1. When you get up to the instrument, log into the computer as **pchem**. The password is **2007compute**.
2. The computer will load, and on the toolbar at the bottom, click on the spectrum icon (VNMR). This will load the NMR instrumentation software.
    a. If the P-Chem DMA sample ISN'T inside the instrument (i.e. someone has taken it out and placed another sample inside), insert the sample into a spinner (correctly, using the diagram next to the instrument). Type **e** <enter> into the command box in the software to eject the sample inside. Remove the sample and place the DMA tube and spinner into the instrument. Type **i** <enter> to insert the sample. You should here a click when the sample is secure.
    b. If the P-Chem DMA sample IS inside the instrument, do not remove it – simply follow the directions below.
3. The first group of the afternoon should do a trial at room temperature. Type **h1** <enter> to load the parameters. You may need to also load the $CDCl_3$ solvent parameters (especially useful if the DMA sample wasn't in the instrument already).
4. Shim the sample. Click the *Aqui* button, and in the window, click the *Lock* button. If the sample is locked, you should see a plateau in the window. If you DON'T see a plateau, do the following:
    a. Turn the lock off
    b. Adjust the top sliding bar (Z0C) until you see a step function (i.e. plateau)
    c. Turn the lock on
    d. If the plateau is off scale (too high), adjust the lock gain down to 10 or 20
5. Once you see a plateau, the instrument should be locked on the sample. You will know this once the window says "LOCKED" in the lower left hand corner. Now click the *Shim* button. Your goal is to make the bottom number ("current lock value") in the top window as large as possible. Adjust only Z1C and Z2C when shimming this sample. Using the leftmost and rightmost mouse buttons, click on the *+/- 16* button for Z1C. If the "current lock value" increases, you are going in the correct direction; if not, click the other mouse button to go in the opposite direction. Follow shimming Z1C by adjusting the *+/-16* button on Z2C. Then, adjust the *+/-4* and *+/-1* buttons for both until the shimming value is at a maximum. Click the *Close* button in the top toolbar when you are done.
6. Once back in the main window, type **nt=1** <enter>. This changes the number of transients to one, allowing you to quickly see if your shim is good or not. Type **go** <enter> to begin the experiment.
7. Once the experiment has completed, the box in the upper right hand corner should say that the instrument is "idle." At this point, type **wft aph f** <enter> into the command box in the main screen. This performs a weighted Fourier transform on the data, autophases it, and gives a full spectrum in the window. If the shim is poor, reshim and type **go** <enter> to try again. If the shim looks good, type **nt=16** <enter> and then **go** <enter> to run the full experiment.
8. Upon completion of the experiment, type **wft aph f** <enter>. Your spectrum should reveal five lines. Type **dscale** <enter> to show the scale in ppm. The first two lines are the two H environments for ethylene glycol.

a. Each group should draw ethylene glycol to figure out which H are in which environments. A couple points: the nomenclature is deceiving – they need to figure out that "ethylene" doesn't mean a C=C double bond and that it is actually the nomenclature given for a H_2C-CH_2 moiety. Also, they should be able to tell you why the first peak is broader than any of the other spectral peaks.
b. Each group should also tell you which H atoms generate the other three signals. They should also tell you why the doublet exists and how it can be changed (the entire point of the laboratory exercise).

