NMR Users Guide
Organic Chemistry Laboratory

Introduction

The chemistry department is fortunate to have a high field (400 MHz) Nuclear Magnetic Resonance (NMR) spectrometer. You will be using this instrument for analysis of products throughout the organic laboratory. As such, you will need to be familiar with its use and the use of the Delta software needed to analyze the resulting data.

NMR Sample Preparation

Liquid samples

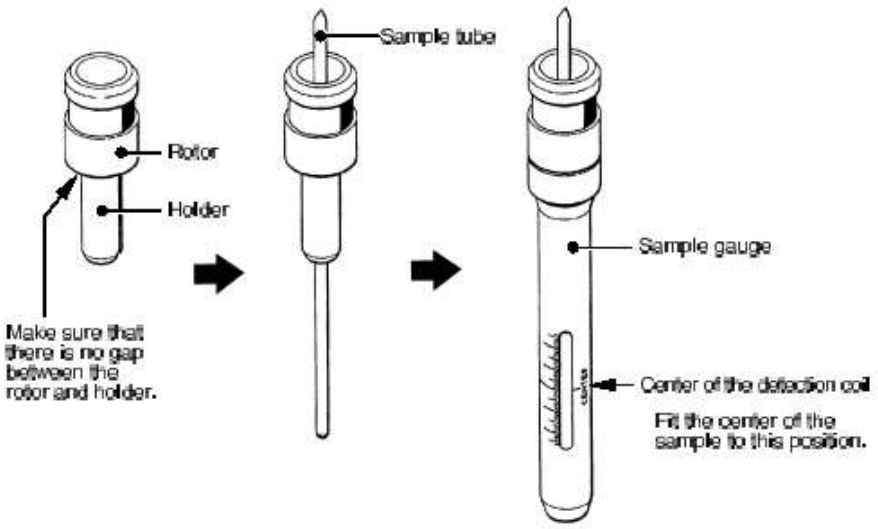
Add the 2-3 drops of the liquid to an NMR tube
Add enough solvent to fill the NMR tube to 35 mm in height (about three fingers full), roughly 0.75 mL.
Gently place a clean cap on the NMR tube
Invert the tube several times to mix the sample

Solid samples

Add 30 mg (0.030g) of sample to a sample vial
Add about 0.75 mL of solvent
Mix to dissolve the sample
Transfer the solution to an NMR tube
If necessary add enough solvent to bring the solvent level up to 35 mm in height
Gently place a clean cap on the NMR tube
Invert the tube several times to mix the sample

Loading the NMR tube

Carefully insert the NMR tube into the wide end of the Teflon spinner
Make sure the inner piece of the spinner is inserted as far down as possible
Use the clear plastic depth gauge to set the NMR tube to the correct depth
Place the assembly in the auto sample holder in the slot whose number matches the one on the spinner assembly
Never reach across the autosampler to load or retrieve a sample



NMR tube cleaning

Remove the NMR tube from the sample spinner and return the spinner to the console
Discard the NMR solution into an appropriate waste container
Place the NMR tube cap on the bottom of the NMR tube
Invert the NMR tube into the NMR tube cleaner (SC111)
Turn on the water aspirator
Rinse the tube with three portions of acetone
Remove the tube and discard the cap
Place the NMR tube in the drying oven (SC 112)

