



Quick guide
400 MHz AV 4 Neo NMR “Mars”
Version 3.0

Institute of Organic Chemistry, University of Innsbruck

Christoph Kreutz, Feb 2021

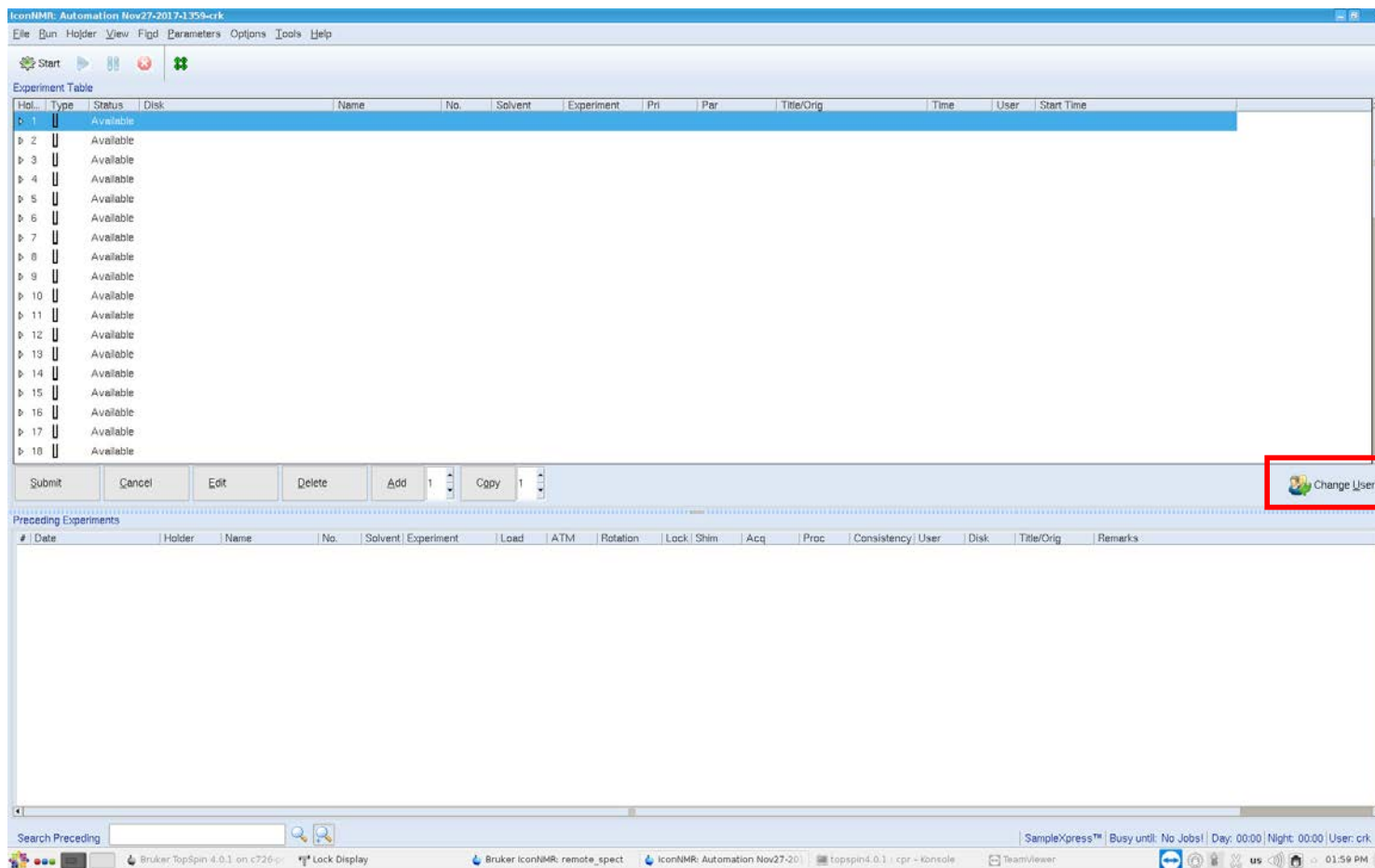
General comments/rules

- Keep the outer door (with the electronic lock) closed at all time.
- No food or drinks in the NMR rooms.
- **No lab coats and gloves in the NMR room.**
- Users should report any accident/incident immediately! Call 57725 or 57730 (e.g. a sample tube breaks inside the magnet or tube is not ejected anymore).
- Do not play around with the other spectrometers (Merkur 300 MHz and Venus 500 MHz) unless you are a trained user!
- Do not use USB sticks to transfer data – NMR data is accessible via the the network – ask for login and password!
- Liquid nitrogen refilling is scheduled every Thursday afternoon (approx. 30 minutes). During the refilling procedure the shim is negatively affected!
- 400 MHz NMR user email list: nmr-orgchem@lists.uibk.ac.at - can be used to discuss issues regarding the 400 MHz NMR.
- **In case of quenching of magnet – leave the room IMMEDIATELY!!**

