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Faculty of Science/
Department of Chemistry/NMR Facility
nmr.chem.ualberta.ca

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

Bruker Icon NMR
UofA Chemistry Step-By-Step Visual Reference Guide

These instructions are currently specific for the new (*i.e.* 2023) Bruker Avance NEO i400 (Room SB-3E) with 60 position SampleCase robot, and will also be applicable to the Bruker Avance NEO ibd500, and 800MHz spectrometers with TopSpin Version 4.x ICON NMR and slightly smaller SampleCase robotic sample handling.

UNIVERSITY OF ALBERTA
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nmr.chem.ualberta.ca

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

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See the subsequent pages for detailed explanation, examples with pictures.

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

Special Notes

Caps Lock Disabled

The caps lock button is disabled on all workstations and spectrometer. Users would leave on and cause havoc.

Do NOT reboot computers

If the computer is not working, frozen, or otherwise misbehaving tell NMR staff.

Do not try to fix yourself.

Processing, printing, annotating etc.

That is meant for workstations, please free up spectrometers as soon as you for other users.

Pick up samples when done.

Don’t leave samples on the robots.

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Faculty of Science/
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Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

How to Run a Bruker Automated NMR Sample

1.) Login … but make sure the last user logged Out

The screen should be asking for Login (See Identify User Panel in **Figure 1**). Scroll through the list or type your name to search and then select your account by double clicking the correct login or select and hit "OK" button.

If the last person has forgotten to logout, or entered the wrong password, you will see **Figure 2**.

If this is the case, please log them out and/or try the password again by using the "Change User" button in the top right. (**Figure 2** – see Red Box and Red Arrow indicating Change User Icon).

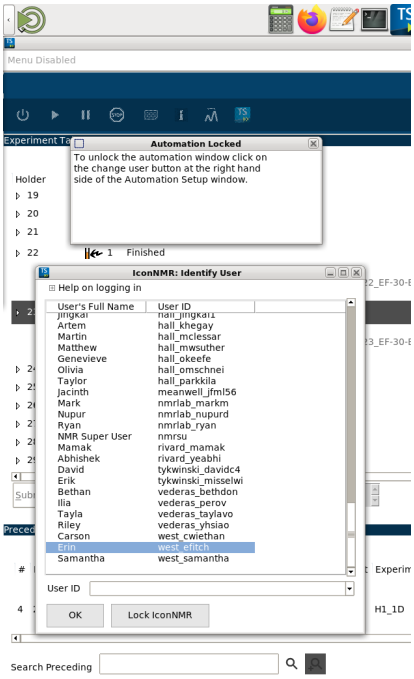

Figure 1 *– Correct Start Screen*

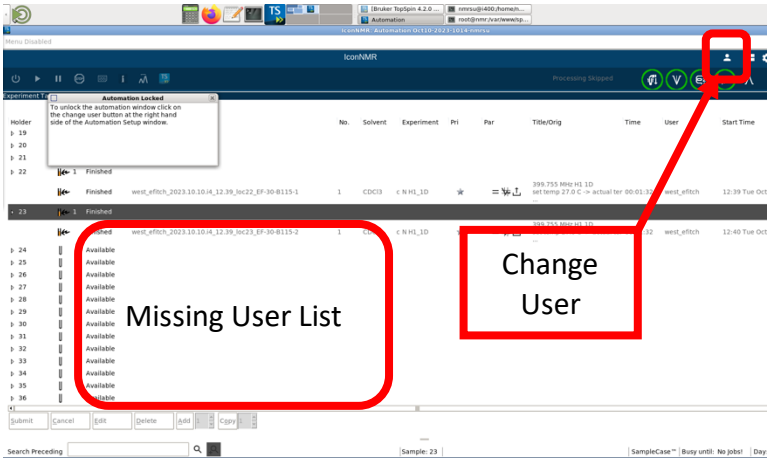

Figure 2 *– Last User Forgot to Logout*

**** DO NOT FORGET TO LOGOUT when done with "Change User" every time, otherwise the next person could use the wrong account and your lab will be charged $$ for the experiments,…** [1]

[1] *…and users will never find their data…great sadness and confusion…re-invoicing of charges, …etc.*

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

2.) Your "password" is required.

Please enter your:

Personal Online User Registration Password

NOT your lab's account password.

*** This is different from the Varian/Agilent Spectrometers**

Yes, the password you use to book spectrometers and entered when you registered. Forgot? Contact NMR staff.

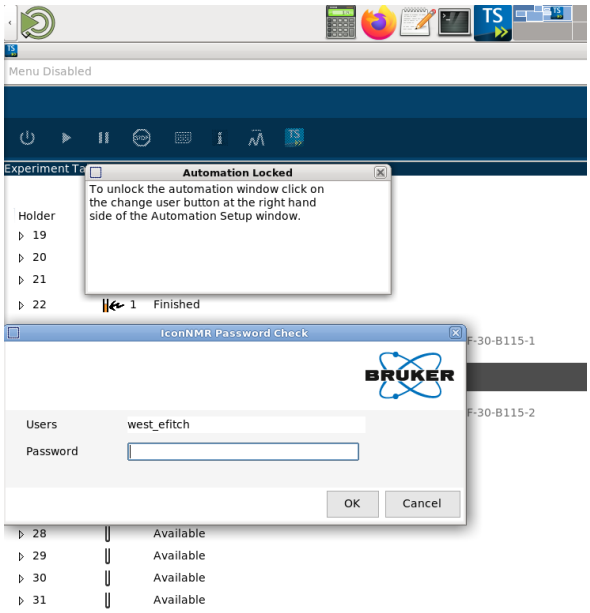

Figure 3 *– Enter your online user password*

3.) Preparing and Loading your Sample

Go over to the sample "Rack" (see **Figure 4**) and select an open position. Attach your label on the rack at the correct location of the spinner being used (see Spinner numbering in **Figure 5**). If all positions are used, you will have to retrieve a **finished** sample from the SampleCase robot and clear a space in the software (see below). Check the Bruker TopSpin-Icon software for which samples have run and are done. When you've found a finished sample, remove the sample from the spinner, reattach the corresponding label (if any) from the spinner rack back onto the NMR tube, and carefully place the re-labelled sample inside the sample return holder (**Figure 8**, next page). You may then use the spinner.

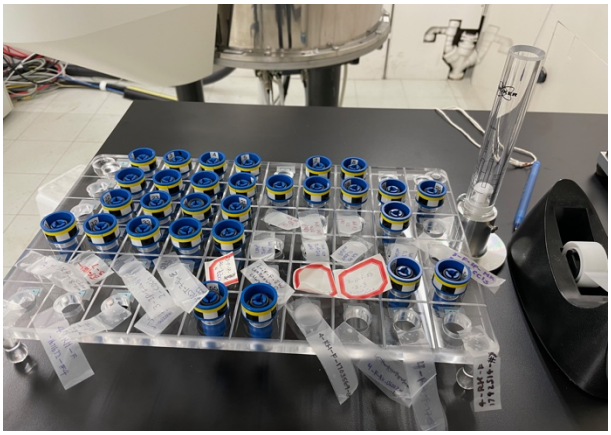

Figure 4 *– Examples of spinners in use and labels at sample positions*

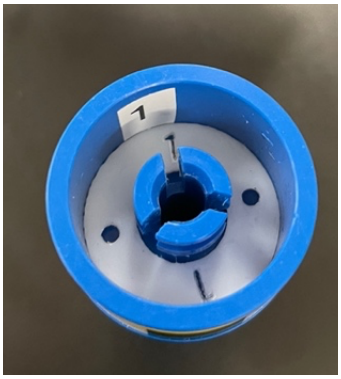

Figure 5 *–Example of Spinner Numbering*

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

Notice the spinner numbers. Always match the correct robot and storage rack position for the correct spinner number.