Running a routine NMR sample

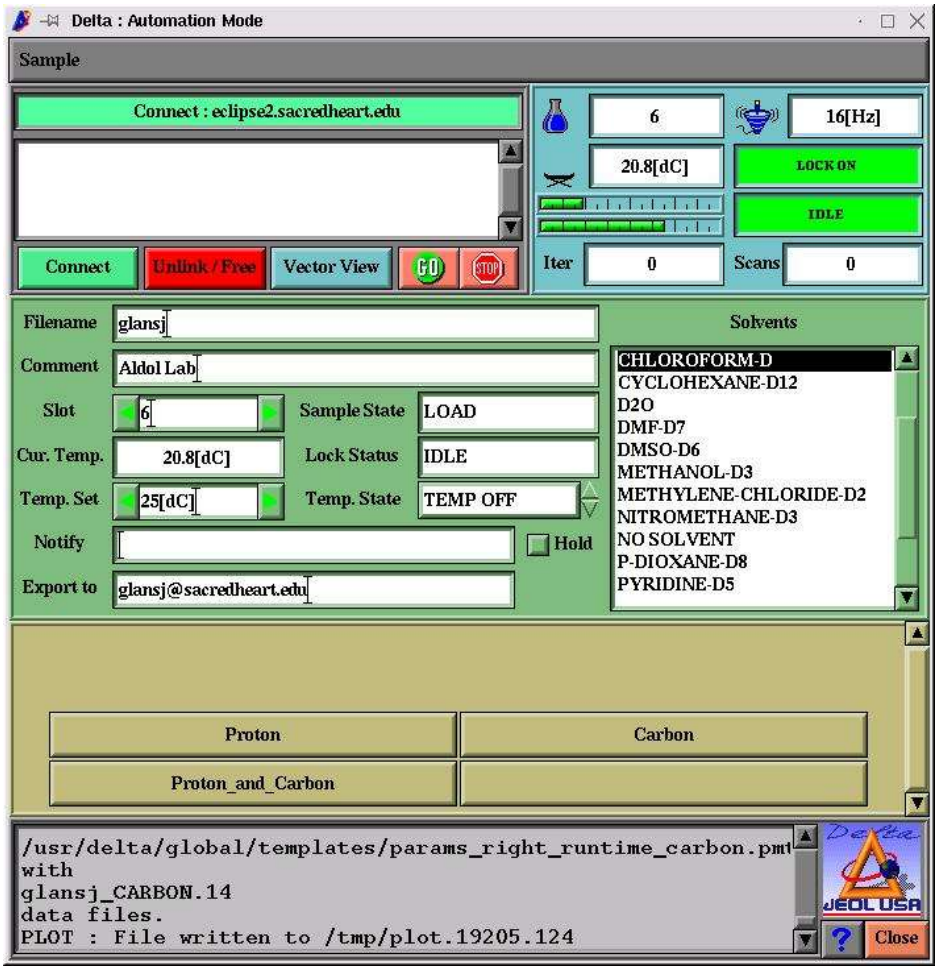
Make sure your sample is loaded in the autosampler and you know the slot number

Entering sample information

Filename: **your SHU username**
Comment: describe the **sample**
Slot: enter the **number** your sample is in
Solvent: select the **solvent** you used to prepare the sample
Recheck all of the entered values BEFORE going to the next step
Enter your email address into the **Export** to dialog box

Select which type of spectrum to be run

Proton – runs a simple proton NMR spectrum
Carbon – runs a simple carbon-13 NMR spectrum
Proton and Carbon – runs both spectra, more quickly than selecting both individually



Delta NMR Software

Installing Delta: Copy the appropriate Delta install file (either PC or Mac) from the department NMR website or one of the CDs in the library to your desktop. Double click on the file to begin the installation. Follow the directions during the install. During the install choose **minimal** for the Installation Set.
If you continue to have trouble loading either version visit the Help Desk, they can do it for you.
Once the program is installed you will need to give it a license key. To do this, open the program. Choose **File:Installation:License Key**. In the blank spaces enter: HN-JS-O4-6O-AW. This will activate your version of Delta. If you do not do this your version of Delta will close after 15 minutes.

Renaming Delta files: The SHU email system sometimes strips the name off of the Delta file during delivery. The resulting file will be named Delta Data. Unfortunately Delta will not be able to open this file without renaming. All Delta files must end in a dash and a number (e.g. filename-1.jdf). So you will need to rename Delta Data.jdf into this format. I suggest not using the same name repeatedly (e.g. delta data-1.jdf) as you may overwrite any other files you have saves on your computer.

Opening an NMR file: Right click on the file and choose 'open with'. Choose Delta. From now on double clicking any NMR file should open Delta.

A detailed manual is available by clicking on the '?' button on the master console. If you run into any problems this is a good place to start.

Analyzing a ^{1}H NMR spectrum (see video on department NMR page).

When you open your spectrum you should see a correctly phased spectrum. If you see an FID instead, you will need to import the appropriate processing list. Do this by clicking on the processing list button (upper right). In the dialog box that opens up click on 'Global'. Choose proton_autophase.list from the list that appears. The program will automatically run the processing list.

Note that holding your cursor over any button will tell you what that button does.

Examine the baseline of your spectrum. It should be nice and flat with no peaks extending below the baseline. If not, the spectrum is not correctly phased. You can touch up the phase by clicking on the 'phasing' bar on the far right. This will open an inset window in the lower right. By clicking on the P0 and P1 buttons you can adjust the phase. Phasing is particularly important in order to get good integrals.

Click on the 'options' bar on the right side, this will open another inset window with a number of useful settings.

Tools: Across the top of the spectrum are a number of tools useful for analyzing your spectrum.
The first is the zoom tool.

