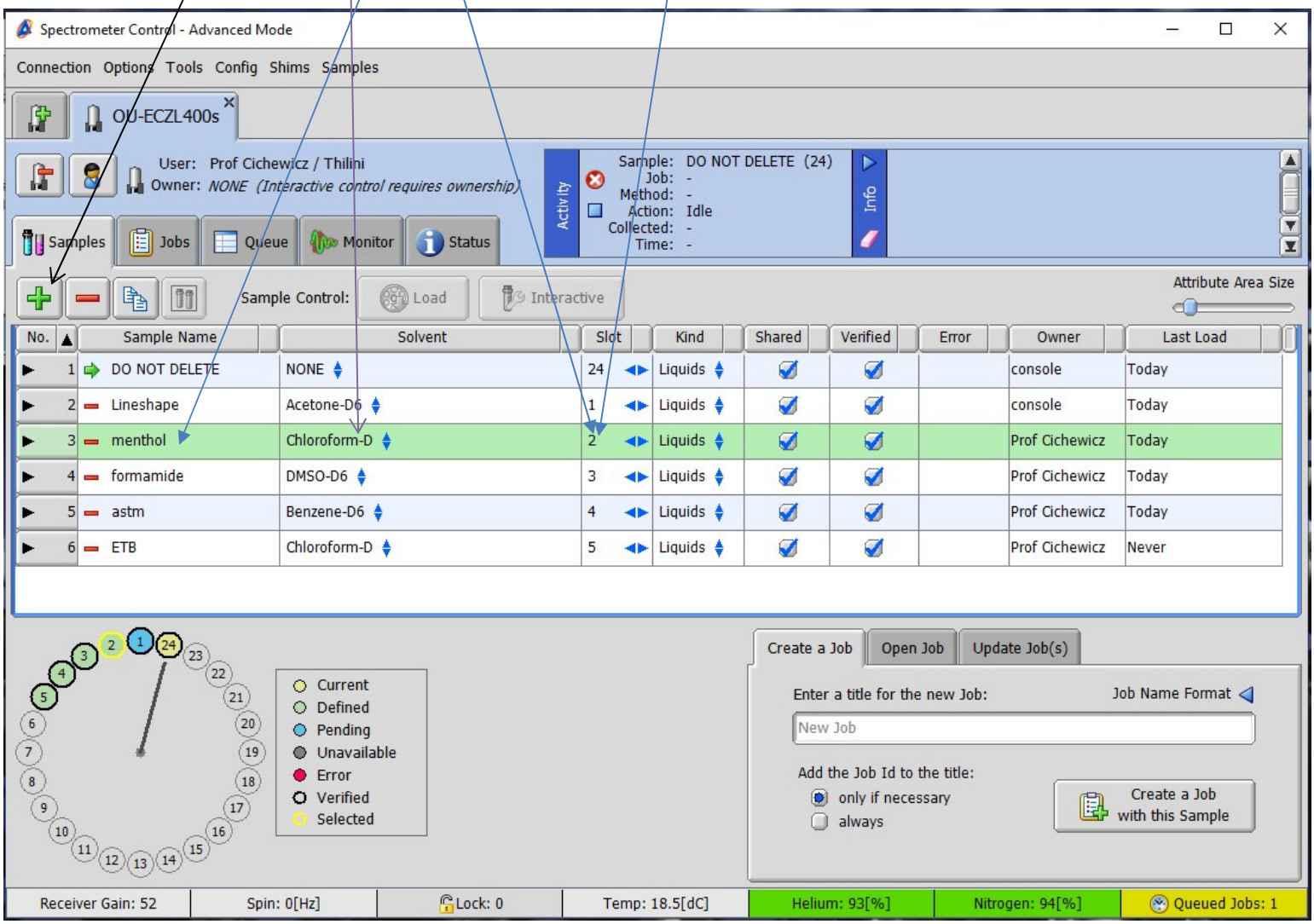
Below are 4 samples created in slots 2, 3, 4, 5 of the automatic sample changer by clicking the button 4 times.

or Double click on an empty slot in the Sample Changer graphic. If one clicks an empty slot (for example slot number 2) in the Sample Changer graphic the slot number (2) will automatically be entered when the sample definition is created



To create a sample definition row, either click on the

Then specify y the sample name, **solvent** and **slot number**



Batch Sample Creation and performing the same experiments (for example Proton and Carbon) for 4 samples (menthol, formamide, astm, and ETB)