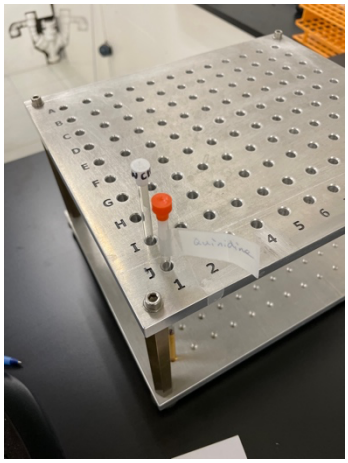

Figure 8 *– Sample Holder*

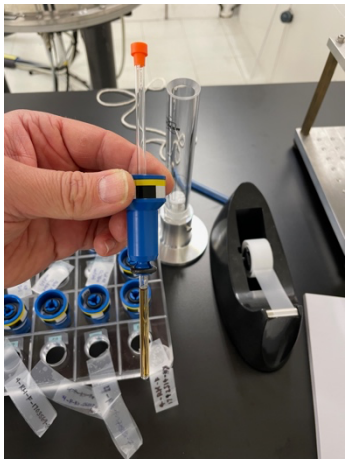

Figure 7 *– Sample in Spinner*

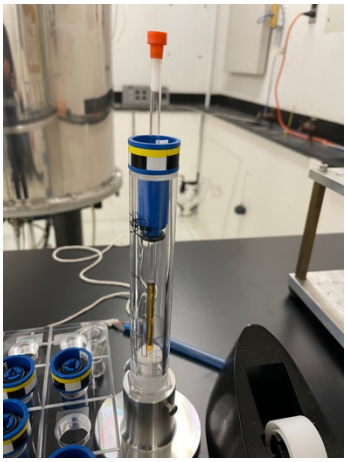

Figure 6 *– Depth Gauge – sample set properly*

Insert your sample in the spinner (**Figure 7**) and set the sample depth using the gauge (**Figure 6**). Then place your sample/spinner in the robot at the matching carousel number for your spinner number.

When placing the sample in the carousel, make sure the carousel is not moving. Gently lower sample into position, do not drop.

Figure 10 *– Emergency Stop and Reset Button*

Do NOT bump/hit the Height Detect Bar (**Figure 9** Red Box). If you do, everything will halt as the robot will panic, and the lights will be red.

Please contact NMR staff and get help.

Figure 9 *– Height Detect Bar*

Emergency Stop Button

You may use the Emergency Stop (**Figure 10**) to halt operations if needed (*e.g.* robot is damaging equipment/misbehaving or hurting someone, *etc.*). Press the red button down, you'll hear a clunk. Contact NMR staff immediately.

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

4.) Selecting Sample Position

Back at the computer you will now need to select the carousel position where you have placed your sample. In the example below (**Figure 11**), Sample 1 is busy acquiring, but the other positions are available to select.

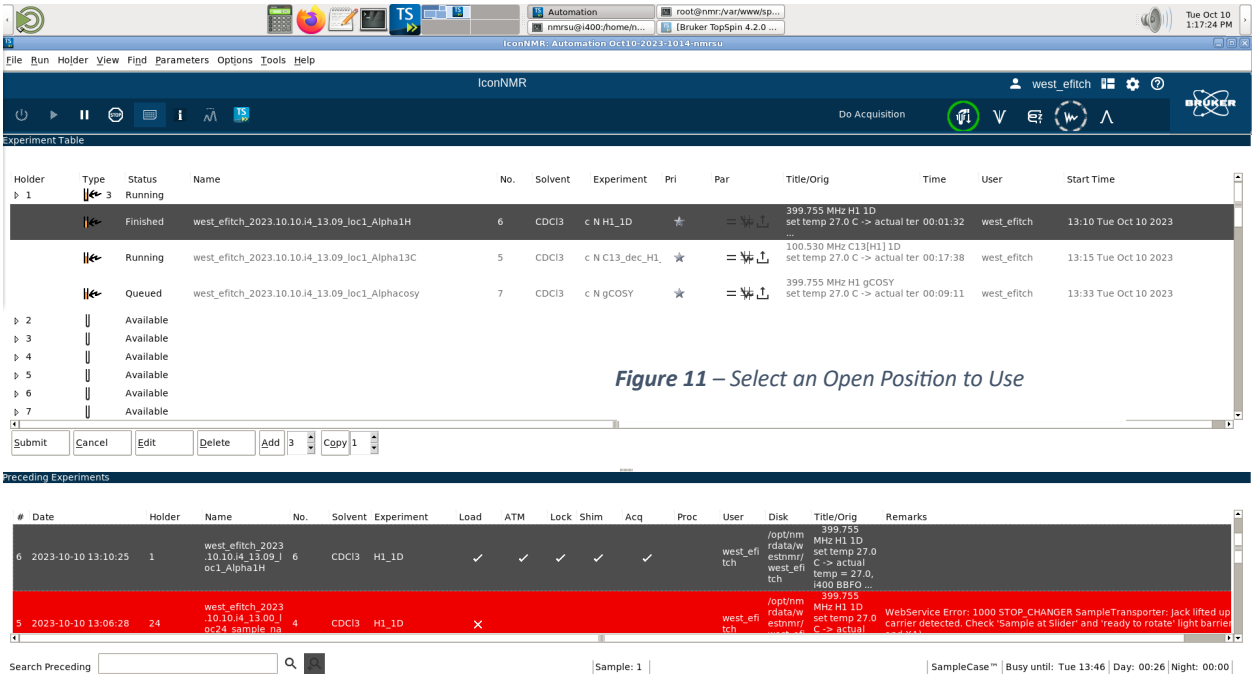

***Figure 11** – Select an Open Position to Use*

You may also scroll further down the sample list for more open positions (**Figure 12**).

