Av400 User Guide – IconNMR Walkup Automation

updated: 2 June 2015 (cgf)

I. Sample Preparation:

A. Economy tubes are fire polished at the top; other tubes have a sharp edge that will damage the cap. *Economy tubes are recommended for the 400.* Currently the facility staff have no specific recommendations about suppliers for tubes (see the table below), although Norell may have some advantages for broader use (e.g., for VT).

B. SampleJet-specific caps ***must always*** be used on the 400. Bruker's caps can be used a "few" (1-3) times. Norell's NorLoc caps appear to be superior, sealing better and remaining useful after many more uses (~10). Please use only the red NorLoc caps. We tried other colors and they do not hold up to organic solvent which will cause problems in the automation queues.

> ### *Tubes and Caps for the SampleJet Robot*
>
> 7" or 4" tubes & SampleJet caps only!!
>
> - Any cap that does not provide a firm fit should be immediately discarded!
> - Throw away any tube that is cracked, scratched, or chipped at the top.
> - Never use parafilm, tape or paper adhesive on tubes or caps.
> - Do not write on 4" tubes; the ink might transfer onto and damage the (very expensive) spinner. Mark the cap if required to track your sample.

C. Improperly dried tubes can seriously damage NMR probes (expensive to repair!). Wilmad's NMR-010 technical note (at wilmad.com) describes how to correctly clean and dry tubes:

> **Drying tubes** at elevated temperatures can reshape and ruin precision NMR tubes. If you dry tubes in an oven, WILMAD recommends placing tubes on a perfectly flat tray at 125°C for only 30-45 minutes. Better is the use of a vacuum oven that will remove water at lower temperatures

Suppliers for Tubes and SampleJet Caps:

Vendor[1]	part no.	quant.	price	comments
Bruker 7" 5mm tubes[2]	Z107374	100	$179.40	w SJ caps[3]
Bruker 7" 5mm tubes[2]	Z114995	5	$ 10.30	w SJ caps[3]
Bruker SampleJet caps[3,4]	Z105683	6×100 1×100	$ 32.90 / 100 $ 50.40 / 100	
Wilmad 7" 5mm tubes[2]	WG-1000-7	1×100	$100.65	
Norell 7" 5mm tubes	SVCP-5-178-96PK	96	$185.12	w SJ caps[3]

| Norell NORLOC caps[5]

PLEASE ORDER ONLY RED CAPS — OTHER ONES ARE NOT STABLE FOR USE WITH ORGANIC SOLVENTS | SVCP-SAMPLEVALULT-5-96PK | 4×96

1×96 | $ 42.22 / 96

$ 52.00 / 96 | |

[1]Bruker and Wilmad tubes are rated to 600 MHz, Norell to 700 MHz
[2]Bruker and Wilmad do not recommend these tubes for VT use.
[3]Includes closed-port caps.   [4]Wilmad also sells SampleJet caps from Bruker.
[5]Norell Sample Vault tubes are rated safe for cold storage and VT use.

D. Use $\geq$ 450 µl of solvent: 600 µl of solvent is recommended. Using less than 450 µl will prevent proper shimming, resulting in significant signal-to-noise loss.

E. Sealed, J-Young, Shigemi, 3mm tubes, etc., cannot be used with the SampleJet on the 400.

II. SampleJet Robot:

The robot displays sample status information in the blue LCD panel. Manual mode is not used for automation. The browser panel (http://samplejet on the spectrometer host computer; just the bottom portion shown below) also displays sample status information and operating mode.

| manual mode **not for automation**	5 mm shuttle mode **no sample present**	5mm shuttle mode **sample changing**	5mm shuttle mode **sample in magnet**
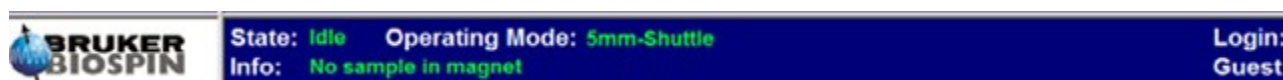
III. Multi-User Automation with Day and Night Queues [see photos following pages]

A. IconNMR should be on-screen, with the Change User panel up. Use your chemistry *username* and *password* to login.

B. **Add** experiments. Use the blue icon to change primary parameters. Use **Parameters → Edit all Acquisition Parameters** to change *any* parameter (careful! don't change it if you aren't certain about what you are doing).