9. Type **axis='h'** <enter> to change the scale to Hz. Click the *Th* button at the top. A yellow line should appear over the spectrum. Move the line so that it is below all the peaks that appear in the spectrum. Click *Th* again to remove the line.
10. Type **dll** <enter> to show the value of each peak in Hz. The values will appear in the grey box below the spectral window. You should only receive five values. If you see more, there are a couple of reasons. The shim might be poor, so you could reshim. If two peaks are there, you may just use the average of the two values – use your best judgment.
11. Subtract the shift (in Hz) of the second peak from the first. This will give you a shift value (in Hz) that you can use to calculate the temperature. There is a sheet next to the computer that has a table of ethylene glycol shifts in a 300 MHz instrument. Use the calculated difference to find the correct internal temperature of the sample. Be aware that it is often considerably different from the value that appears on the box next to the computer.
12. Click on the *Main Menu* button, and then click the *Data* button. This will cause text to appear above the command box. The text is the folder in which each spectrum will be saved. Make sure someone writes this down for use with MestrCe. Type **svf** <enter> to enter a file name.
    a. Let's say Bryce, Amanda, and Emily are in a group. They ran their DMA sample, and the temperature calculated from the internal standard was 23.0°C. They would save their file as **bae.dma.h1.cdcl3.230** <enter>. The first ones are the initials of the group members, followed by sample name, nucleus, solvent, and temperature to the tenths position (without the decimal point).
13. You are now ready to change the temperature – let's say to 30°C. You MUST do the following steps in QUICK SUCCESSION in order to be most efficient. Type **h1** <enter>, **temp=30** <enter>, **su** <enter>. This sets up the H parameters for the experiment and sets the temperature to 30°C. If you don't do the steps in quick succession, the temperature will fall quickly when you load the hydrogen parameters, and it will take you longer to raise the temperature. Remember that the temperature that you type in is reflected on the box next to the computer and is likely not an accurate representation of the temperature of the sample (which is what you're studying in the experiment – crazy how that works).
    a. Only raise the temperature in small increments (on the box) – no more than ~15°C per time. This prevents the instrument from overheating by raising the temperature too quickly and negative effects on the sample itself.

14. The temperature will rise, and it'll probably get to a temperature that's about 0.1-0.2 degrees less than the one you put in. When the temperature remains at either one of these "close" temperatures or the temperature itself for at least 2ish minutes, you are ready to shim the sample, starting at #4 on this list.
    a. Ask the group why you need to shim each time. Their answers should be that the goodness of the shimming is dependent on temperature, so a new temperature requires a good shim. Shimming quality is also dependent on the sample itself, which is why you need to reshim every time you insert a new sample. Another related concept is that Z1C is affected by Z2C and vice versa, which is why you need to shim both every time.
    b. Make sure the group members are observing the positions of the doublet peaks during each experiment. They should see them move closer together, evidenced either by a lack of resolution to baseline between the peaks or a smaller difference (in Hz) of the two peaks (can be found from the data generated in step #10).
15. Repeat steps 4-14 until each member of the group has had a chance to shim the instrument and run the experiment. <u>This is really important – everyone deserves a chance to try it out and see how it works</u>. The first group of the afternoon should go from room temperature to ~55-65°C on the box (about four steps for four people in the group) and collect the relevant data. The last person of the first group should set up the temperature for the next group, so that in the transition time, the probe can be heated up.
16. <u>Watch the temperature rising carefully</u>. If the temperature is increasing too rapidly, hit the bright orange-red power button on the box next to the computer. This will shut off the heating mechanism of the probe.
17. After all experiments have been completed (usually when the temperature on the box says ~110-130°C), the group should see one peak for the methyl groups. Ask the group what that indicates. Don't use a temperature higher than 120 ºC on box.
18. Let's say the final temperature is 120°C. Before leaving for the day, you must decrease the temperature of the probe IN STEPS. Type in **temp=90** <enter> and **su** <enter>. The temperature will now decrease rather rapidly, and equilibrate to ~90°C. <u>You must wait AT LEAST 10-15 minutes with the temperature at the 90°C mark</u>. Then, type in **temp=60** <enter> followed by **su** <enter>. <u>You must wait AT LEAST 10-15 minutes at this temperature before doing the next step</u>. Take the temperature down at the same increment and WAIT AGAIN. Finally, take the temperature down to ~22°C. When it's near this temperature, log out of the software by typing **exit** <enter> and clicking the *log off* button. Press the power button on the box to turn off the probe's heating mechanism. The sample can be left inside because it has a chloroform solvent.