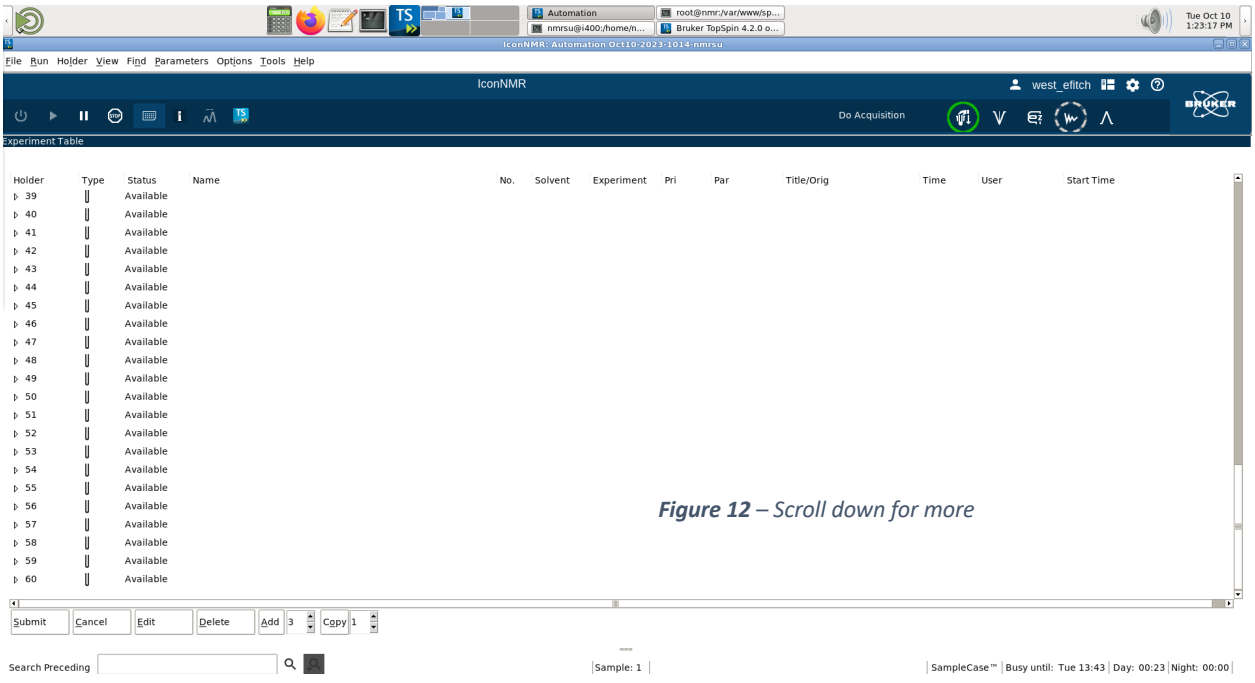

***Figure 12** – Scroll down for more*

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Department of Chemistry/NMR Facility
nmr.chem.ualberta.ca

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

5.) Free up a used Sample Slot (if all spots used)

If all sample positions are used, you will need to release a "Finished" sample spot. **Figure 13** shows an example of us selecting Sample 24 (*i.e.* not the experiment slots) then going down to the "Delete" option to remove the entire sample selection (e.g. we changed our mind). You may remove "Finished" samples (see Status window below), or a sample you were about to submit. Another option is to right-click the Holder Number, and select "Delete" if finished.

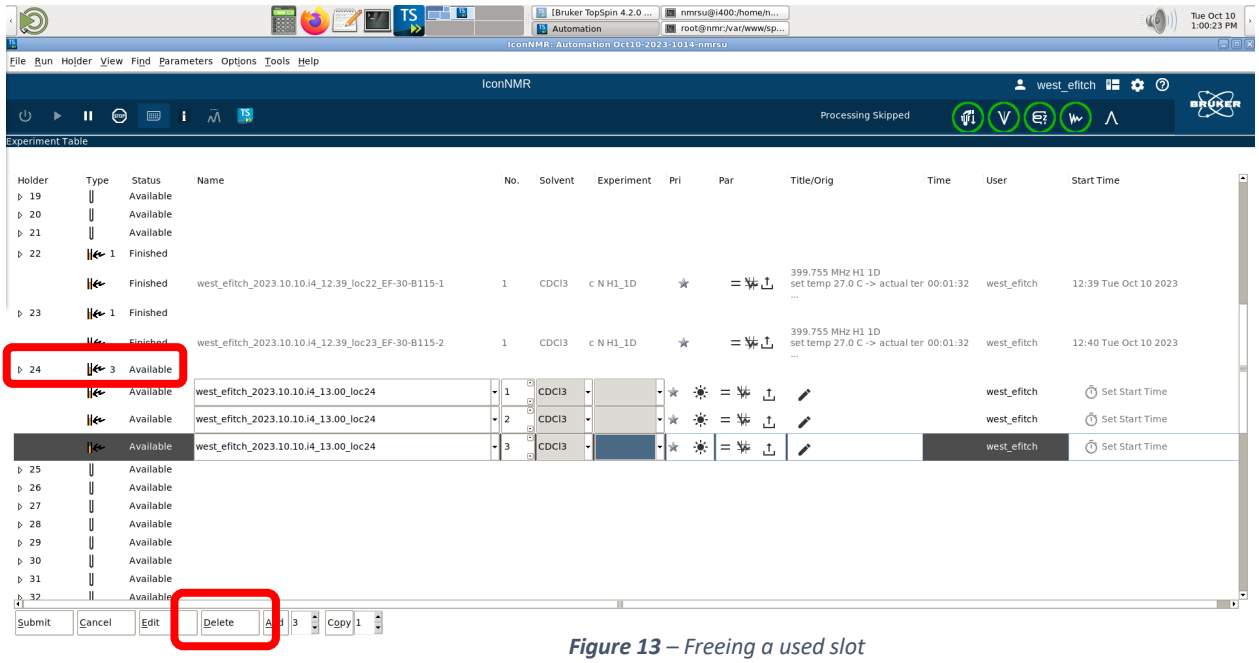


Figure 13 *– Freeing a used slot*

ONLY remove Status = "Finished" samples. Never try to interfere with a running sample, or samples submitted to the queue.

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

6.) Add Experiments for the Sample

In this example we have clicked on and chosen position 24 (left box) in the list, then opened "3" slots by selecting three experiments (bottom box) then hitting the "Add" button (see **Figure 14** red boxes). We now have 3 open experiment slots to use, we can add more, cancel, remove later, or modify as desired.