C. Change any parameter (even d1 from 2 to 2.0) to get an estimate of the length of the experiment.

D. Current limitations are (which may change):

maximum experiment length during DayQueue: 15 min
maximum combined experiment time DayQueue: 40 min
maximum length of NightQueue job that will run during idle Daytime: 30 min
maximum combined experiments allowed for NightQueue: 2 hr
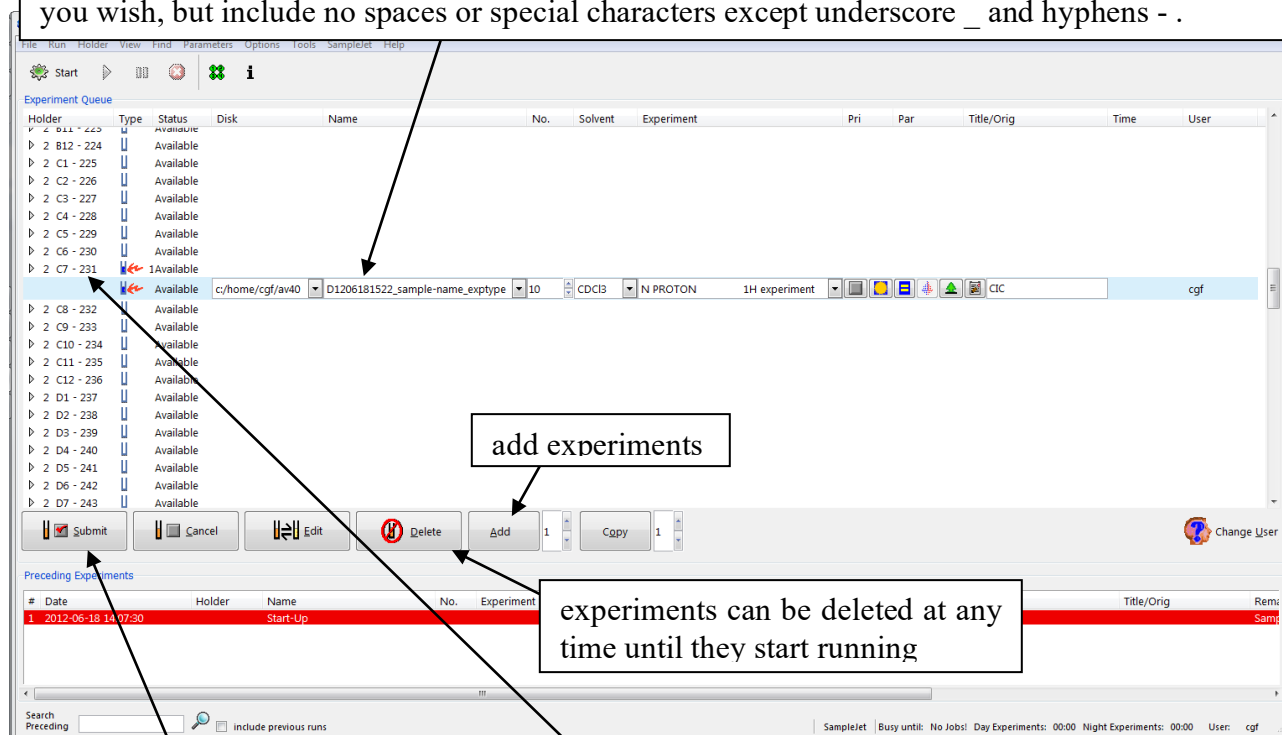
E. Common Gotcha's and Hints:
   → click on the holder row in a multiexp setup before pressing Submit:
      if you click Submit while on a single exp, it will submit only that experiment
   → select your group on the first experiment before adding more
   → to transfer data to your computer, use an sFTP program, such as FileZilla or WinSCP, to connect to castor.chem.wisc.edu using your chemistry username and password

