Bare Bones Guide for the Bruker AV-III 300 MHz NMR Spectrometer, using ICON NMR® in Automation

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- Before you Start: The 300 uses the BACS-60 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].
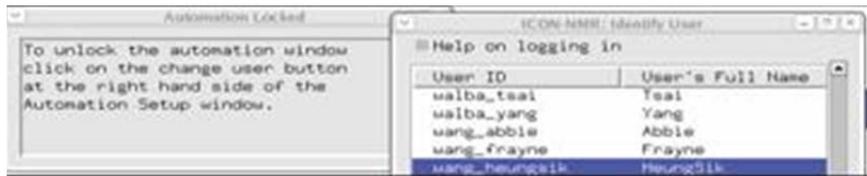
   Otherwise, click the "Change User" icon: , then select your User Account, and *click* [OK]. You should notice that you are Logged-In at the lower-right corner of the window: `User: NMR_rshoe`. **If you don't login** your data will be saved in the wrong directory, and your NMR time will be charged to 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*)

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.

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:
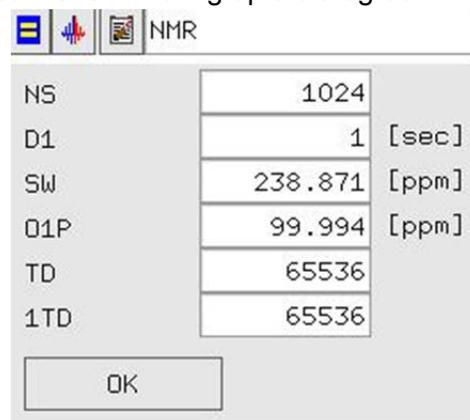   a. Type your filename in the Box under "Name"
   b. ONLY letters, numbers, dashes(-), or underscores (_). NO SPACES in filenames, and NO SYMBOLS!

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:

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

   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 if increments in 2D experiments. If you want to change a parameter, *click* in the box, and enter the new value.. then *click* [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:
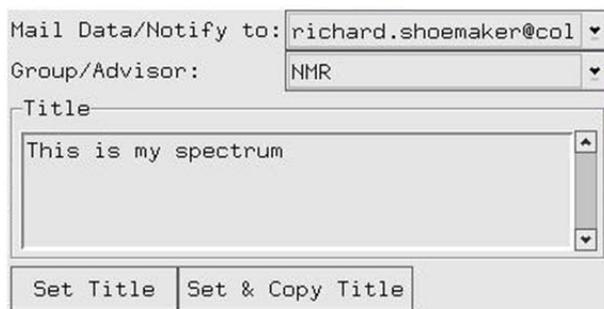
   Mail Data/Notify to: richard.shoemaker@col ▼
   Group/Advisor: NMR ▼
   ┌Title──────────────────┐
   │ This is my spectrum   ▲│
   │   ▼│
   └────────────────────────┘
   [Set Title][Set & Copy Title]

   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

   🌑 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
   [☑ Submit] 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] [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**
   [🔵 Change User] 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. <u>INTERACTING WITH YOUR DATA AS IT IS ACQUIRING</u>: 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 "Preceeding

   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:
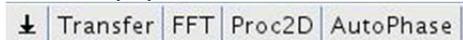
   i. *click* [Transfer] and wait until you see the confirmation message in the lower-left corner of the screen that the data has been transferred
   ii. *click* [FFT] to process the data, then [AutoPhase] to phase the spectrum.
      1. These buttons are simply shortcuts to commands you can type in the Topspin command-line near the bottom of the window:
         a. [Transfer] = tr <Ret>.
         b. [FFT] = efp <Ret>.
         c. [AutoPhase] = apk <Ret>.
         d. [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:
      i. Hold down the Left Mouse Button, and drag to zoom-in.
      ii. moves the baseline to the bottom of the display window.
      iii. expands the spectrum, showing the full view.
      iv. Hold the LMB over this icon, and drag the baseline up/down
      v. 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 repeateadly [Transfer][FFT], and decide when to stop.
      i. To halt the acquision, 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.
         1. 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.