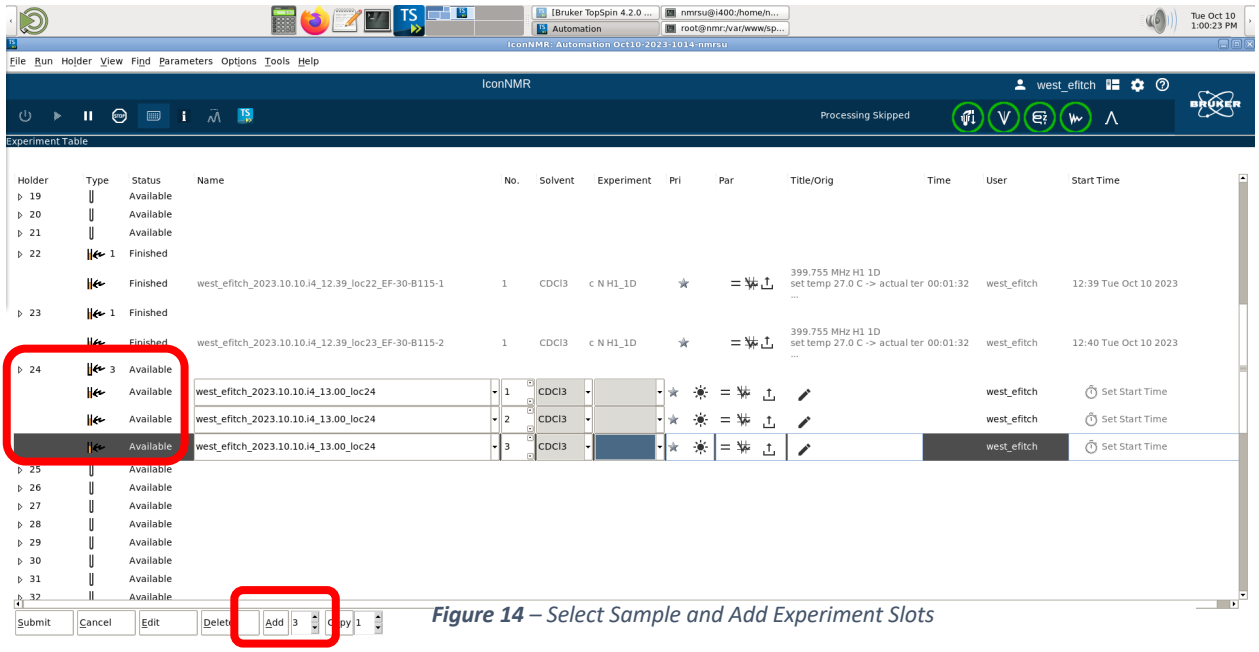


***Figure 14** – Select Sample and Add Experiment Slots*

It is important to note that the position number itself (*e.g.* 24) doesn't let you do anything until you add experiments.

7.) Name the Sample Experiment in EACH Experiment Slot

In the example below (**Figure 15**) we've named the first experiment "_sample_name", the second"YYY", third "XXX". Name yours appropriately, and remember we add date, instrument, time, *etc.*

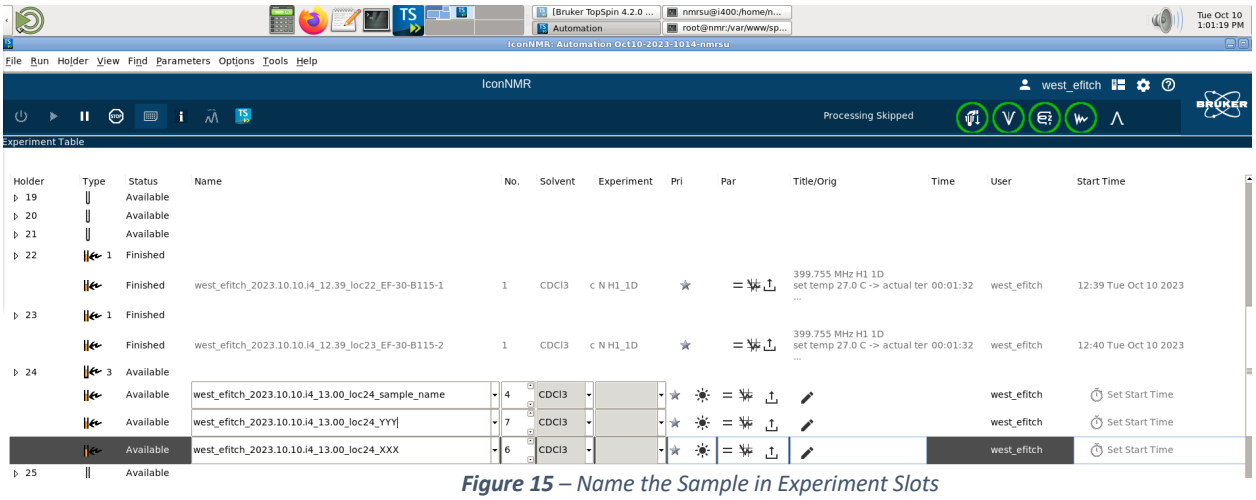


***Figure 15** – Name the Sample in Experiment Slots*

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

8.) Select Solvent

For each experiment slot, please specify the solvent for your sample (see **Figure 16** and **Figure 17**).

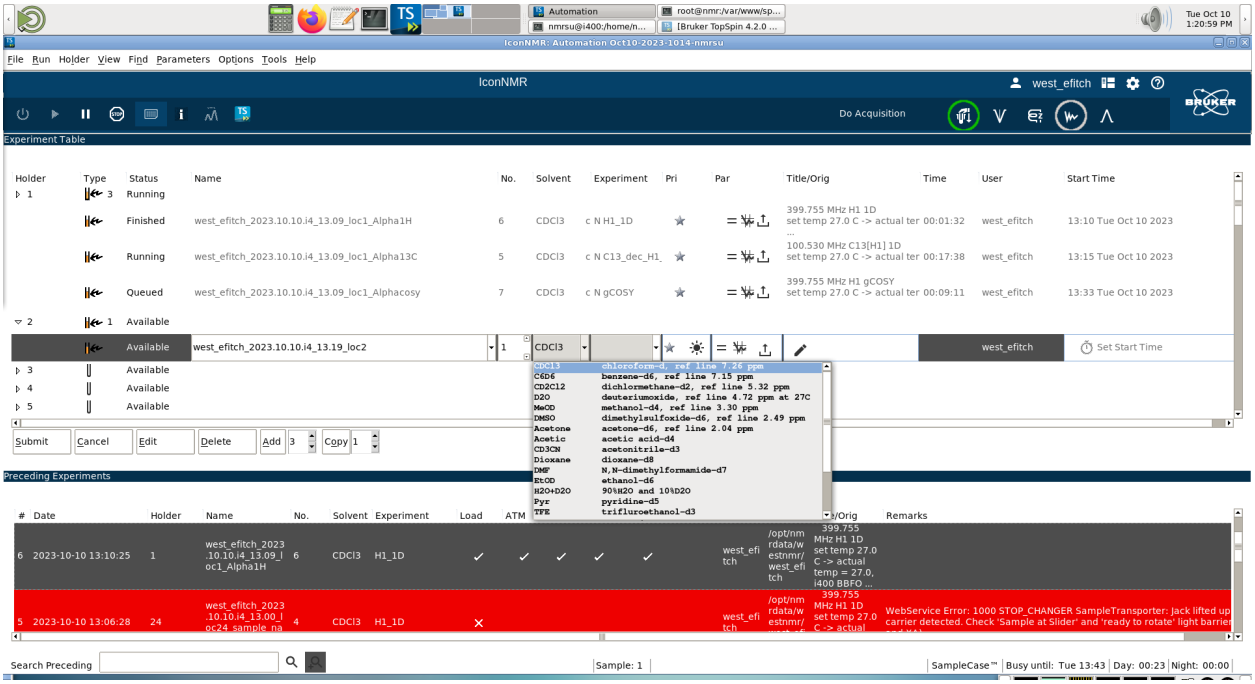


Figure 16 *– Solvent selection*

Close up view of solvents, scroll down for more options.