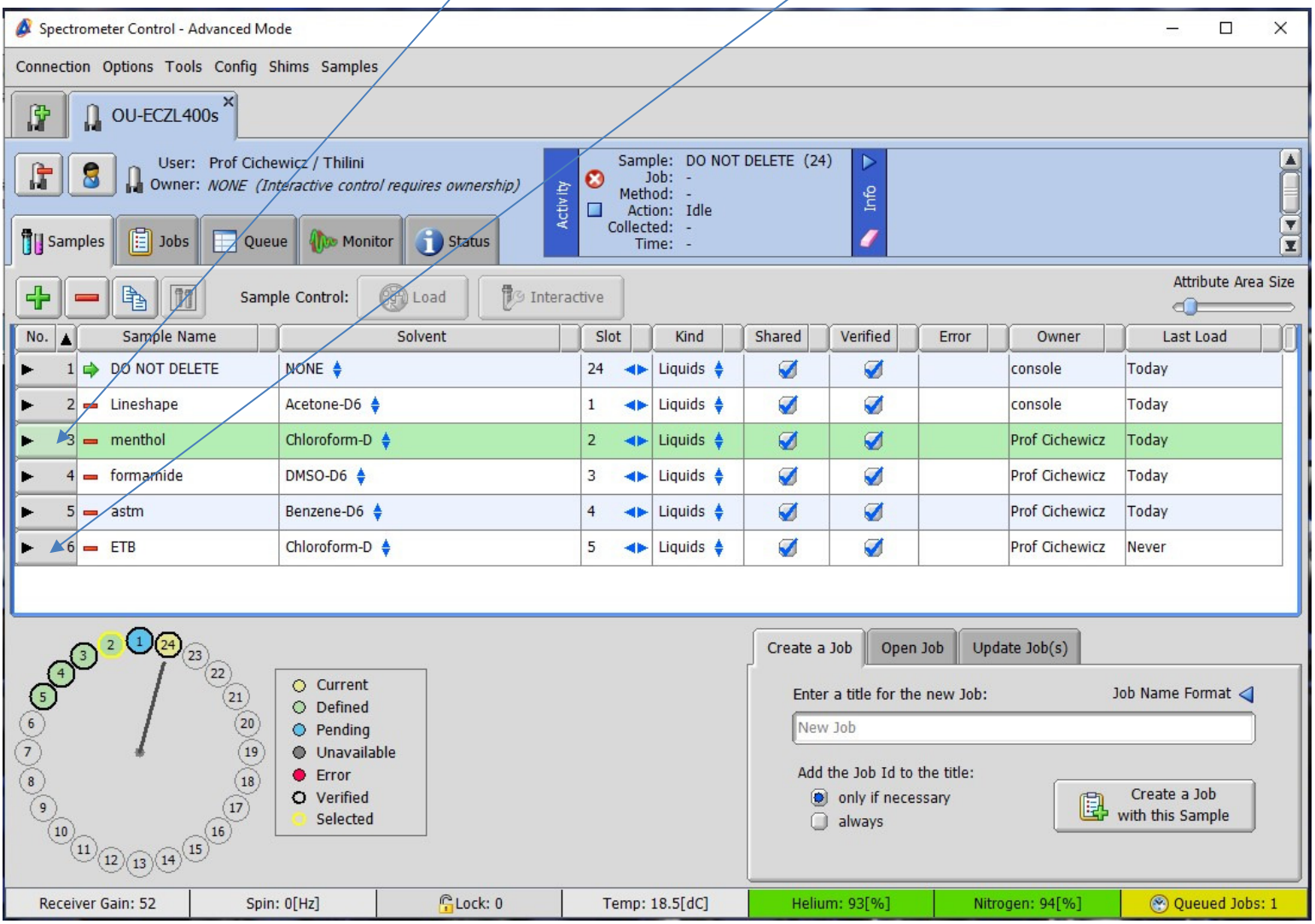
Selecting a contiguous set of samples: Select the first sample, hold the SHIFT button on the keyboard down, select the last sample. All Samples between the first (#3) and last (#6) will be selected.

Shift

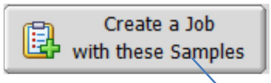
Selecting multiple non-contiguous samples: Select the first sample, hold the CONTRL key down, select additional samples.

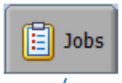
Note: It is important for selection of the entire row click to the right of the black arrow.

Shown below is an example of contiguous samples selection. 1st Sample, SHIFT click last sample

