

Chem 136 - Using the ARX400 Sample Changer

I. Sample Preparation:

- A. You must use a deuterated solvent.
- B. Label your sample with a Sharpie pen using a unique name (not "A" or "1"). Do not use tape or tags.
- C. Make sure the NMR tube is ≥ 16 cm. (below the cap) and that the column of liquid in the tube is ≥ 4 cm.
- D. Make sure the NMR tube is clean on the outside.

II. In the NMR lab (Room 1421 Molecular Science Building):

- A. If you have a heart pacemaker, do not enter the NMR lab! (See your T.A.) Do not take metal items (e.g. spatulas) into the area near the magnet. If you do not wish to demagnetize your credit cards or magnetize your mechanical watch, also do not take them near the magnet.
- B. Insert your sample into a spinner and set the depth with the aluminum depth gauge labeled "Maximum Depth".
- C. Place your sample in the sample changer rack in the position where the dummy sample sits. Move the dummy sample to the next rack position after your sample(s). Note the number(s) of the rack position(s) of your sample(s). This is probably the holder number that you will use.
- D. Unless you are following someone from Chem 136, select the button on the lower right labeled "Change User"
- E. Type in **chem136** as the user name. (Be sure to hit "Enter" on the keyboard. If you use the mouse to select OK, it will ignore what you have typed and select the highlighted user.) The password is **chem136**.
- F. Double-click the number that corresponds to your sample. (Check that the user name at the right edge is **chem136**. If not, move your sample to the next higher holder number that has no user name already and continue with this new number.) You will need to select a solvent and an experiment, and input a title.
 - 1) At the top of the title area, you **must** input your Account no. (**ct3136**) or the sample will not run. In the box below the Account no. box, you may input a title that will appear on the plot.
 - 2) If you would like to change the number of scans, select **Parameters** on the menu bar. Move the mouse down and select **User Specific Parameters/Commands**. In the list you will find **ns** which means number of scans. Use multiples of 8.
 - 3) If you would like to add another experiment to this sample setup, select **Add**. (The "wheel" to the right of the Add button indicates how many experiments will be added. Make sure it is set properly before you select Add.) You will then only need to select an experiment. You can change the title of additional experiments if you want.
- G. Once all the required information for the sample has been input, select the line with the sample holder number. Then select the "button" on the lower left labeled **Submit**. The sample will not run until it has been submitted!
 - 1) If the sample has not started running, you can make changes in the setup. Select the experiment to be changed. If only parameters are to be changed, do so as described above for changing **ns**.
 - 2) To change to a different solvent or experiment, select **Cancel**, then **Edit**. After making all the required changes, again select **Submit**.
- H. After you have finished the setup and submission for your sample(s), select the button on the lower right labeled "Change User". (This will assure that the next user does not accidentally set up spectra in your directory.)
- I. A plot of your spectrum will come out on the printer in Room 6096 Young Hall.
 - 1) If a proton spectrum has peaks downfield from 9.4 ppm, these peaks will be plotted on a 2nd page. Be sure to check the printer for possible 2nd pages for proton spectra.
 - 2) If your ^{13}C spectrum contains only the solvent peaks, you will obtain a blank spectrum. Consider making your sample more concentrated. If this is not a possibility, talk to your T.A. about running the ^{13}C spectrum some time other than during your class.