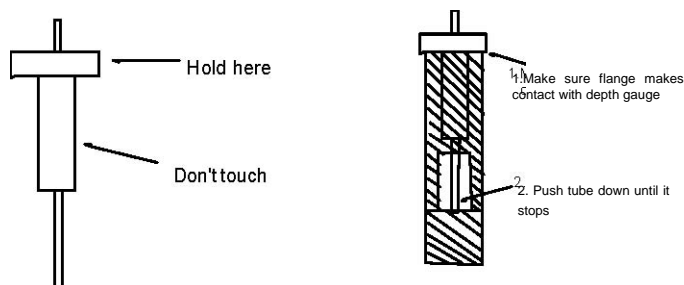


Organic Chemistry and NMR Walkup Automation and VNMR Software

Sample Preparation

1. Comments on NMR solvents: NMR solvents are expensive! Please treat them with care and do not contaminate them. Always put the lid back on the bottle and place in the desiccator. Deuterated solvents will undergo exchange if exposed to atmospheric moisture.
2. **Use only 8" tubes with the Robot!**
3. Use about 70 mg (or 70 μ L if liquid) of sample. Add 0.7 mL of solvent.
4. Make up the solution in a vial first and then transfer to the NMR tube by pipette or syringe.
Make sure that there are **no particles** in your tube. You may need to filter the solution prior to placing it in the NMR tube.
5. Cleaning NMR tubes: NMR tubes are also expensive. Please clean them out after each use and rinse with acetone. Do **not** place them in an oven to dry since this will deform them – let them air dry or, to speed things up, pull air into the bottom of the tube using narrow teflon tubing attached to a vacuum line.
6. Carefully insert the tube into the sample holder. *The spinners are not to be taken out of the NMR room.* Be sure to hold the spinner by the black portion near the top and not the lower surface of the spinner. The spinners cost \$400/piece so do not drop them.

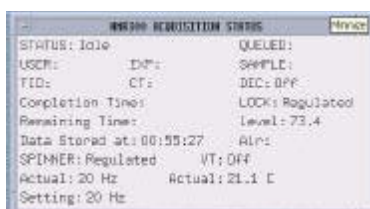


7. Place the sample holder into the depth gauge, making sure that the flange comes in contact with the depth gauge and then push the NMR tube down until it stops. Wipe the tube and the lower narrow part of the sample holder with a Kimwipe to remove fingerprints and particles. Place the sample and holder into the appropriate position in the changer.

Caution!!!! Before using WALKUP automation, be sure that the **corner sample position is empty**. The instrument may put the currently loaded sample into that position. If another sample is there, it will crush it!!!! Note the **emergency halt button** on the side of the table.

Using Walkup Automation

1. Be sure to log into the Account **Auto01** when using Walkup. See your TA or prof for the Password.
2. Click on the spectrum on the bottom of the screen to load VNMR.
3. Click on **[Walkup]** and **[Enter Sample]**.
4. Click **Sample Number**, **User Selection**, **Solvent Selection**, and all **Experiments** that you want to perform. Give the experiments a name under **Text**. The experiments will be performed in the order that you choose them. The data will be stored in **/export/home/username/vnmrsys/data** in directories titled **filename01.fid/**, **filename02.fid**, etc.
5. The approximate times for each experiment are given in brackets for each experiment. Choose **Add Sample** to start your experiments. Repeat Step 4 for the next sample. **Note: If you are the last one using the machine, run a “chaser sample” which should be one of the sealed solvent tubes marked in BLUE.** This sample is *d*-chloroform. Choose **H1** as your experiment for this sample. (Another sample with known solvent can be used also if the Blue one is missing.)
6. Watch the Acquisition Status Window for events that are taking place. The current sample in the magnet will be retrieved and placed in its former position or in the corner position by default.



After the your sample is loaded, the status window should show that the instrument is acquiring without spinning. This first acquisition is finding the **lock signal (z0)**. The second acquisition begins the **gradient shimming process**. It should repeat the shim process 2-3 times, after which time the sample will be spun and locked, followed by the actual experiments. If it can't find a good shim after 10 tries, the experiment will quit and go onto the next sample. Once the shimming is complete, the sample will spin and lock and then acquisition of the data will begin.

Retrieving your data using VNMR software.

To retrieve your spectrum, click **[Status]** and see if your completed data appears in the list. Highlight it and click **Retrieve Data**.

If it does not appear there, click on **[File]** on the Main Menu of VNMR. Click on **[Parent]** until you get to **/export/home** as noted in the workspace above. You will see a list of user names. Click on your **user name** and then **[Change]**. Click on **vnmrsys**, **[Change]**, then **data**, **[Change]**. A list of the fid directories will appear. Click on your sample directory, click **[Return]** and then **[Load]**. The fid is loaded. To process it, click on **[Autoprocess]**.

Retrieving your data for Magnetic Resonance Companion (MRC) in Computer Labs

In the Computer Lab, login and then load Filezilla. The IP address of the NMR computer is 172.20.152.56. The User Name is auto01. The password can be obtained from your TA. Locate [/export/home/username/vnmrsys/data](#) and find your sample directory. Transfer it C:/Temp and then open and process with Magnetic Resonance Companion. For use of MRC, see the NMR Homepage, <http://www.uaf.edu/chem/green/NMR.htm> Actually, Filezilla is currently pointed at the correct network location. Just hit Enter, then transfer the folder with your spectrum.