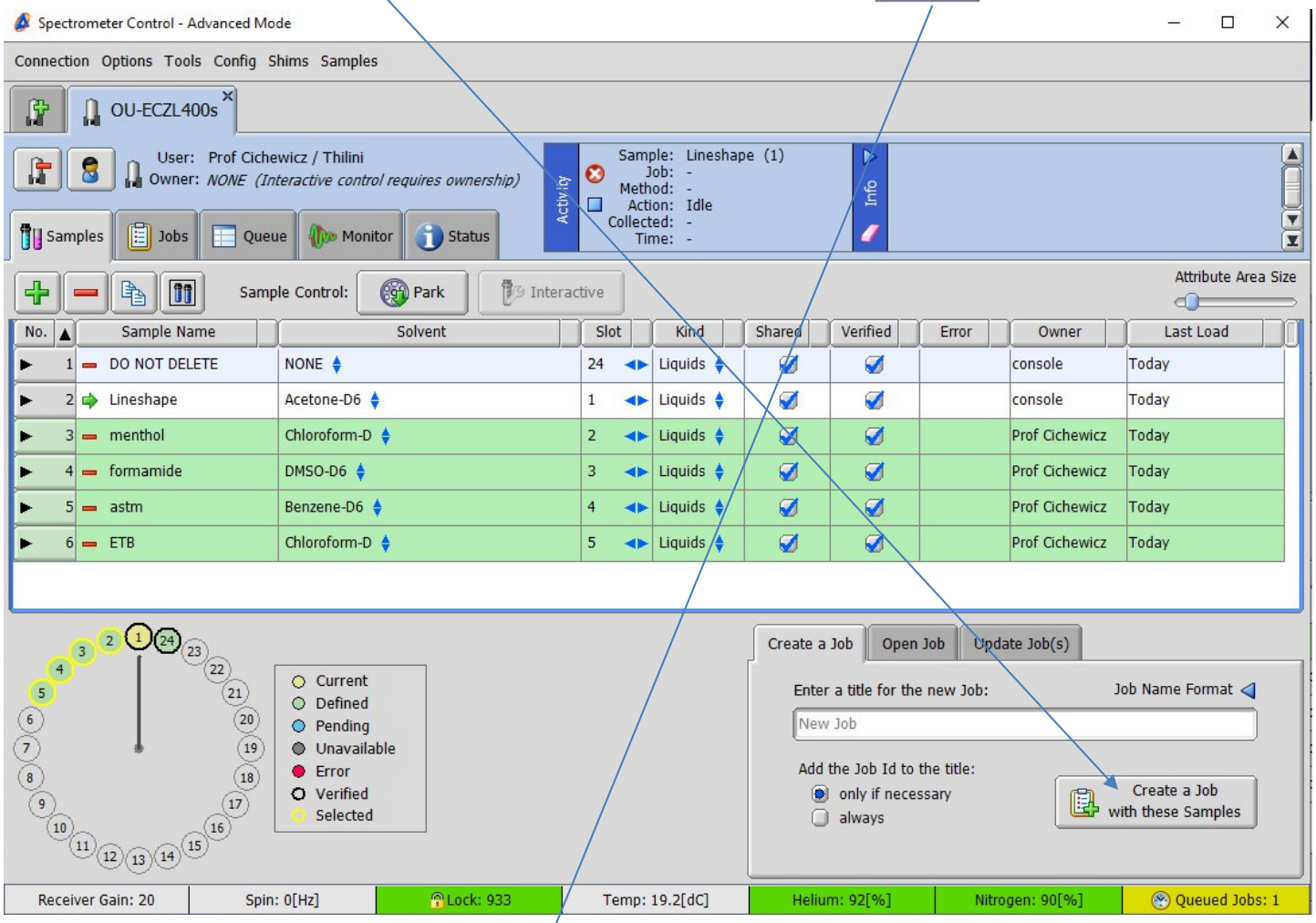


After the samples are selected the rows will be highlighted in light green as shown below.

Hit 

to associate the selected samples with a Job to be created. The Spectrometer Control window will switch to the 

tab.



Spectrometer Control - Advanced Mode

Connection Options Tools Config Jobs

OU-ECZL400s

User: Prof Cichewicz / Thilini
Owner: *NONE (Interactive control requires ownership)*

Samples | Jobs | Queue | Monitor | Status

Activity
Sample: Lineshape (1)
Job: -
Method: -
Action: Idle
Collected: -
Time: -

Info

Open Jobs

New Job

Job waiting to be associated with Experiment (s)

Sample Name	Solvent	Slot	Kind	Preparation	Comment
menthol	Chloroform-D	2	Liquids	TRUE	
formamide	DMSO-D6	3	Liquids	TRUE	
astm	Benzene-D6	4	Liquids	TRUE	
ETB	Chloroform-D	5	Liquids	TRUE	

Samples to run

Proton | Carbon | COSY | DEPT

Available Methods

- Standard
  - Proton
  - Fluorine
  - Carbon
  - Quantitative Carbon
  - P31{1H}
  - P31{19F} optional {1H}
  - 1H{31P}
  - B11{1H}
  - B11{1H},{19F}
  - Deuterium
  - Si29{1H}
  - Proton and Carbon
  - DEPT
  - APT
  - Edited Dept
  - Presaturation
  - Watergate
  - COSY
  - DQF COSY

Available experiments

Add Experiment

Job Parameters: New Job

allow printing	to PDF
folder	Cichewicz
project	

Policy: Choose a scheduling policy

Start: [dd-mmm-yyyy] hh:mm[:ss]

