JOEL 270 MHz Broad Band Multinuclear FT-NMR is Located at Carson Taylor Hall Room 306

Initial Preparation

Sample Preparation

The NMR tube is 20.5 cm (8 inches) long. The sample volume fills the tube to a depth of 4.5 cm. The sample solution is clear and a single phase. For proton NMR spectra, samples should be prepared as follows:

A) weigh 5- 10 mg into an appropriate container (NOT the NMR tube!);
B) add 0.7 - 1.0 ml of deuterochloroform ($CDCl_3$) and stir or shake to dissolve any solids;
C) filter to remove any undissolved solids or alternatively, cleanly pipette off the clear (NO SOLIDS !) supernatant liquid;
D) D) pipette the solution into a 8 inch undamaged NMR tube; and
E) Label the NMR tube containing the sample and store in the proper sample rack location.

LOGGING ONTO NMR AND CONNECTING TO COMPUTER TO NMR SPECTROMETER

1. Sign in Log book
2. Turn the compressor on
3. Remove dust cover from top of magnet.
4. Confirm lock is on.
5. Make sure printer is on.
6. Click your **Delta** Icon then click Login button
7. In **Toolchest** select **Programs** then **Delta**

(At any time while in Delta you can reach the online manual by clicking the blue Question Mark located in any of the NMR program

windows. Right click and hold any icon button and the name or function will appear.)

8. In the **Delta** Window**:**
9. Left click the Spectrometer Control Icon **(magnet- picture of the magnet)**
10. Select text **"Delta 2 – Free – 270**"
11. Left click the **Connect** button

INSERT SAMPLE, LOCK AND SHIM

1. In the **Spectrometer Control** Window**:**
2. Left click the **Sample** button
3. In the **Delta 2** window**:**
4. Click the eject icon **(red arrow up with E)**
5. Switch your **sample** with the **standard** sample
6. Check the **correct height** of NMR tube using **height checking device**
7. Click the **solvent** for your sample (usually **CHLOROFORM-D**)
8. Click the load icon **(green arrow down with L)**
9. Click the Autolock Icon (**lock picture with "auto" sing on it**)
10. Wait for lock to change from working to idle **(red, yellow to green)**
11. Click Z1 and Z2 buttons to maximize signal green bars

ONLY IF AUTOLOCK FAILS with instructors help

1. click **Sawth** button in the Spectrometer Control Window
2. Increase lock level to 180 or greater to see good **deuterium signal**
3. Click on largest part of **FID** (sawtooth to center in the plot)
4. Click **Cancel** Sawthooth button and got to step 9 above

LOAD AND ACQUIRE ^{1}H NMR

1. Click **Expermnt** button In the Spectrometer Control Window
2. In the Other experiment Window**:**
3. Click the **Home** Icon (middle icon)
4. Select the **proton.exp** file and click the ok button

In the Experiment Window (Proton.exp):

5. Type **file name** in text box (cursor must be in box to enter text)
6. Change other parameters as desired (**scans** > 100)
7. Click **Submit** button

8. Click the **GO** button in the Inform Window

9. Otherwise click the **GO** button in the Spectrometer Control Window

ACQUIRE ^{13}C NMR

1. Decoupled ^{13}C NMR

Same as proton experiment except in the Other Experiment window select the **carbond.exp** file (**Scans** >500)

2. Coupled ^{13}C NMR

Same as proton experiment except in the Other Experiment window select the **carbond.exp** file (**Scans** >1000)

PROCESS AND PLOT THE SPECTRUM

In the Processing window:(lock state will take about a minute, watch instrument status in Delta window)

Expansion

1. Hold down the **shift key** (cursor converts to magnifying glass) and drag the **cursor** below the **x scale** to expand desired region

Threshold and Peak Picking

2. To adjust the threshold for peak pick- **right click mouse** and hold in the spectrum without moving the cursor. In the drop down menu, select **Options/Statistics**. This creates **green** and **red** threshold line (for positive and negative peaks) and a **grey** baseline peak.

3. Click and hold the **selection** icon in the upper right corner of the spectrum. Select **Peak** from the selection bar, click **Adjust Baseline** Icon (the **diamond on a line**)

4. Move **cursor** to grey line, **click** and **drag** to **baseline** if necessary

5. Click **Adjust Peak Threshold** icon (**diamond between two lines**)

6. Move **cursor** to **green line**, **click** and **drag** to a position avoiding noise and unwanted peaks

7. Click the **Auto Peak Pick** icon (**Octagon with X**) to display peak position in ppm below the scale

INTEGRATION

1. **Expand** region around first peak to be integrated **(see above)**

2. **Click** and **hold the spectrum selection** icon and select the Integral Icon**.**

3. Remove previous integrals by **clicking** the **remove integral** Icon **(integral with X)** on the selection bar and **click** on each integral to be removed**.**

4. (To delete all integrals simultaneously, **Click** the **menu** Icon **(K in two boxes), click** select all**,** and **hit** the **delete** button on the keyboard**.**
5. **Click** on the **Integral** Icon
6. **Click** and **drag** the **mouse** below the X scale for peak of interest
7. On the Keyboard**, hit** the **Home** button to see the whole spectrum
8. Repeat for other peaks

Normalization

9. **Click** the **select Integral** Icon **(diagonal solid arrow with Integral).**
10. **Select** an Integral to be a **reference**
11. **Hit control N or**
12. **Click Options/Set Integral Normalize**
13. With cursor in text window **type** number and hit **return**

Integral corrections

14. **Click Adjust Integral** Icon **(Integral with perpendicular arrows)**
15. **Expand** around integral to be corrected
16. **Drag Blue box** up or down on left baseline to **adjust integral offset**
17. **Drag blue box** up or down on right baseline to **adjust integral slope**

COUPLING CONSTANTS

1. **Select Peak** mode in the Cursor Tool.
2. **Click Create Peak** icon **(diamond with very small plus sign)**
3. **Click** two desired **peaks** with the **cursor (shift will be listed below peaks)**
4. On the peak tool bar **click Select More Peaks** icon **(arrow with two diamonds beneath it)**
5. **Click** the same two **peaks (highlights with yellow)**
6. **Type** j and the coupling constant in hertz is displayed above the peaks and the average peak position is displayed below between the peaks

PRINT

1. **Click Printer** Icon (near top of window).
2. **select Parameters**
3. To check parameters, in Delta window, **select Tools/Param View**

SHUT DOWN

1. From the **Delta2** window **(Sample Console) Eject** sample, **place 0.1% ethylbenzene standard** in probe, load and lock standard**.**
2. Click **no spin** icon**.**
3. From the Spectrometer Control window, click **unlink**
4. **Close** Spectrometer control window
5. **Close** Delta Console window**.**
6. In Toolchest **select** Desktop/logout
7. Select **Yes for "Do you want to log out now?"**

DEFALUT DISPLAY PREFERENCES

1. To change the default spectral display (such as grid or statistics)

In the Delta window select File/Preferences

2. Click the **Data** icon (green spectrum on black)
3. Scroll to grid and click adjacent button to turn off/on
4. Scroll to Statistics and click adjacent button to turn off/on

Other commonly changed options include

5. Comment (prints in upper left corner)
6. Filename (prints in upper left corner)
7. Integral precision