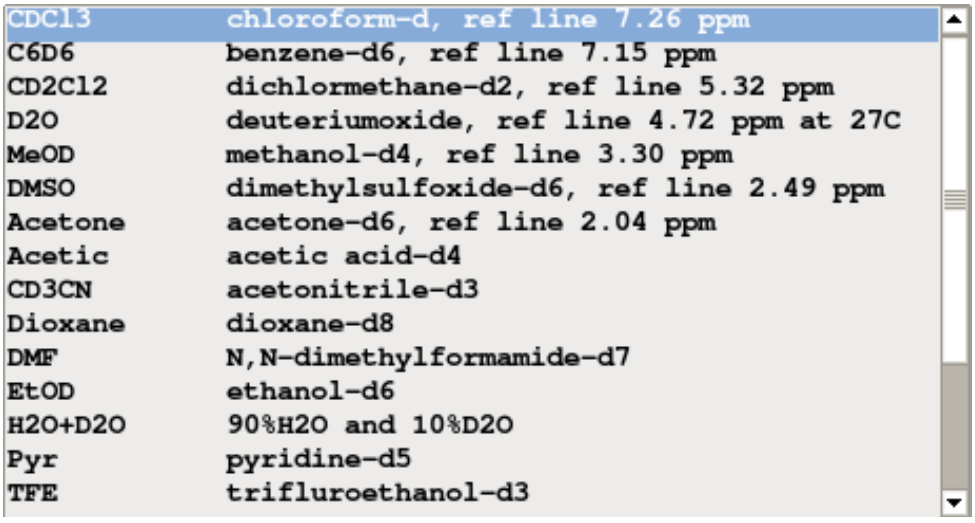


Figure 17 *- Zoom in of available solvents*

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

9.) Select Experiments

From the drop-down list (**Figure 18** and **Figure 19**), select the desired experiment for each slot. If you want more experiments, add more slots.

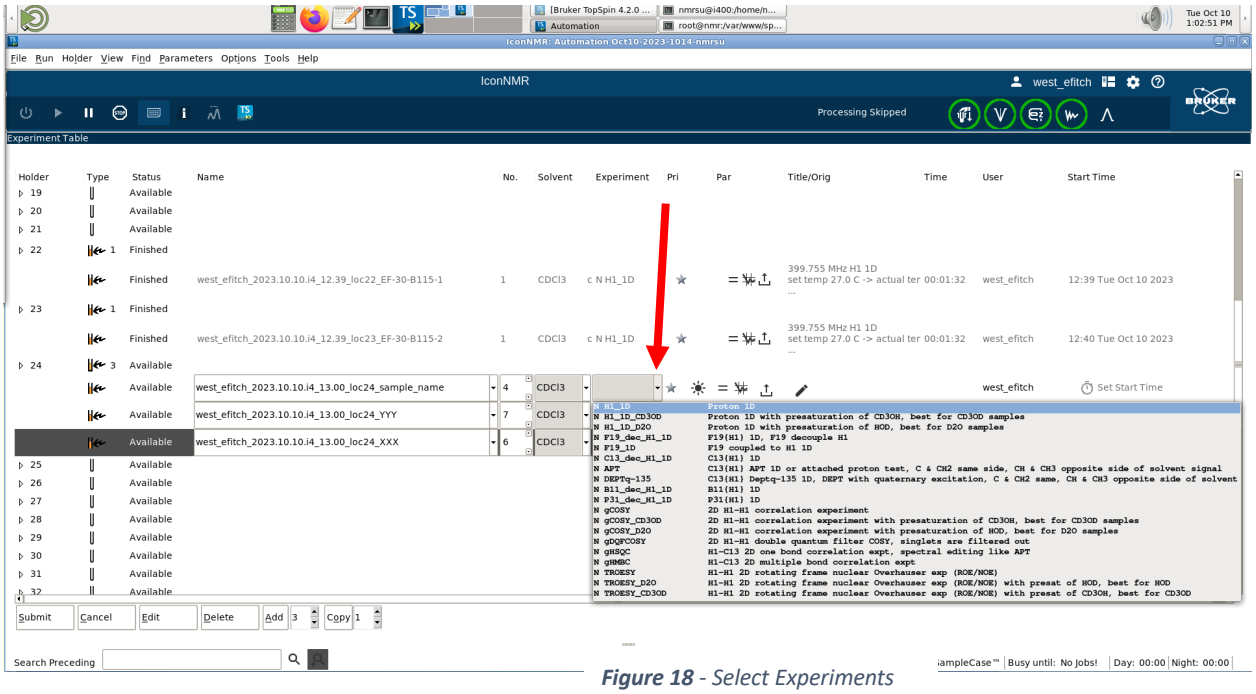


Figure 18 *- Select Experiments*

Below is shown a zoom-in of the available experiments pre-approved for Chemistry samples.

```
N H1_1D                 Proton 1D
N H1_1D_CD3OD           Proton 1D with presaturation of CD3OH, best for CD3OD samples
N H1_1D_D2O             Proton 1D with presaturation of HOD, best for D2O samples
N F19_dec_H1_1D         F19{H1} 1D, F19 decouple H1
N F19_1D                F19 coupled to H1 1D
N C13_dec_H1_1D         C13{H1} 1D
N APT                   C13{H1} APT 1D or attached proton test, C & CH2 same side, CH & CH3 opposite side of solvent signal
N DEPTq-135             C13{H1} Deptq-135 1D, DEPT with quaternary excitation, C & CH2 same, CH & CH3 opposite side of solvent
N B11_dec_H1_1D         B11{H1} 1D
N P31_dec_H1_1D         P31{H1} 1D
N gCOSY                 2D H1-H1 correlation experiment
N gCOSY_CD3OD           2D H1-H1 correlation experiment with presaturation of CD3OH, best for CD3OD samples
N gCOSY_D2O             2D H1-H1 correlation experiment with presaturation of HOD, best for D2O samples
N gDQFCOSY              2D H1-H1 double quantum filter COSY, singlets are filtered out
N gHSQC                 H1-C13 2D one bond correlation expt, spectral editing like APT
N gHMBC                 H1-C13 2D multiple bond correlation expt
N TROESY                H1-H1 2D rotating frame nuclear Overhauser exp (ROE/NOE)
N TROESY_D2O            H1-H1 2D rotating frame nuclear Overhauser exp (ROE/NOE) with presat of HOD, best for HOD
N TROESY_CD3OD          H1-H1 2D rotating frame nuclear Overhauser exp (ROE/NOE) with presat of CD3OH, best for CD3OD
```

Figure 19 *- Zoom of available experiments*

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

10.) Edit Parameters

Experiments can be modified (see "="icon, **Figure 20**). Please note that 2D parameters will still appear but are not applicable to 1D experiments so leave alone as unexpected results may occur.