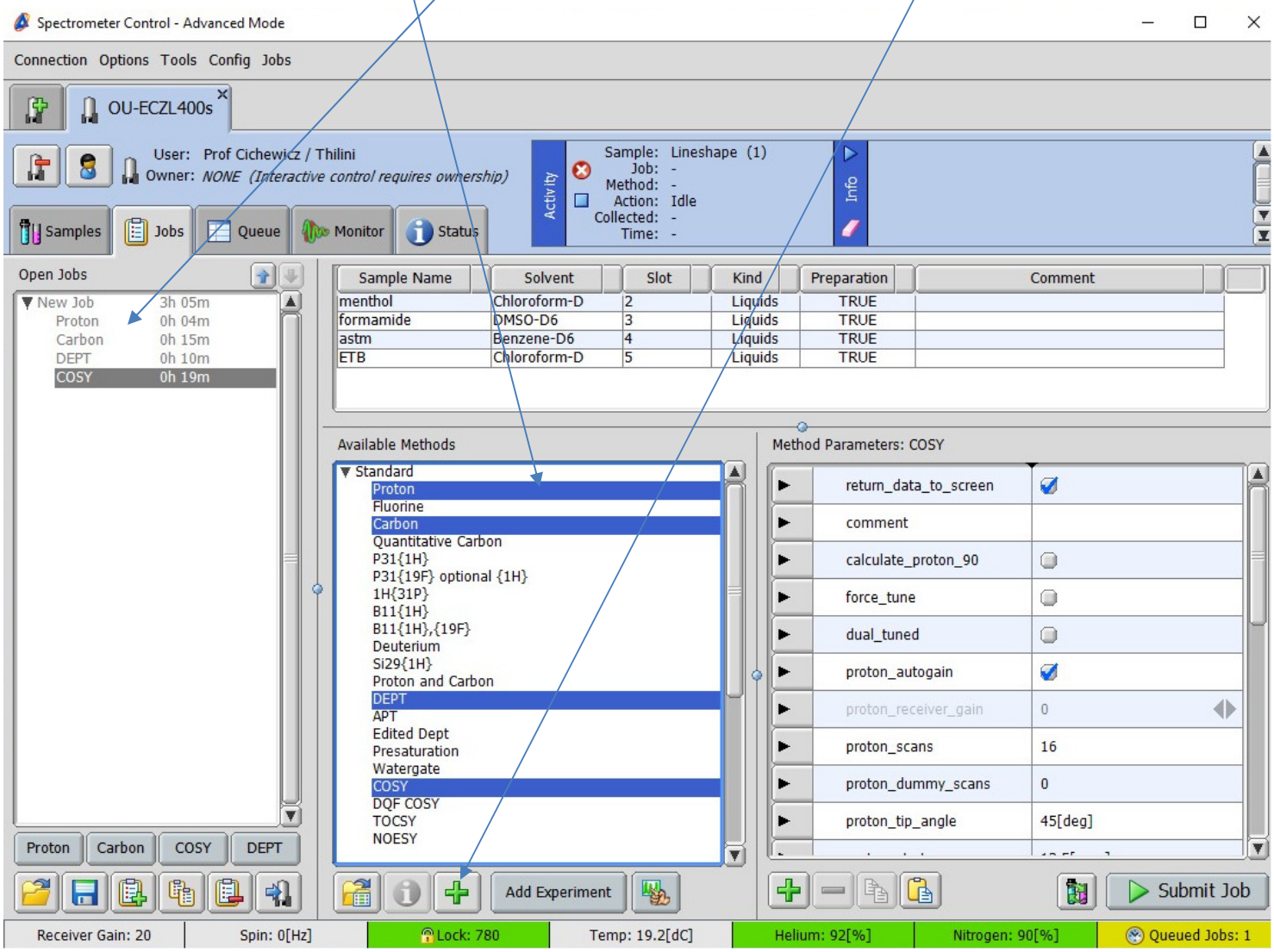
Submit Job

Receiver Gain: 20 | Spin: 0[Hz] | Lock: 874 | Temp: 19.2[dC] | Helium: 92[%] | Nitrogen: 90[%] | Queued Jobs: 1

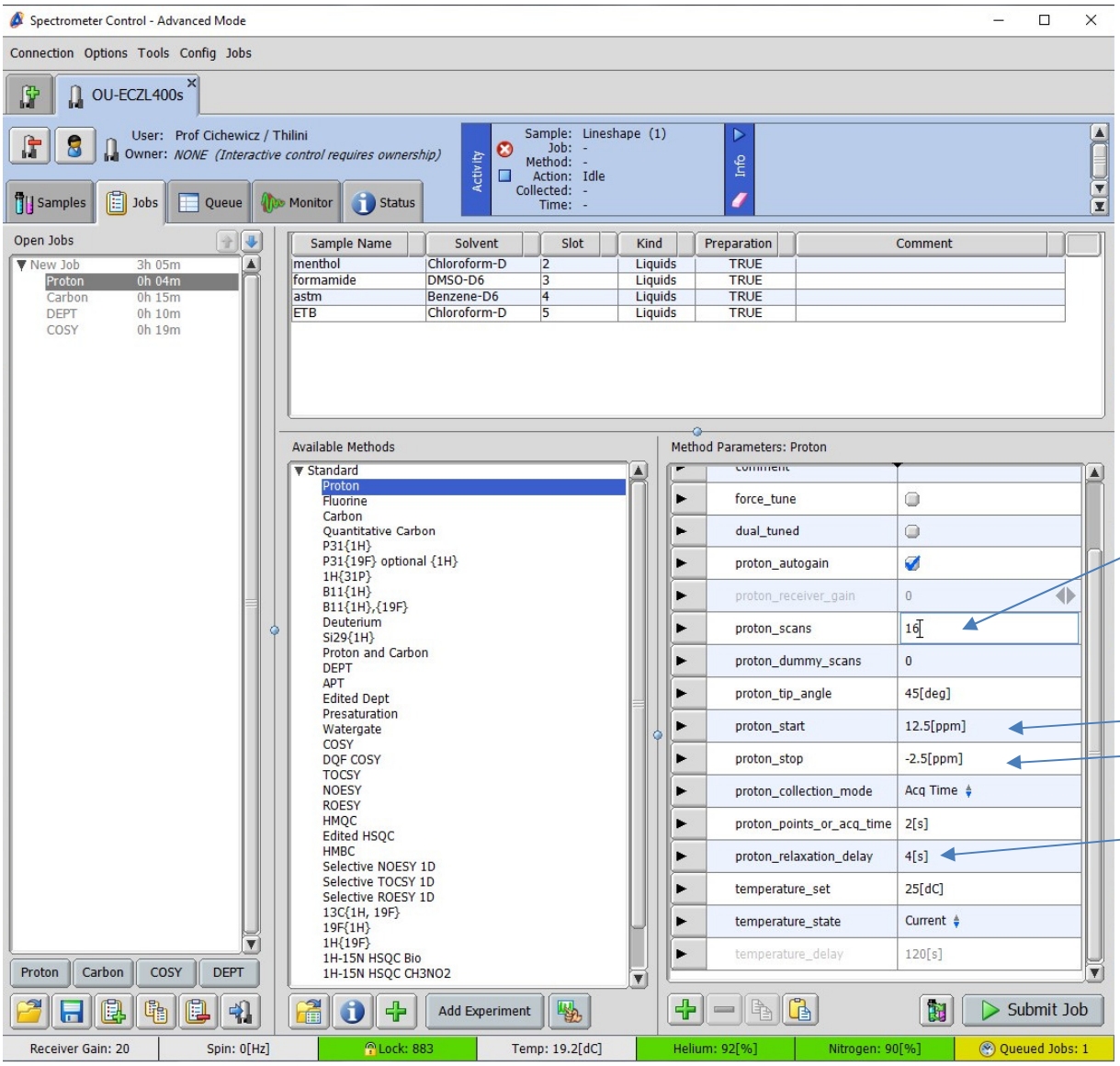
Association of Experiment (s) with Sample (s)

In the middle column are available experiment (s) that can be added to the currently empty Job (New job). Double click on the Experiment tab to add it to the "New Job" column to the left or select the first experiment (Proton), hold the CONTRL key down, select additional experiments (Carbon, DEPT, and COSY) then click the button. Selected experiments are highlighted **in dark blue**.

In this example the Proton, Carbon, DEPT, and COSY experiments are added to the empty "New Job".



The parameters of selected experiments (Proton, Carbon, DEPT, and COSY) that can be **modified** prior to submission.
Adjust "scans" depending on the concentration of your sample.