General comments regarding NMR samples/sample handling rules

- **The sample should be fully dissolved in the deuterated NMR solvent.** Particles, precipitates etc. will negatively affect the shimming procedure.
- **Use approximately 600 µL deuterated NMR solvent!** If all users stick to this volume, the better and faster the shimming procedure.
- **Only use NMR tubes in good condition (no scratches, not broken, etc.).** NMR tubes can be recycled, do not use paramagnetic cleaning agents (e.g. Chromic Acid). As a last resort use HNO₃ to remove persistent impurities, afterwards wash thoroughly with water then acetone. Drying NMR tubes at elevated temperature can reshape tubes and ruin precision NMR tubes. In the final step wash the NMR tube with acetone and dry the tubes in vacuum.
- **All NMR samples must be properly labeled.** It is advisable to directly label the tube!
- **NMR tubes must be cleaned with green towels** from dust and fingerprints before being inserted into the magnet. **Adjust the sample position with the depth gauge (marked with 400 MHz).**
- **Daytime 8.00 to 20.00, nighttime 20.00 to 8.00 and all Saturday and Sunday.** Nighttime experiments shorter than 25 minutes are reshuffled to daytime queue during IDLE time.
- Maximum duration of a **day time experiment** is **20 minutes**. Number of scans of nighttime experiments can be varied as needed. Nighttime NMR experiments with an **acquisition time > 4h** should **be submitted on Friday** to make use of the extended nighttime queue during the weekend.
- Samples should be removed **ASAP(!)** from the SampleXpress after the experiments are finished. Finished samples are removed every Monday morning (9.30) and are put into a glass beaker. Unlabeled samples will be disposed promptly, labeled samples are kept for 3 weeks, then disposed.
- **PLEASE DO NOT USE special characters (such as /&%\$; etc.) and spaces for sample names! – Linux does not like such characters.**

Getting started

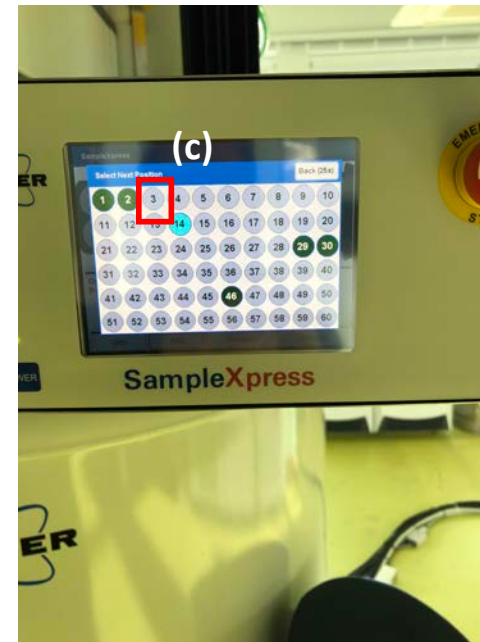
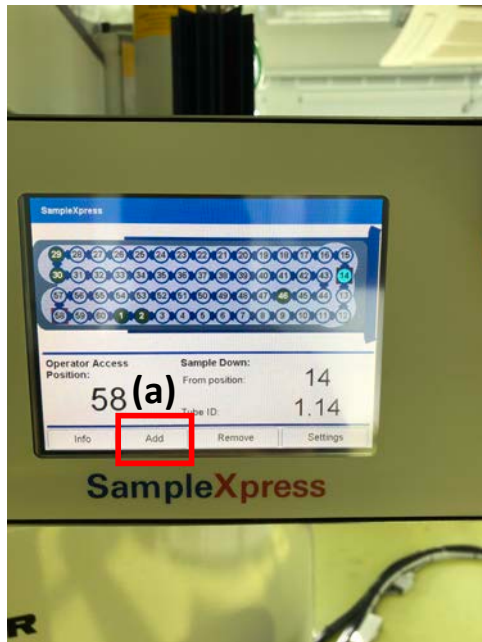


(1) Left-click on change user, in next menu pick your login and left-click OK; enter your password.

