

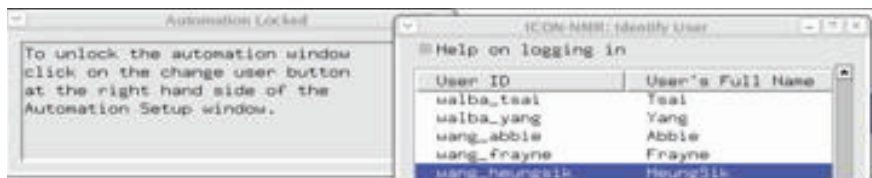



Bare Bones Guide for the Bruker AV-III 400 MHz NMR Spectrometer, using ICON NMR® in Automation

Revised 5/23/2012

- **Before you Start:** The 400 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 might 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 is STRONGLY recommended.**

1. First Log-In to your User Account...
If you see the “Identify User” screen, just select your user account, and **click [OK]**.



Otherwise, click the “Change User” icon: , then select your User Account, and **click [OK]**. You should notice that you are Logged-In at the lower-right corner of the window: User: **NMR_rshoe**. **If you don't login** your data will be saved in the wrong directory, and your NMR time will be charged to the wrong group... this would be bad.


2. **Note the next available sample holder number:** In the picture below, the sample in position #12 has been completed, and the next available holder is Holder #13. You will be putting your sample in holder #13. (**Red**=sample completed, **Yellow**=experiment submitted, **Green**=experiment in progress)

Holder	Type	Status	Name	No.	Sol...	Expe...	Par	Title / Orig	Pri	Time	User
9	U	●●●									
10	U	●●●									
11	U	●●●	ysh-I-096-crude	10	CDC13	proton	zhang	ysh-I-096-crude	00:01:14		zhang_huang
12	U	●●●	HHS-2-066-Product	10	CDC13	proton	wang	HHS-2-066-Product	00:01:14		wang_heungsik
13	U	●●●	HHS-2-069-crude	10	CDC13	proton	wang	HHS-2-069-crude	00:01:14		wang_heungsik
14	U	●●●									
15	U	●●●									
16	U	●●●									

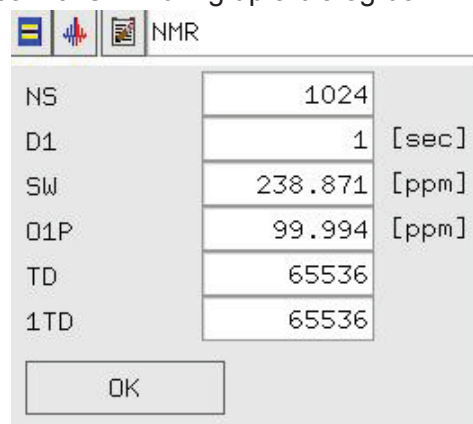
3. Take one of the unused Blue sample spinners from the rack, and insert your sample as follows:
 - a. Wipe-off your NMR tube, so that it is CLEAN (use isopropanol if necessary)
 - b. Insert your sample tube in the spinner, and wipe it again (to be sure it is clean).
 - c. Set the **sample depth** using the Clear, Bruker depth gauge. If your sample is *shorter* than normal, use the black bars on the clear gauge to center the sample.
4. **Carefully** place your sample in the correct holder position, according to the number noted above (in this example it would be **holder #13**). You are now ready to setup your experiment in Holder #13. **NOTE: ONLY add/remove samples when the “Dome” status light is GREEN!!**
5. **Double-Click** in the row of Holder #13 (i.e. on the box w/ 3-colored circles). The row will “open up”, and you can enter values into the boxes under the individual headings:
 - a. Type your filename in the Box under “Name”
 - b. **ONLY** letters, numbers, dashes(-), or underscores (_). **NO SPACES** in filenames, and **NO SYMBOLS!**

Holder	Type	Status	Name	No.	Sol...	Expe...	Par
9	U	●●●					
10	U	●●●					
11	U	●●●	ysh-I-096-crude	10	CDC13	proton	
12	U	●●●	HHS-2-066-Product	10	CDC13	proton	
13	U	●●●	HHS-2-069-crude	10	CDC13	proton	



- c. Click on the Solvent Box, and a pull-down menu will appear.. select your solvent from the pull-down Menu.
- i. Note: The “*Experiment Number*” (under “No.”) will be set to 10 by default. If you want to learn more about Experiment Numbers, you can ask for more information.


- d. You can change basic parameters by clicking on the  icon: this will bring up a dialog box in which you can change basic parameters:


- i. NS is the number of scans
 - ii. D1 is the relaxation delay
 - iii. SW is the spectral width in PPM
 - iv. O1P is the center of the spectrum
 - v. TD is the number of FID points acquired
 - vi. 1TD is the number of increments in 2D experiments.
- If you want to change a parameter, *click* in the box, and enter the new value.. then *click* [OK].