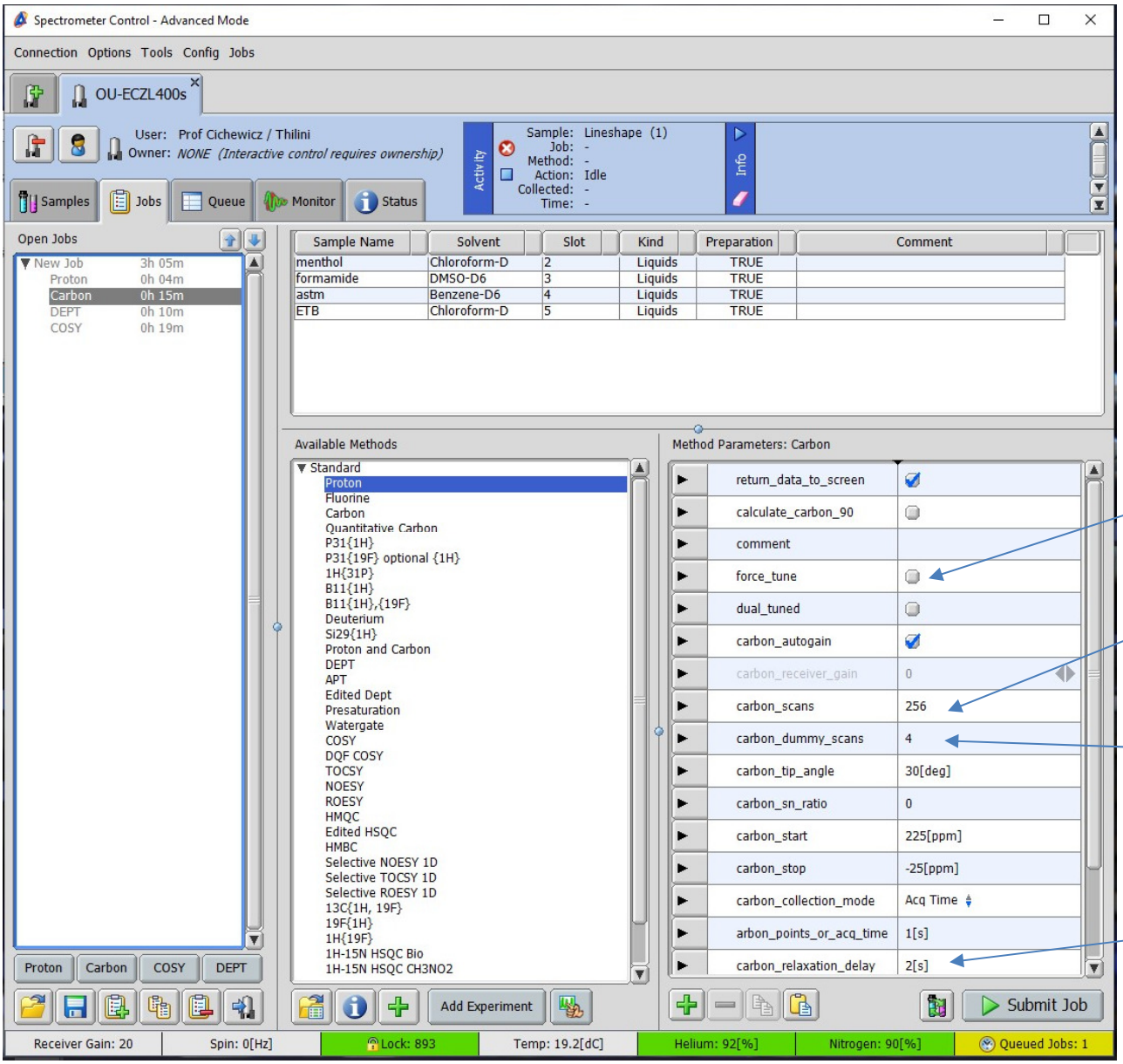
Change the number of scans if necessary. For example, set scans to **4, 8, 16, 32, 64, 128** etc

Change the chemical shifts range if necessary.

Set relaxation delay to **1[s]**

Changing **parameters for Carbon** prior to submission

Adjust "**scans**" depending on the concentration of your sample.