(1)

Adding your sample

- (1) Position the sample in a spinner using the depth gauge.
- (2) Use the touchscreen at the SampleXpress to add your sample at any free position to the queue:
 - (a) Add;
 - (b) Go to position or Go to free;
 - (c) if go to position select next free position (here 3); wait until position is reached, then add sample.



Setting up experiments

PLEASE DO NOT USE special characters (such as /&%\$;) and spaces for sample names! No DOTS at the end of name!!!- Linux does not like such characters!!!!

The screenshot shows the Bruker IconNMR Automation software interface. The main window is titled "IconNMR: Automation Nov27-2017-1359-crk". The interface includes a menu bar (File, Run, Holder, View, Find, Parameters, Options, Tools, Help) and a toolbar with icons for Start, Stop, and Refresh. The "Experiment Table" is the central component, with columns for Name, No., Solvent, Experiment, Pri, Par, Title/Orig, Time, User, and Start Time. A red box labeled (3) highlights the "Holder" column for the first row. A red box labeled (4) highlights the "Title/Orig" column for the first row, which contains the text "Testsample in CDCl3 10 mg recrystallized". A red box labeled (5) highlights a small blue button with a plus sign in the "Par" column for the first row. A red box labeled (6) highlights the "Add" button in the toolbar, which has a value of "1" next to it. A red box labeled (7) highlights the "Submit" button in the toolbar. A red box labeled (8) highlights the "Cancel", "Edit", and "Delete" buttons in the toolbar. Below the "Experiment Table" is a "Preceding Experiments" table with columns for #, Date, Holder, Name, No., Solvent, Experiment, Load, ATM, Rotation, Lock, Shim, Acq, Proc, Consistency, User, Disk, Title/Orig, and Remarks. The Windows taskbar at the bottom shows the system tray with the time 02:03 PM and the user crk.

(3) Double-left click on the holder position where you just placed your sample.

(4) A row will appear in which you can enter your sample details:
Data directory (**NO editing necessary**), Name, No. (**NO editing necessary**), Solvent (choose from pull down menu), Experiment (choose from pull down menu), Title/Originator (add sample information if desired).

(5) Some parameters can be modified by clicking on this button.

(6) Change this value to the desired number of additional experiments and left click **Add**.

(7) Once all experiments are set-up submit them by clicking **Submit**.

(8) Experiments can be removed from the queue by **Cancel**, and can then be **Edit(ed)**. **Delete** erases the experiment.

A daytime characterization

The screenshot shows the Bruker IconNMR Automation software interface. The main window displays an "Experiment Table" with the following data:

Exp. No.	Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	Consistency	User	Disk	Title/Orig	Remarks	Start Time	
1	Available	/home/nmrsu/spectra_ck	Testsample	1	CDCl3	chl N 1H_daytime	★	☑	☑	☑	☑	☑	☑	☑	☑	☑	Testsample in CDCl3 10 mg recrystallized	crk	00:01:12
2	Available	/home/nmrsu/spectra_ck	Testsample	2	CDCl3	chl N COSY_daytime	★	☑	☑	☑	☑	☑	☑	☑	☑	☑	Testsample in CDCl3 10 mg recrystallized	crk	00:04:46
3	Available	/home/nmrsu/spectra_ck	Testsample	3	CDCl3	chl N HSQC_daytime	★	☑	☑	☑	☑	☑	☑	☑	☑	☑	Testsample in CDCl3 10 mg recrystallized	crk	00:04:50
4	Available	/home/nmrsu/spectra_ck	Testsample	4	CDCl3	chl N 13C_daytime	★	☑	☑	☑	☑	☑	☑	☑	☑	☑	Testsample in CDCl3 10 mg recrystallized	crk	00:08:55

The interface also shows a "Preceding Experiments" table at the bottom, which is currently empty. The taskbar at the bottom indicates the system is running on a Windows machine with the time 02:04 PM.

This setup will run a ^1H 1D, ^1H -COSY, a ^1H - ^{13}C -HSQC and a 1D - ^{13}C in **19 minutes**.