login and out of IconNMR

queue and user info
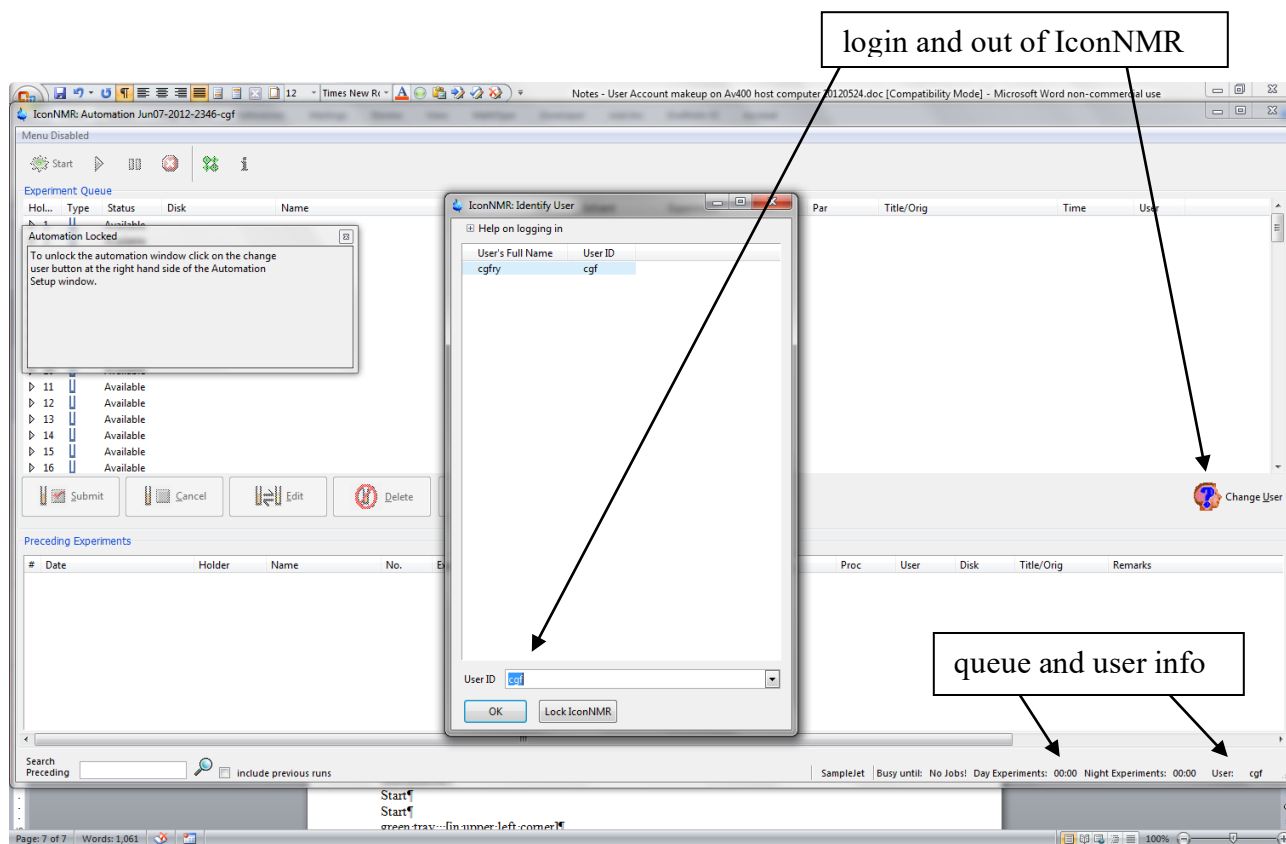
Click on holder position to quickly find the correct row in Experiment Queue. Use any open position in the outer "Open Shop" ring (7" or 4" tubes) or racks (4" tubes only).
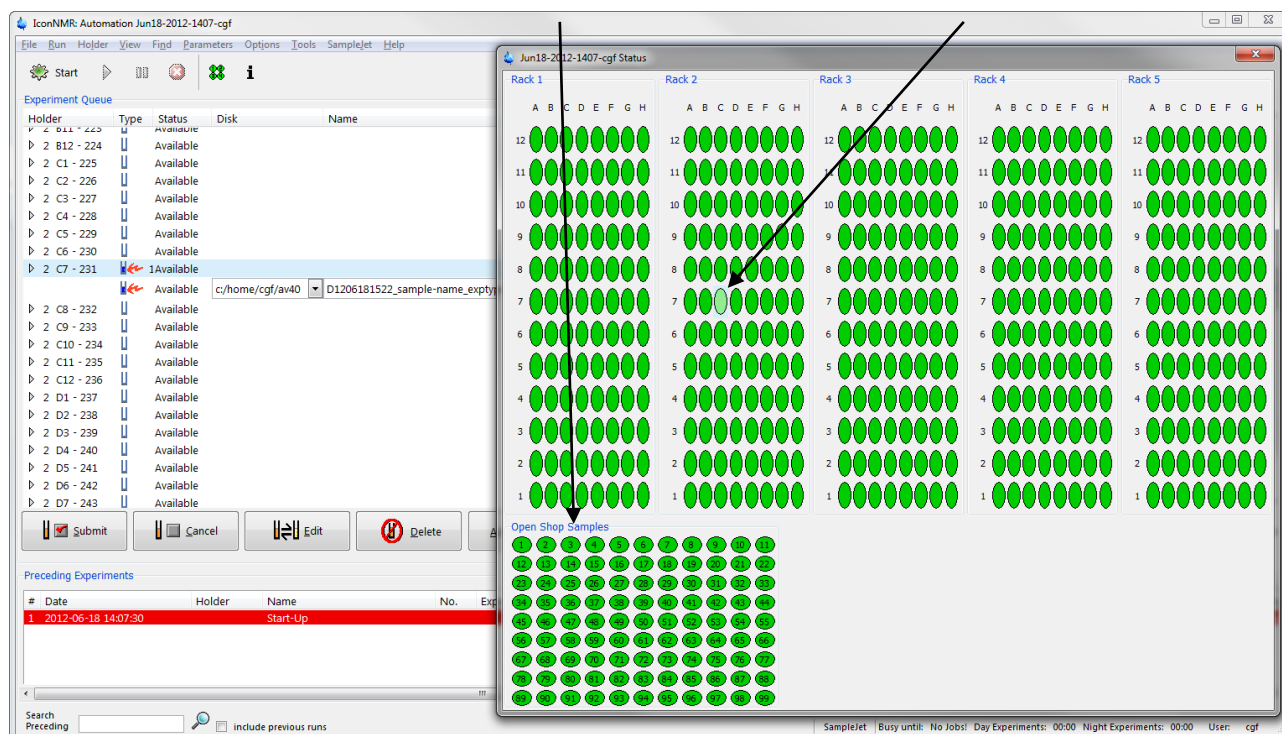
Leave the initial name string as is: it insures the folder is unique. It is formed from the date&time of creation: DYYMMDDHHMM == year/month/day/hour/minute. Add details as you wish, but include no spaces or special characters except underscore _ and hyphens - .