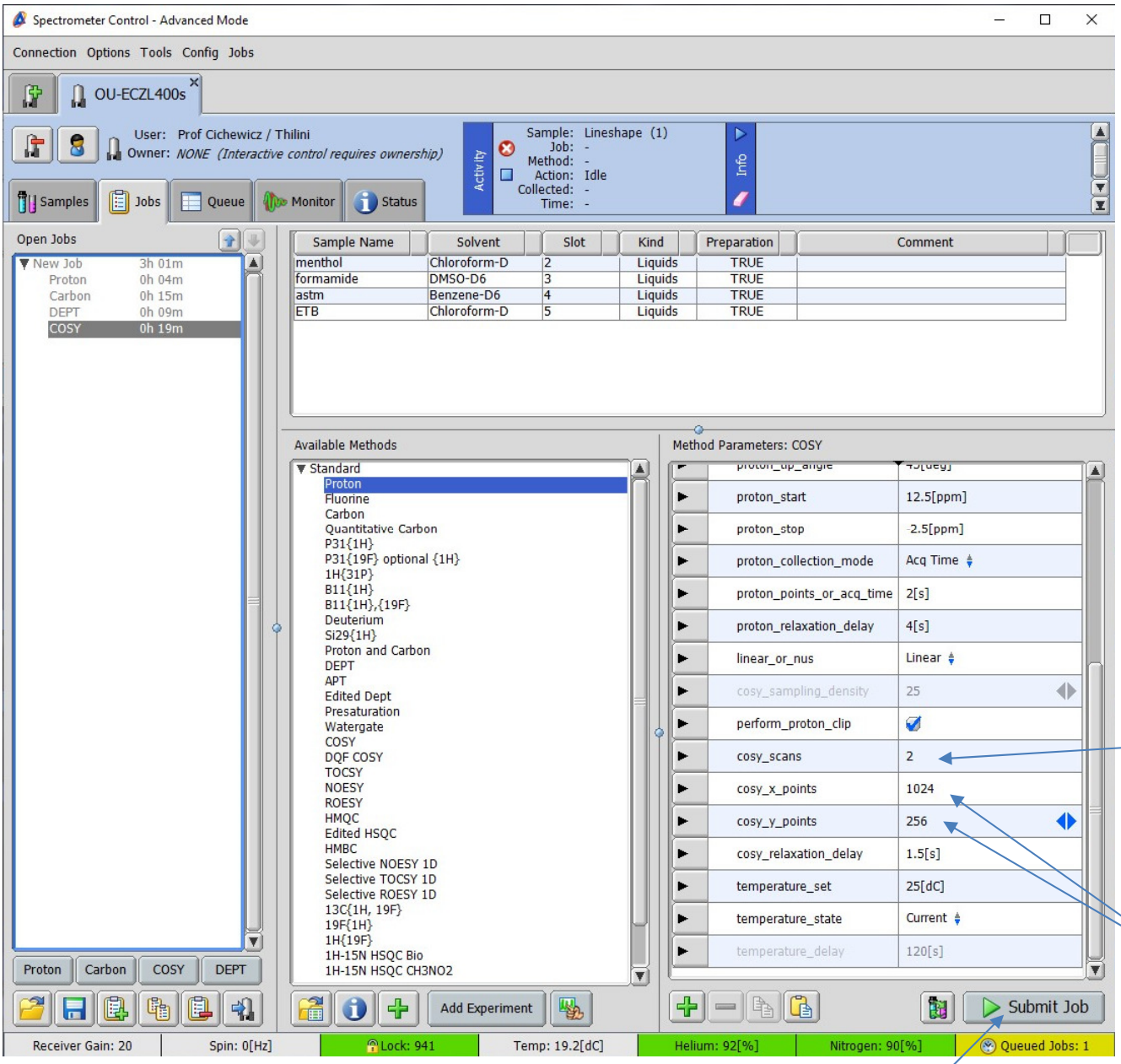
Select force_tune, Click the grey button.

Change the number of scans if necessary. For example, set scans to **128, 256, 512, 1024** etc

Set dummy scans to **0**

Set relaxation delay to **1[s]**

Changing **parameters for COSY** prior to submission

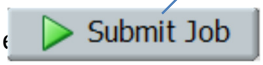


This part of parameters belongs to **PROTON**

Change number of scans if necessary

This part of parameters belongs to **COSY**

Set x-points to 2048 Change y points to **512** if necessary

After changing the parameters of the selected experiments, click the 

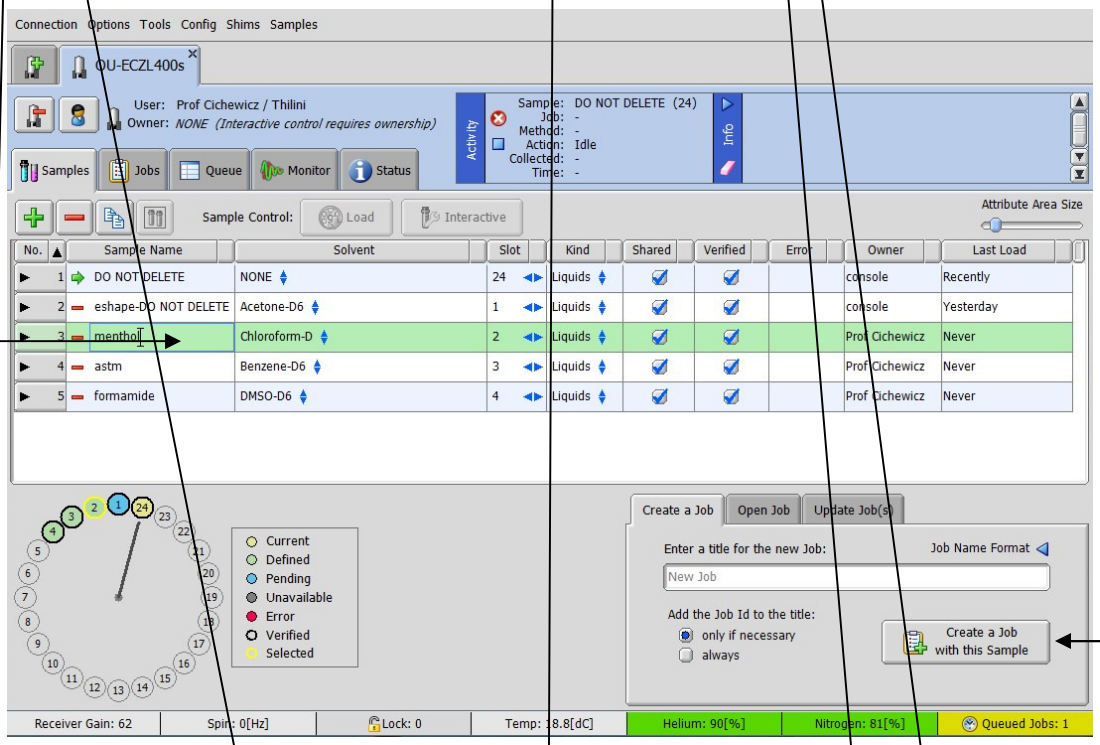
button to start the experiments and to add the Job to the queue.