- Clicking and dragging below the x axis will zoom the x axis only
- Clicking and dragging to the left of the y axis will zoom the y axis only
- Note the key short cuts above and to the right of the spectrum, these will allow you to quickly move about the spectrum. Reset will remove all zoom to give the full spectrum. Unzoom will undo the last zoom command.

Setting the reference

- Make sure the X Ref in the options box is 0
- Click on the paste reference tool
- Click on the TMS peak, this will now be set to zero
- If no TMS peak is present, change the X Ref to the solvent and click on the solvent peak (usually chloroform)

Integration

- Click on the Auto Integrate Button at the very top of the screen
- This will integrate all peaks in the spectrum
- Split any overlapping peaks
    - Select the integral with the 'select' tool
    - Click on the upper left grab box
    - A pair of scissors will appear
    - Drag these midway between the two peaks and release
    - Repeat as necessary working from left to right
- Normalize the integrals
    - Select a known integral (e.g. a methyl group)
    - Type the integer value into the 'normal' box in the option and hit return
    - Make sure all of the integrals are near integer values, adjust as necessary
- Select and delete to remove unwanted integrals

Picture in Picture

- Allows expansion of multiplets
- Select PIP tool
- Click and drag in an open area of the spectrum
- Use the zoom tool to select the peak(s) of interest
- Use the select tool to move the window around and adjust its size
- Use the delete key to remove any unwanted PIP windows

Final Review

- Set the x axis to 0-8 ppm (unless you have peaks at >8 ppm)
- Make sure all peaks have a normalize integral
- Make sure there is an expansion for all defined multiplets

Analyzing a ^{13}C NMR spectrum (see video on department NMR page).

Save the file to your computer and double click it to open. It should open as a fully processed spectrum, but if only an FID is present click on the open processing list button and in the global directory select carbon_autophase.list. This should automatically process, if not, click the process button.

Open the phasing and options windows
Make sure the phasing is correct, use the manual phasing buttons (P0 and P1) if not.
All of the tools work the same way as they do for a proton spectrum. Carbon spectra are not quantitative so they are never integrated. It is also usually not necessary to expand any portions of the carbon spectrum as they tend to be quite simple. It is a good idea to peak pick them however.

- Select the automatic peak pick button on the top line
- Adjust the threshold button as necessary to include all peaks but not the noise.

Set the full spectrum to 0-200 ppm (unless you have a peak above 200)

Analyzing an NMR spectrum (see video on department NMR page).

1. identify any impurity peaks such as solvent or water (see Table 1). For instance, in the proton spectrum $CDCl_3$ has a residual peak at 7.27 ppm and water shows up at 1.5 ppm (see the table below for other possibilities).
2. draw the structure on the spectrum and label each proton or carbon
3. using the chemical shift charts in your text assign each of the peaks to a nuclei on the structure
4. all peaks on the spectrum should be assigned to either the compound or an impurity

Plotting your spectrum: The most reliable way to print within Delta is to use the built in PDF printer. This has the advantage of giving you an electronic copy of your spectrum as well. Click on the plot button on the top line. Select JEOL PDF. This will open a dialog box prompting you where to save your file. Once you open this PDF file you can print is normally.

Table 1. Chemical Shifts for Common NMR Solvents.

Solvent	*^{1}H Shift (ppm)*	*^{13}C Shift (ppm)*	*Water Shift ^{1}H (ppm)*	*fp/bp ^{o}C*
Acetic Acid-d4	2.05 11.7	20.0 179.0	-----	17/118
Acetone-d6	2.05(5)	29.5(7) 206.7	2.8	-94/57
Acetonitrile-d3	1.94(5)	1.39(7) 118.7	2.1	-45/82
Benzene-d6	7.23(1)	128.0(3)	0.4	5/80
Chloroform-d	7.27(1)	77.0(3)	1.5	-64/62
Deuterium Oxide	4.80	none DSS=-1.85	-----	4/101
Dimethylformamide-d7	8.03(1) 2.92(5) 2.75(5)	162.3(3) 34.9(7) 29.8(7)	3.5	-61/153
DMSO-d6	2.50(5)	39.5(7)	3.3	18/189
Dioxane-d8	3.53(mult)	66.7(5)	2.4	12/101
Methanol-d4	4.87(1) 3.31(5)	49.0(7)	-----	-98/65
Pyridine-d5	8.74 7.58 7.22	150.4(3) 135.9(3) 123.9(3)	5.0	-42/116
THF-d8	3.58 1.73	67.5(5) 25.4(5)	2.4	-109/66
Trifluoroacetic acid-d	11.5	116.6(4) 164.2(4)	-----	-15/72