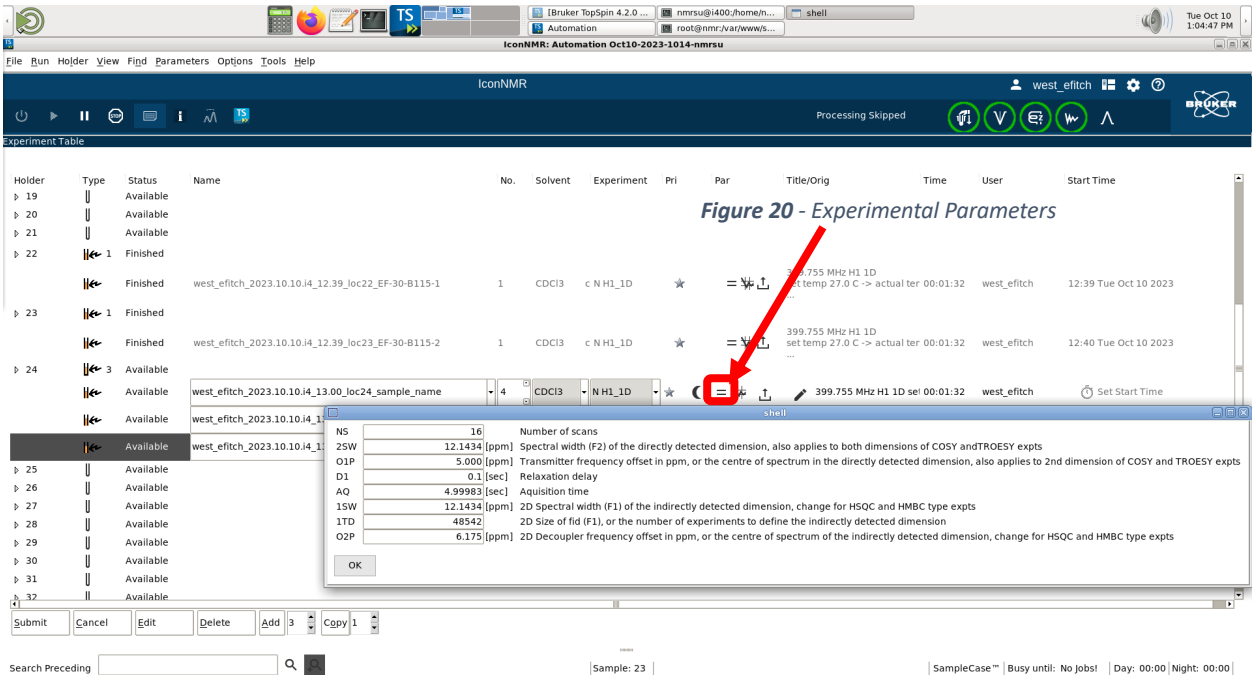


Figure 20 - Experimental Parameters

11.) Select Day or Night Queue

Select the queue based on the length of experiment time required (**Figure 21**).

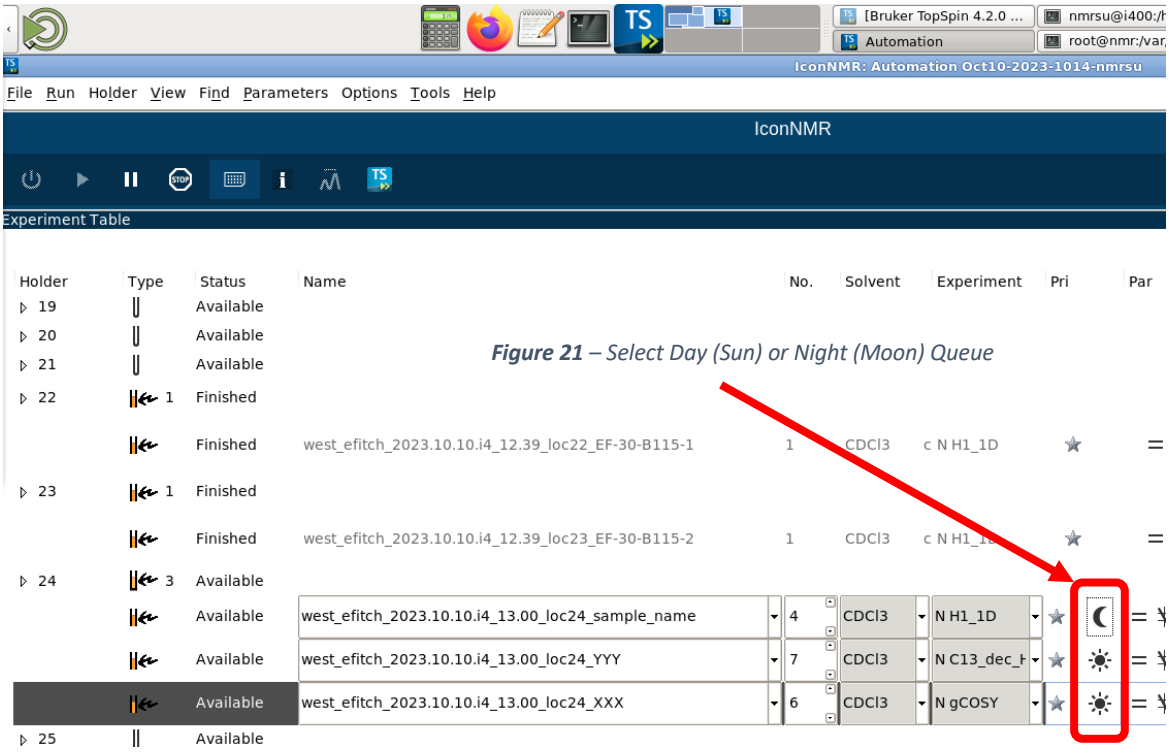


Figure 21 – Select Day (Sun) or Night (Moon) Queue

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

12.) Everything ready for submission

Figure 22 below shows a sample ready for submission to the study queue. The sample position and experiments are chosen, named, and ready to go.

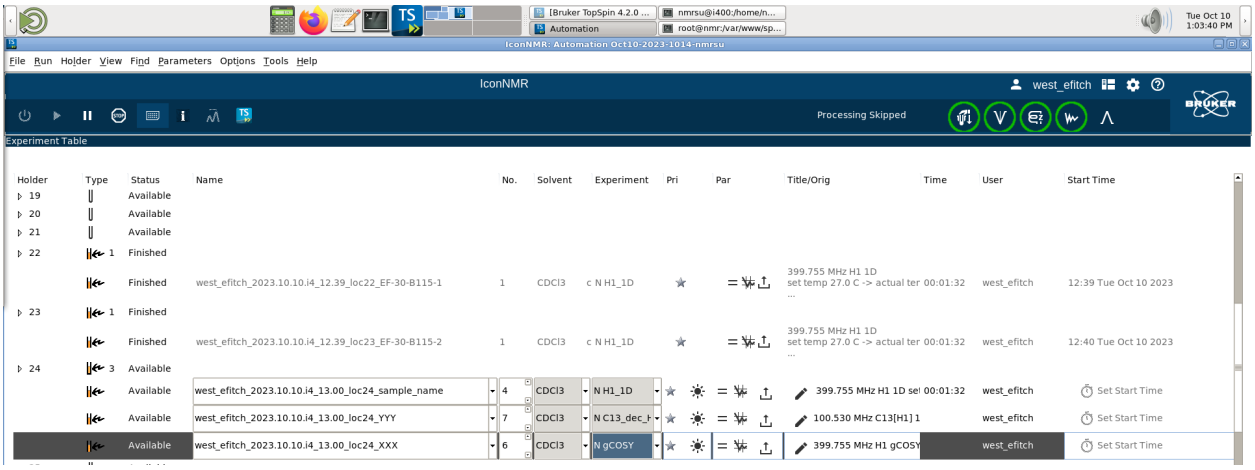


Figure 22 – Ready for submission to queue

13.) Submit Experiments

Once you've made all your selections, click the **Submit button** to finalize (**Figure 23**). The figure below shows Sample#1 successfully in the queue after hitting the Submit button (red box bottom).