This experiment setup is recommended for 30 mM+ samples – e.g. 10 mg sample (Mol.wt. 600) in 600 μl deuterated solvent.

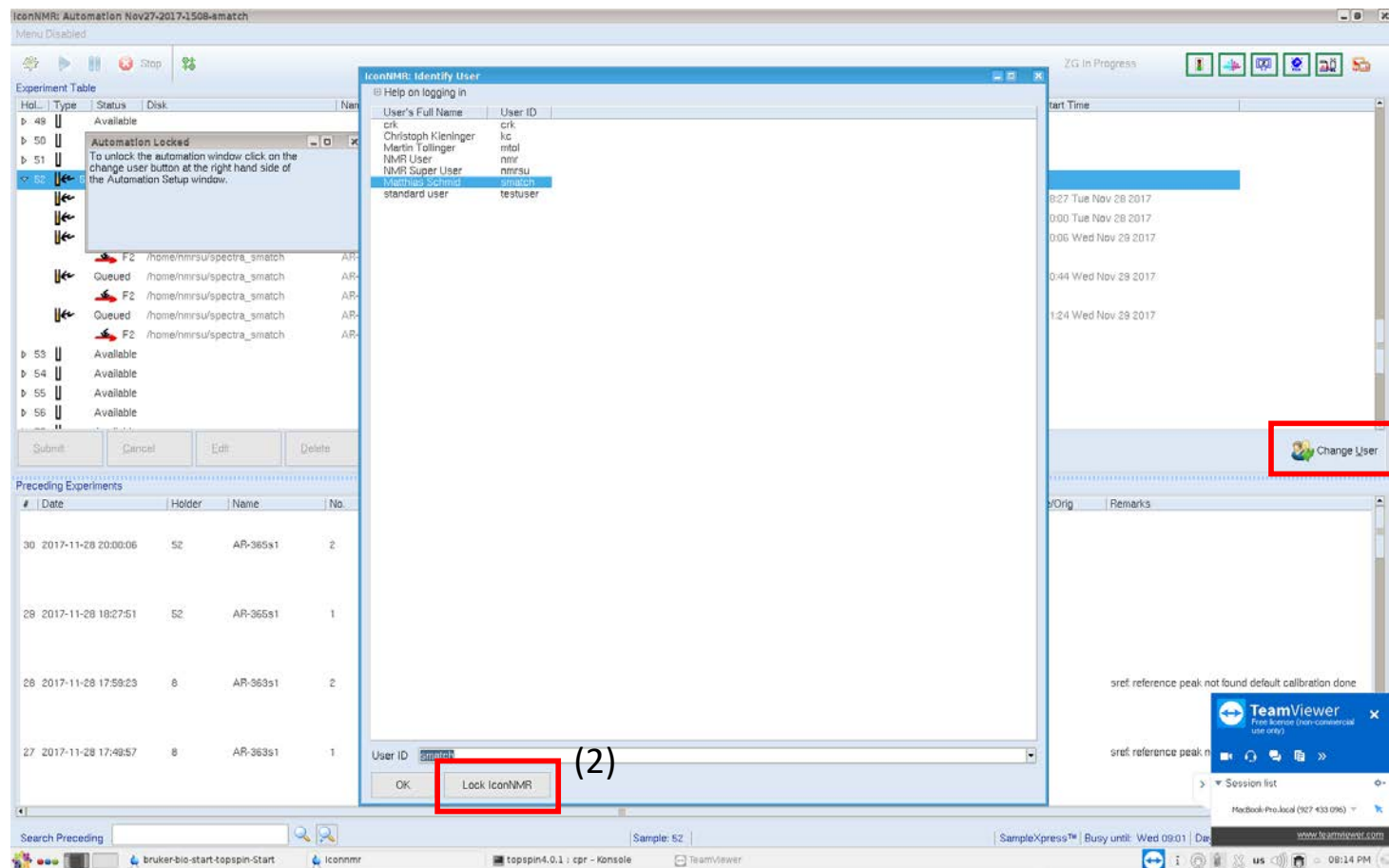
For lower sample amounts use the night time experiments or use high field NMR instrumentation.