add experiments

experiments can be deleted at any time until they start running

Submissions will occur only on the experiment you have highlighted. To submit a set of experiments on a single sample, highlight the holder position, then press submit.

Solvent Suppression

A. *Initial Setup:*

1. Always start by acquiring a one-scan 1H spectrum.

   It is not required, but best to run solvent suppression experiments on-resonance to the solvent peak. If you believe this is not optimal, find cgfry for further discussion.

2. Put the solvent peak on-resonance by clicking ↯, then choose O1. Retake the one-scan 1H spectrum to verify that the peak is in the center of the spectrum. [Another, possibly simpler, method for going on resonance is to enter the shift of the solvent peak in the parameter **o1p**. This will not be as accurate, since it won't account for solution/temperature/etc variations in chemical shift.]

3. Copy parameter into a new expno using: **iexpno**

B. *Presaturation:*

1. In the new expno, run **ased** and change the first parameter PULPROG to **zgpr** .

2. The critical new parameter is **PLW9**, which will perform a low-power cw pulse on-resonance. You can raise the power of this parameter to decrease the intensity of the residual signal, but setting it too high will damage the probe!

   PLW9 ≥ 25 –dBW (i.e., the value in the 2nd box on the ased screen should ≥ 25)

3. Take new data with **ns = 4×i** and **ds = 2×j** (i,j = 1,2,4,8,…). Run an **rga** prior to doing **zg** .

C. noesygppr1d *Presaturation:*

1. Do the same steps above, but read in the Bruker parameter set **watersup** . I.e., do

   rpar WATERSUP all

2. Change **o1** or **o1p** to match the value found in A.2.

3. Do an **rga**. Take data with **ns = 8×i** and **ds = 4×j** (i,j = 1,2,4,8,…).

4. PLW9 again might be smaller than optimal. Same conditions apply as in B.2.