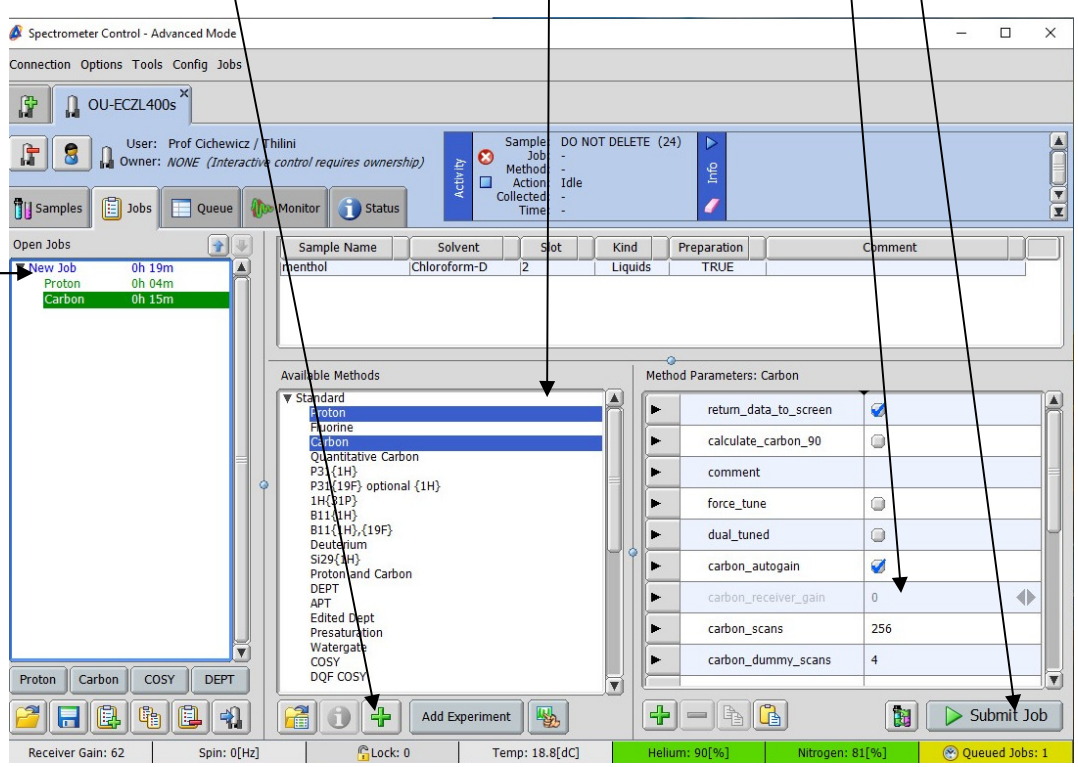
Creation multiple samples (for example 3) and performing various experiments on individual samples automatically.

For example, if you want to run experiments such as 1H, 13C, only on the menthol sample that is in **slot number 2**. Select the menthol row, which will be **highlighted in green**, and then click the **Create Job button with the sample**. A new job is being created. In the list of experiments, select the "**Proton" and "Carbon"** experiments and click the **"+" button** to add them in the "New Job" window. **Modify parameters** (number of scans, relaxation delay, and dummy scans set to 0.) for each experiment before submission. The number of scans is adjusted depending on the concentration of your sample. Once all required parameters have been changed for each experiment, click the **Submit Job** button to begin collecting data.

Click

Click





The same steps apply to formamide, and ASTM samples.

Select raw formamide (slot #3), create a job with this sample, under the newly created job add the experiments you want from the list of experiments like **DEPT, Edited-HSQC, NOESY**; Change the required parameters before submitting. After changing the parameters, click the "Submit Job" button to collect the corresponding NMR spectra.

Select raw astm (slot #4), create a job with this sample, under the newly created job add the experiments you want from the list of experiments like **APT, HSQC**; Change the required parameters before submitting. After changing the parameters, click the "Submit Job" button to collect the corresponding NMR spectra.

All submitted jobs (experiments) will be queued. The locking, shimming, and tuning will be performed automatically for each sample.

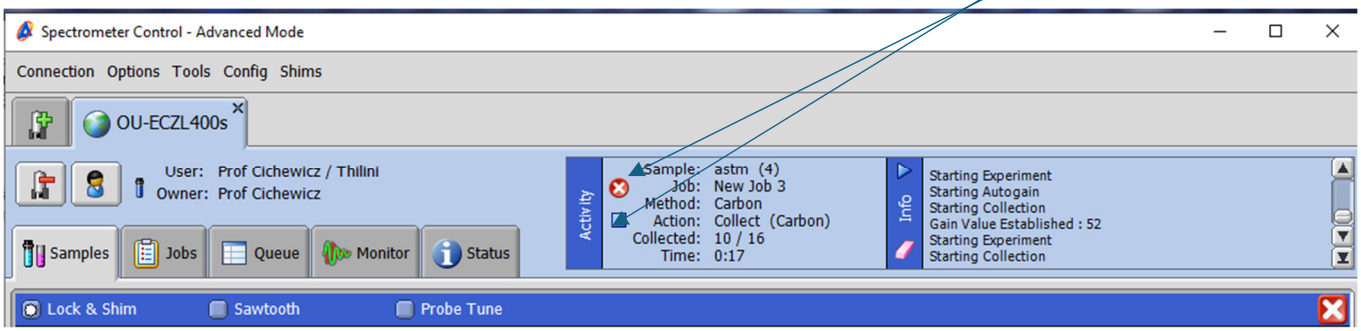
Once data collection is complete, a spectrum will be automatically displayed for each sample.

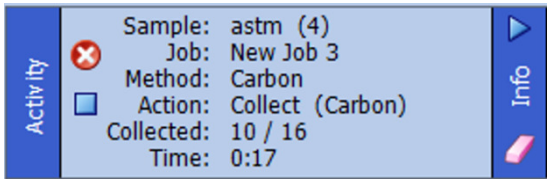
SAVING NMR DATA (FID)

When measurement is complete, the data are saved automatically with the specified name.

Stopping an Experiment

There are two ways to stop an experiment during the acquiring data: In the activity bar click the button; experiment stops immediately without waiting for the end of accumulation or click the button: experiment stops after accumulation is completed.





Deleting sample names from the Spectrometer Control window after completing experiments.

Select a sample raw then click the button above the sample tab. The sample raw will be deleted. Also you can delete the sample by clicking the button on the left side of each sample name.