List of the available experiments

Daytime	Experiment time	Nighttime	Experiment time
1H_daytime (8 scans)	1min 12s	1H_nighttime_2 (128 scans)	13min 15s
1H_daytime_2 (128 scans)	13min 15s	COSY_nighttime (8 scans/increment)	23min 32s
19F_daytime (32 scans)*	57s	HSQC_nighttime (16 scans/increment)	52min 54s
COSY_daytime (2 scans/increment)	4min 46s	HMBC_nighttime (32 scans/increment)	2h 9min 54s
HSQC_daytime (2 scans/increment)	5min 36s	13C_nighttime (2048 scans)	1h 57min 31s
HSQC_daytime_20min (8 scans/increment)	19min 46s	NOESY_nighttime (8 scans/increment)	1h 7min 56s
HMBC_daytime_20min (4 scans/increment)	19min 48s	ROESY_nighttime (8 scans/increment)	1h 10min 55s
HSQC_NUS (2 scans/increment)	2min 28s	TOCSY_nighttime (16 scans/increment)	1h 29min 31s
13C_daytime (256 scans)	14min 53s		
195Pt_1Hdec (512 scans)	19min 5s		
31P_1Hdec (128 scans)	4min 6s		

* ¹⁹F experiment: adjust center of spectrum (o1p) and spectral width (1sw) to your sample requirements.

Logging out after experiment setup



(1) Left-click on **Change User**

(2) Left-click **Lock IconNMR**

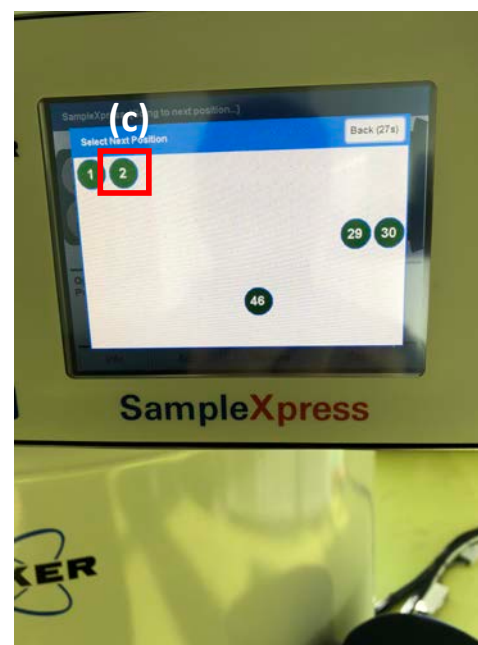
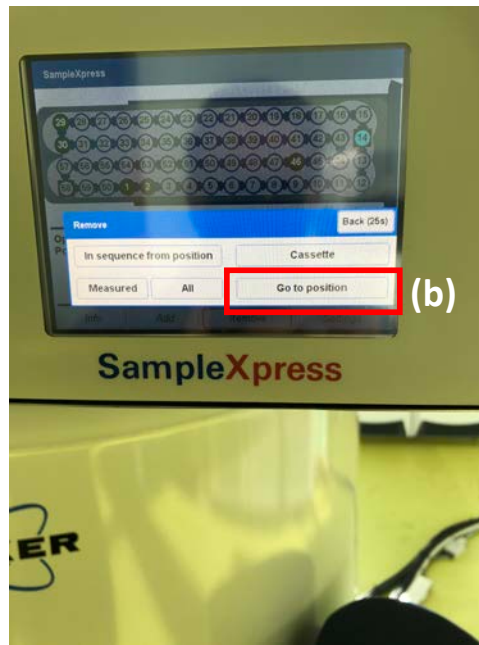
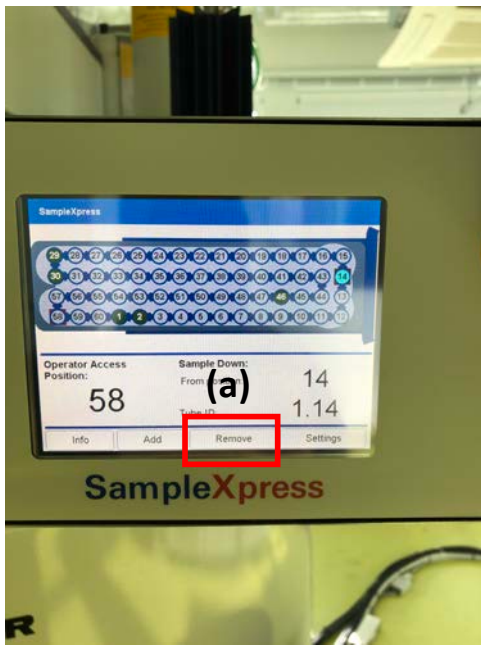
(1)