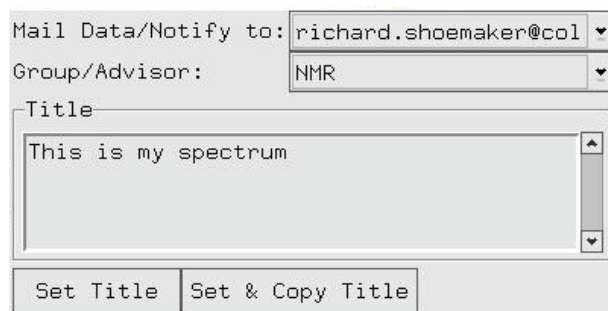


NMR	
NS	1024
D1	1 [sec]
SW	238.871 [ppm]
O1P	99.994 [ppm]
TD	65536
1TD	65536
OK	

(If you change any parameter from the default, the  icon will change to  .. this is normal)

- e. Click the  NMR box (your group will be displayed, instead of “NMR”), and you will see the dialog box for you to add a “Title” (very useful for MestReNova), and also for entering your Email address to have the raw data emailed to you as a .zip file attachment:

- i. You can *copy/paste* your filename into the title easily, using the *middle-mouse-button* (*scroll wheel, used as a button*).
 1. select the text in the “Name” box using the Left-Mouse Button
 2. Point in the “Title” box, and *click* the scroll-wheel button.
- ii. Click [Set Title] when everything is entered the way that you wish.
- iii.  (default) will be run during the day



Mail Data/Notify to: richard.shoemaker@col

Group/Advisor: NMR

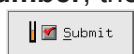
Title




This is my spectrum

Set Title Set & Copy Title


 will submit to the overnight queue... *click* to change between day/night queue.

- f. Double-Check that everything in the row is correct, confirm that your sample is properly placed in the correctly numbered holder in the sample-changer, **Select the row containing the Holder Number**, then *click* the



button. The status indicator will turn yellow  other experiments are already running, or green  if not  is running ahead of you. Once green, the instrument will insert your sample, tune the probe, lock on your solvent, shim using Topshim®, and proceed with your experiment.

- g. ADDING another experiment for the same sample: Select the row containing a previously

defined experiment, and *click* the  [Add] button. This will create a new row, using the same filename, and incrementing the Exp.# by one. You can type a new *filename* under the “Name” column, then tab over to Experiment. Select your next experiment from the list, then edit the Title-Box as desired. You may [Submit] the next experiment as soon as all of the information has been entered.

6. **If you have no more samples or experiments to submit, you should Log-Out by clicking on the**




icon. If you forget, you will be logged-out automatically after 10 minutes. Logging-out is a **good idea** because if someone uses your account, your lab account will pay for their NMR expt.

7. After your sample is done, **wait until the eject air turns off**, or until the next sample is inserted, before removing your sample from the rack. Removing your sample too soon may crash the system.

8. **INTERACTING WITH YOUR DATA AS IT IS ACQUIRING:** ICON-NMR is really a graphical “front-end” for the full Bruker NMR software package called “Topspin”. The two programs work together, allowing to interact with you spectra, either after acquisition, or during acquisition. This makes operating the spectrometer using the ICON interface extremely powerful and flexible. This tutorial will only mention a truly “bare-bones” description of Topspin commands because a full treatise on Topspin is beyond the scope of this guide. You can also use the instructions below to open/view/process a spectrum that has been completed earlier, by simply *double-clicking* on the appropriate row... see below:

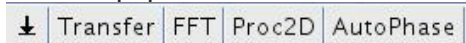







- a. Once the acquisition of your spectrum begins, you will see the  icon flashing... and the FID counter should be counting scans. You can *double-click* on the row in the “Preceding

Preceding Experiments											
#	Date	Holder	Name	No.	Experiment	Load	ATM	Rot...	Lock	Shim	Acq
9	2009-07-10 15:57:22	12	HHS-2-069-crude	10	proton	✓	✓	✓	✓	✓	✓
8	2009-07-10 15:52:38	11	HHS-2-066-Product	10	proton	✓	✓	✓	✓	✓	✓

Experiments” window, and you will be transferred to the Topspin program, and the dataset you *double-clicked* on will be automatically loaded. If it is a previously completed experiment, you can skip the [Transfer] step because the FID is already saved.

- b. On the Varian instruments, the accumulating data is *transferred* to the computer every “bs” scans automatically. With the Bruker, you have to tell the system to transfer the data by either typing “tr” in the Topspin command Line, or *clicking* the [Transfer] button:



- click* [Transfer] and wait until you see the confirmation message in the lower-left corner of the screen that the data has been transferred
 - click* [FFT] to process the data, then [AutoPhase] to phase the spectrum.
 - These buttons are simply shortcuts to commands you can type in the Topspin command-line near the bottom of the window:
 - [Transfer] = tr <Ret>.
 - [FFT] = efp <Ret>.
 - [AutoPhase] = apk <Ret>.
 - [Proc2D] = xfb <Ret>. (no Transfer is necessary for 2D expts.)
- c. Manipulating your spectrum is pretty easy to figure out, and is done using the icons on the toolbars at the top of the Topspin window:
- Hold down the Left Mouse Button, and drag to zoom-in.
 -  moves the baseline to the bottom of the display window.
 -  expands the spectrum, showing the full view.
 -  Hold the LMB over this icon, and drag the baseline up/down
 -  Hold the LMB over this icon to drag/slide side-to-side (if display is zoomed)
- d. If you are looking at data that is currently being acquired (i.e. a ^{13}C spectrum that was setup using a large number of scans (ns), and you want to stop it when it looks *good enough*... you can repeatedly [Transfer][FFT], and decide when to stop.
- To halt the acquisition, save the data, and have ICON-NMR continue to the next experiment, *click* the  button, or type **halt <Ret>**. This stops at the end of the current scan.
 - if you want to halt/save at a certain scan number, you can type **halt # <Ret>**. to stop after scan “#”. For example **halt 256 <Ret>**. will halt after scan 256, save the data, and continue with the next experiment in queue.