If multiple samples are selected using the Shift Control keys you can delete the samples as batch.

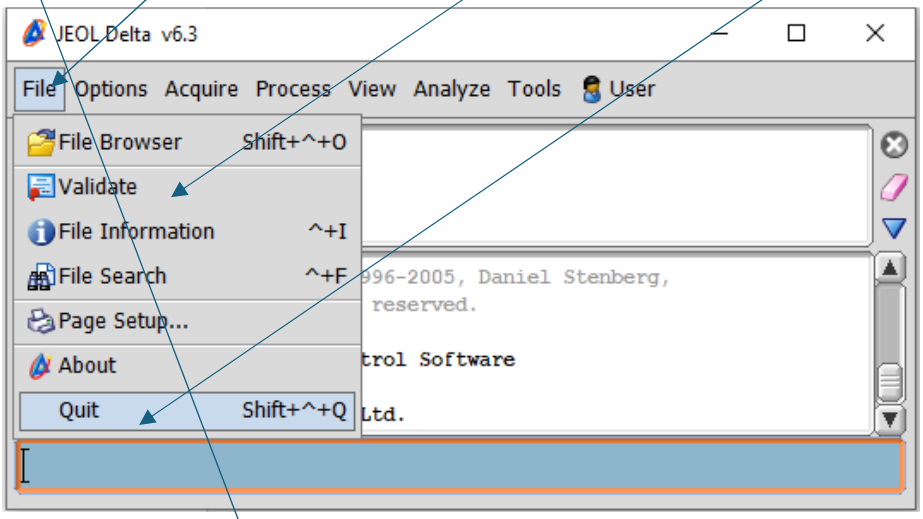
Deleting a JOB

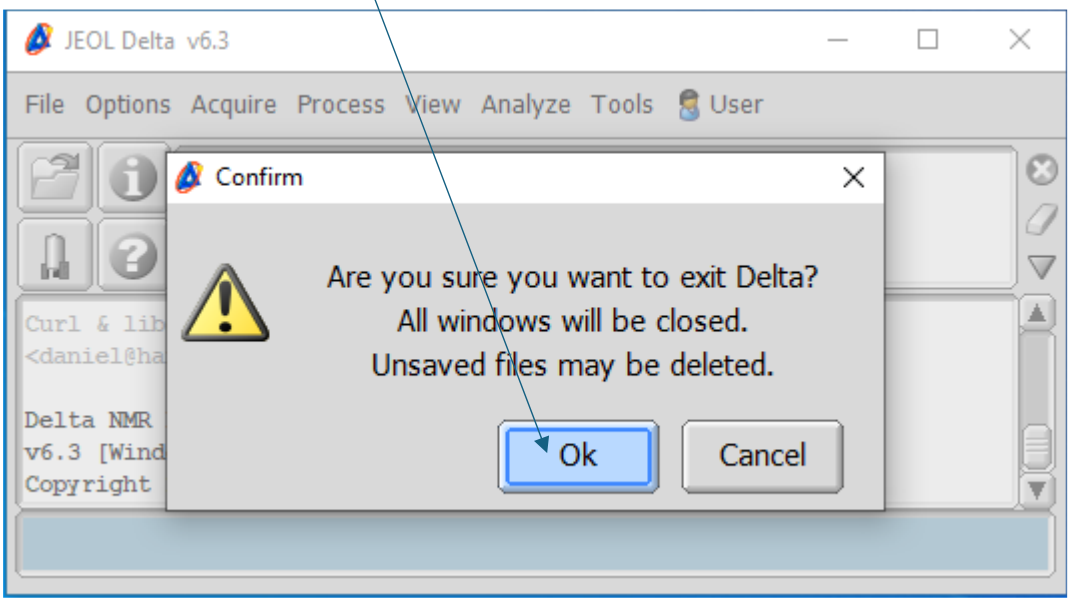
You can delete unnecessary Jobs that have created by mistake by using the button below the Job list. You can also delete multiple Jobs selected by suing Shift and Ctrl keys as a batch.

Ending the Delta program

After measurement or NMR data is complete terminate the Delta program as follows:

In the Delta Console window select File a drop-down menu will open then click the Quit button and confirm your selection by clicking OK. The Delta program ends. All available windows on the screen will be closed automatically.



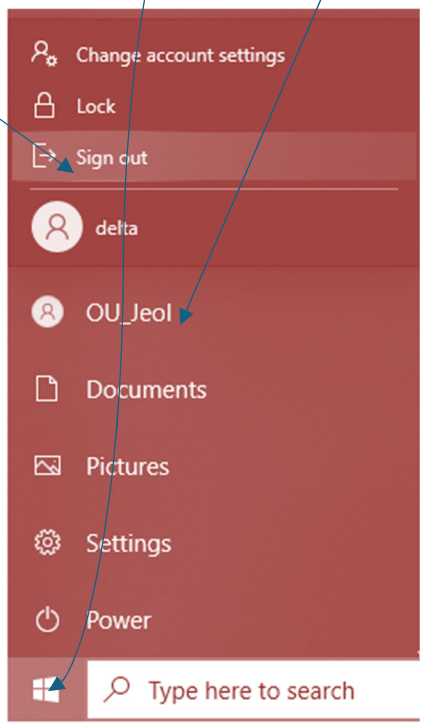


Data Processing using DELTA software is optional to learn. All data

processing referencing integration pick picking and base line corrections should be done on your own computer using MNOVA software.

Exit from the OU_Jeol account.

When you are completed your NMR experiments you need to logout first from your account; secondly from the OU_Jeol account)

Before logging out from your account be sure there are no experiments in progress. You must stop your experiment before logging out from your account.

Click on the left bottom corner the button of NMR computer screen; a menu window opens; Click the **OU_Jeol** button then click the **Sign out** button to exit from the OU_JEOL NMR account