(2)

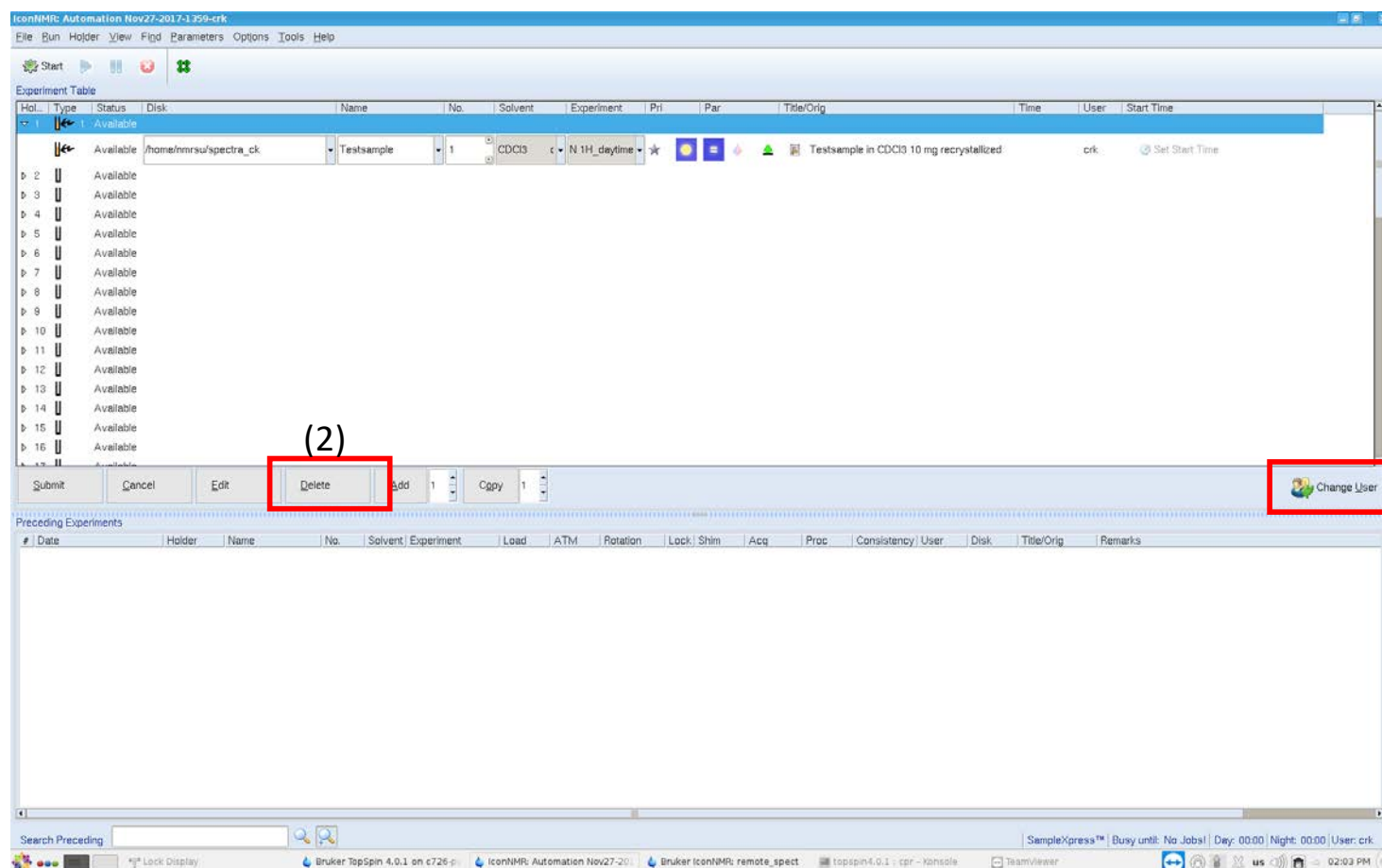
Removing the sample

Use the touchscreen at the SampleXpress to remove your sample from the queue:

(a) Remove; **(b) Go to position;** **(c) Pick your sample (e.g. 2);** wait until position is reached, then remove sample.



Removing the experiments from queue



- (1) Login by clicking **Change User**
- (2) Select your holder position and then click **Delete**

This will make the holder position free for other users.