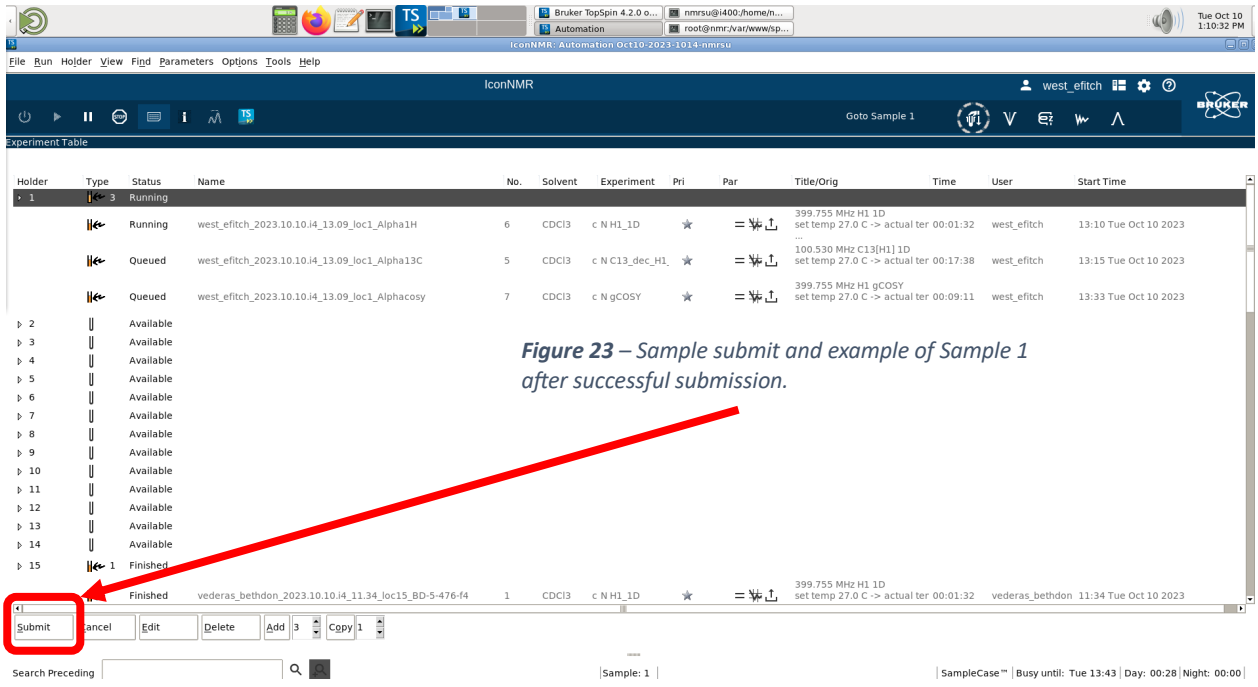


Figure 23 – Sample submit and example of Sample 1 after successful submission.

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

14.) Do not forget to "Change User" (*i.e.* Log Out) – You are Done!

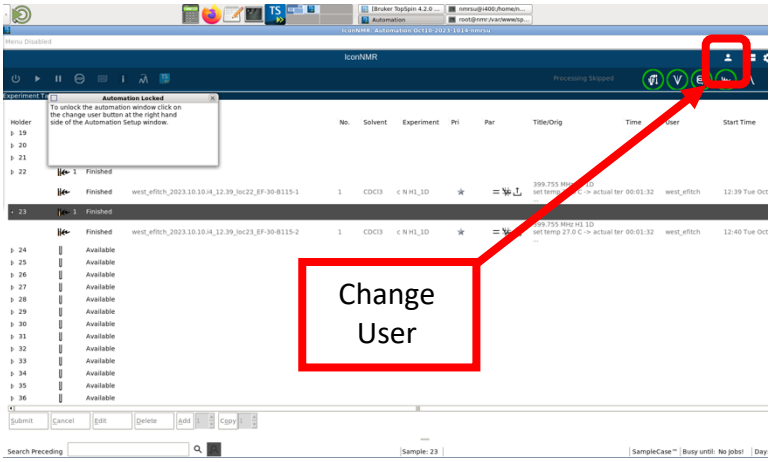


Figure 24 *– The last part of submission is to logout using the Change User Icon.*

Remember that last thing you need to do is log out using the "Change User" (**Figure 24**)button so the next person can proceed.

15.) How to retrieve a blocked sample from the carousel.

Figure 25 *– The tube prevents access when a sample is in the magnet. Leave that spot alone.*

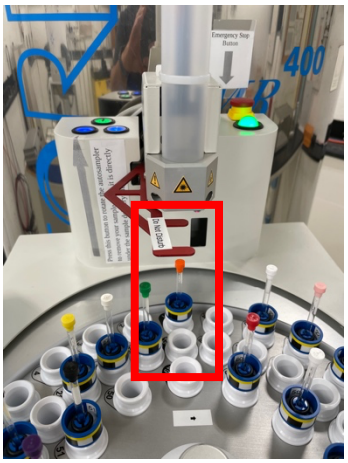


Figure 27 *– When done your sample will be returned to the carousel but may be blocked (red box in center above).*

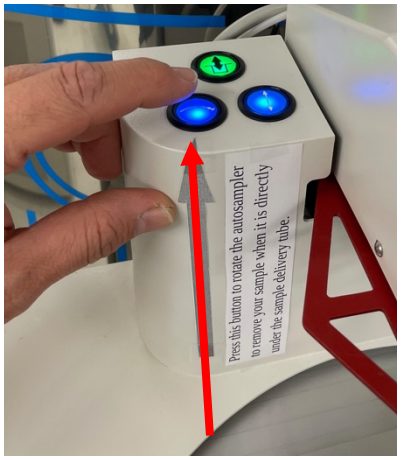
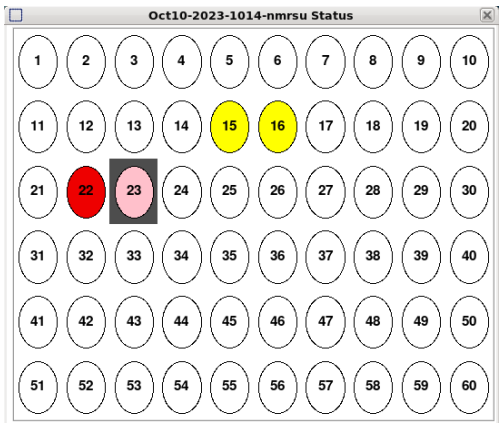


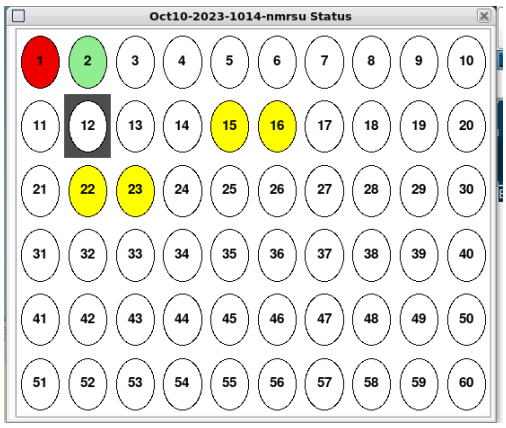
Figure 26 *– To move the carousel position, press the blue tray advance buitton (i.e.button with left-right arrows) as in the picture above.*

If you can not retrieve you sample under the robot (**Figure 27**), use the move sample button (**Figure 26**).