Display Mode

In order to interact with the spectrum, you must be in the display mode. To insure that you are in this mode, type **ds**. The interactive buttons on the menu will appear when you do this.

Expansions

The left and right mouse buttons control vertical cursors and can be used to select regions for expansion. Frame the region for expansion using left and then right mouse buttons, then select **[Expand]**.

Vertical Scaling

The middle mouse button controls the vertical scale of the spectrum. Click with the middle button above a peak to increase the scaling. Click below the peak to decrease the scaling.

Phasing

Usually autophase (type **aph**) command works sufficiently. However, in some cases you may want to manually phase. Select **[Phase]** and then click on a peak in the upfield (right) region of the spectrum. Click and drag up or down using Left (coarse) or Right (fine) buttons until the peaks appear phased. Now click a peak in the downfield (left) region of the spectrum and again phase by clicking and dragging. Click on the **[Box]** or **[Cursor]** to exit Phasing. This process changes **rp** and **lp**. If things get messed up, set **rp=0** and **lp=0**.

Referencing

Click on **[Dscale]** to display the scale in ppm. To display the scale in Hz, type **axis='h'**. To set it back to ppm, type **axis='p'**.

Usually, referencing is not necessary. However, if you chose the wrong solvent in Walkup, you will need to re-reference. First, you will need to find either the solvent reference line or the line for TMS if it is present. The solvent reference ppm values can be found on the solvent table posted in the lab.

First, expand the reference line, click with the left button on that line (if a multiplet, click the middle peak). Type **nl** (nearest line) and then **rl(0p)** for TMS or **rl(7.24p)** for chloroform or use the appropriate value for your solvent.

Peak Picking

Peak picking is useful especially for ¹³C NMR. First adjust the threshold by selecting the **[Th]** menu button in the display mode (type **ds**) and moving the yellow line up and down with the left mouse button. Then type **dpf** to display the peaks above the peaks. Type **dll** to display the peaks in the text region below the spectrum. Type **pll** to print the peaks.

Integration

To integrate, first clear all integral resets by typing **cz**. Next, click **[Partial Integral]**. The button will change to **[Full Integral]** when you do this and a solid green integral line will appear. If the line is not flat where there are no peaks, use the **[lvl/tilt]** button to flatten it (works like the phasing above). Select **[Resets]**. Now use the left mouse button and click to the right and left of each peak that you want integrated. Regions that are integrated are **solid green lines**; non-integrated regions are dotted green lines. Click on **[Box]** to get out of **[Resets]** mode. When the integral lines are displayed, clicking and dragging with the middle button will vertically expand them. To get out of integration mode, click on **[No Integral]**.

If you want to set an integral to a certain value (e.g. a methyl group to 3.0), place the cursor on the integral and select **[Set Int]** in the display mode (**ds**) and then enter the value. To display the integral values, make sure **vp=12** and type **dpir**.

Useful VNMR Commands (C), Macro Commands (M), and Parameters (P)

aph autophases spectrum (C) **axis='h'** displays axis in hertz (P)
axis='p' displays axis in ppm (P) **bc** performs baseline correction of non-integrated region of spectrum (C) **cexp(x)** creates experiment where x=1 to 9(C) **dg** displays acquisition/processing parameters of experiment. (C) **dli** display a list of integrals (C) **dll** display a list of line frequencies and intensities (C) **dpf** displays peak frequencies (C) **dpir** display integral amplitude below spectrum (C) **dps** display pulse sequence (C) **ds** display spectrum, allows interaction (C) **dscale** display scale below spectrum (C)

dscale(10) display scale at vertical position 10 (moves scale only)
(C) **e** ejects sample, not needed with Walkup Automation. (C) **I**
insets sample, not needed with Walkup Automation (C) **jexp(x)** joins
experiment x that you created. (C) **lf** list files in directory (C) **nl**
position cursor at nearest line (C) **np** number of data points (P) **nt**
number of transients (scans) **pll** plot line frequencies and intensities
in columnar form.(M) **pw** pulse width (P) **pw90** 90° pulse width (P)
rl(77.0p) sets peak with cursor on it to 77.0 ppm (C) **spinner** opens
the Spinner control window (C) **svf('filename')** save fid file
directory (M) **sw** spectral width in hertz (P) **tn** nucleus for observe
transmitter (P) **vp=10** displays both scale and spectrum at vertical
position of 10 (P) **vs=10** sets vertical scale to 10 (variable) (P)

Troubleshooting

In most cases, contact Professor Green (474-1559) for problems with the NMR. Do not try to fix the problem yourself unless you know exactly what you are doing. Under no circumstances should you reboot the computer!!! This can cause problems. The Unix system needs to shut down with specific commands.

If you see that the Acquisition Status reads “Find Lock Res.”, this means that the autolock function, **alock**, is probably not set correctly. You need to click on **[Abort Acq]** and Type **alock='n'**. Then restart your experiment.

If the **Status Acquisition** Window reads **Status: Inactive**, click on **[More]** and then **[UNIX]**. A Unix window opens. Type **su acqproc**. The window should now read **Status: Idle**.

VNMR User Manuals (in NMR Room)

Getting Started User Guide: Liquids NMR VNMR Command
and Parameter Reference UAF NMR Website:

<http://www.uaf.edu/chem/green/NMR.htm>