Ryan McKay
Tel: 780-492-9950
ryan.mckay@ualberta.ca

Appendix – Colours of the Queue





Queue Window

Red = Sample Running – Do not use
Green = Submission in Progress (not complete)
Yellow = Complete and safe to remove from queue/use spinner
Pink = XXX

UNIVERSITY OF ALBERTA
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Ryan McKay
Tel: 780-492-9950
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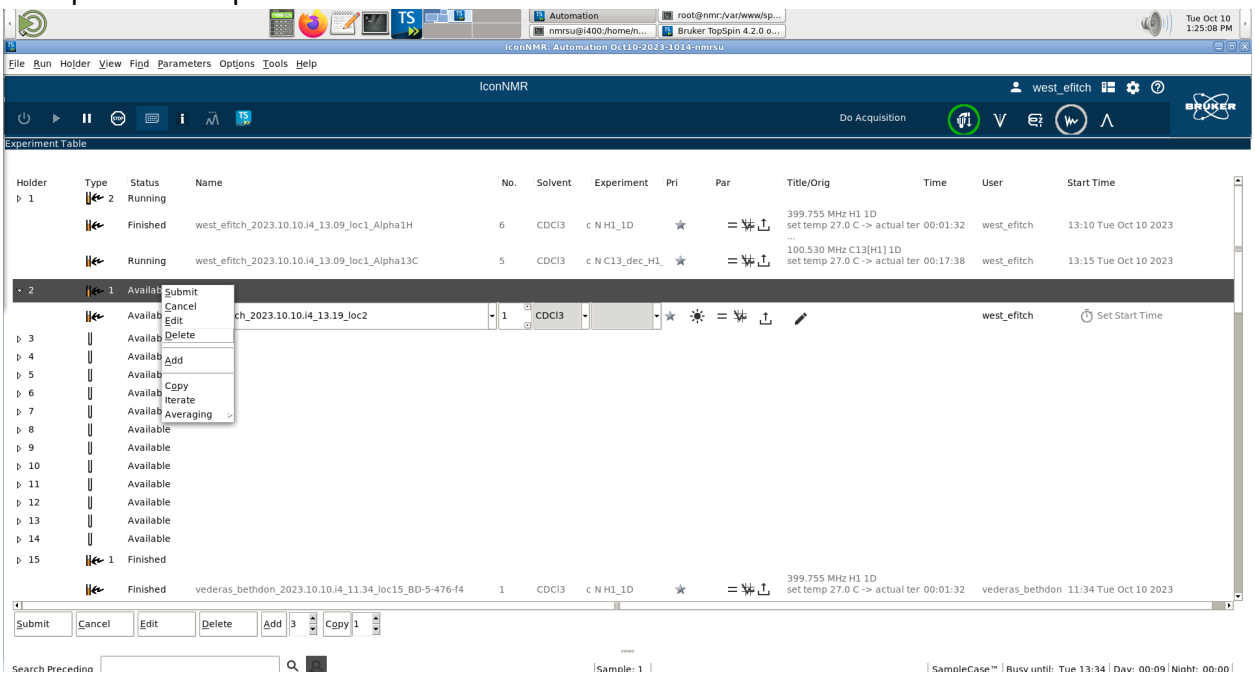
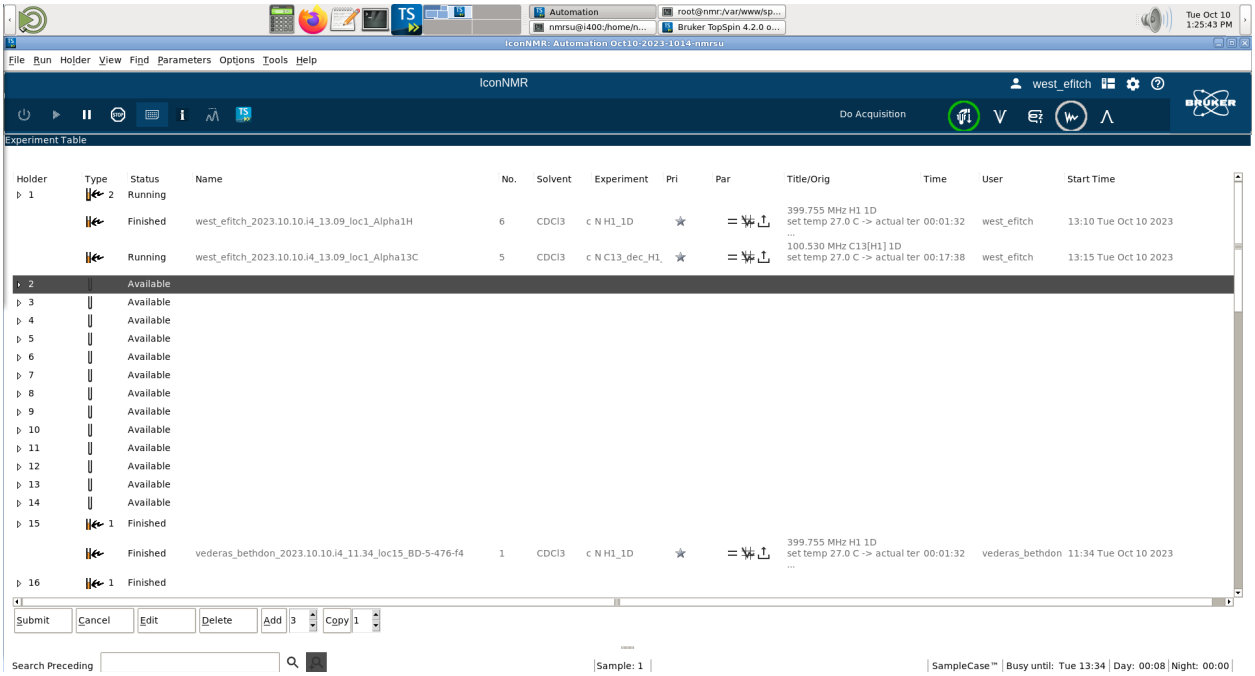
Appendix – Another Example of Freeing up Slot for Complete Sample

Example Freeing up used slot – control-click or right-click the completed Sample number, example below is position #2. Select "Delete"



Successfully Removing Completed Sample #2 to Free up Spinner