

Introduction-

This document includes step-by-step instructions for recording a $^1\text{H-NMR}$ spectrum. It also contains comments describing what various commands do, as well as a collection of Figures that show what you should see as a result of each step. In the on-line version of this document, the Figures are embedded in the text. In the hard copy that is kept by the spectrometer, they are collected at the end of the document. The step-by-step instructions are given in black.

Comments are italicized in red.

Sample Preparation-

While it is possible to obtain a satisfactory spectrum using as little as 1 mg of compound, a sample size of 10-20 mg is recommended. It is important to use clean, dry glassware when you are preparing your samples. To dry an NMR tube, slide it onto a 12-inch needle connected to your nitrogen line. Turn on the nitrogen and heat the tube with a hair dryer for one or two minutes.

In a clean, dry vial dissolve your sample in 600-700 μL of CDCl_3 . Use a clean, dry syringe to measure the solvent. Filter the solution through a filter pipet directly into a clean, dry NMR tube as shown in the diagram. Place a cap on the tube.

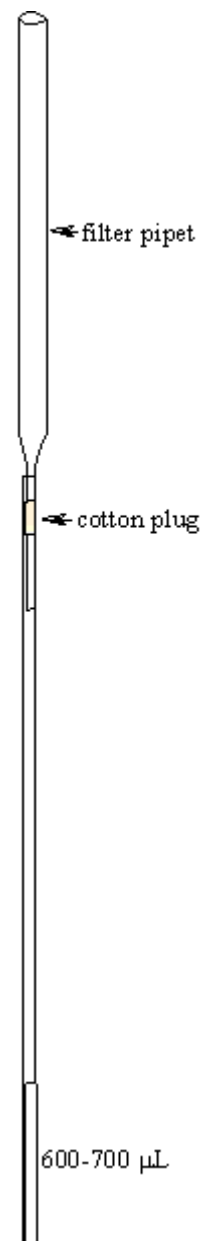
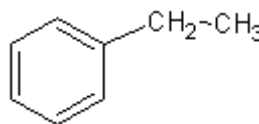
Instruction Format-

Keyboard commands are enclosed between < and >. Thus <zg> means type the letters zg, <Enter> means press the Enter key. <Alt + Tab> means to press the Alt and Tab keys simultaneously.

Saving files-

Obtaining an NMR spectrum involves two programs. The first is called PNMR. It generates the raw data that the second program, Nuts, processes to create your spectrum. The data from the PNMR program is written to a temporary file called "pnmrfid". You will save your processed data in a folder you create on your flash drive.

If this is the first time you are using this instrument, you should record the spectrum of the reference sample (5% ethylbenzene) so that you can compare your results to those indicated at various points in the directions that follow. A set of instructions without annotations is available for more experienced users.

**Collecting Data-**

1. If the PNMR and Nuts programs are not already running, click on the shortcut icons along the bottom of the desktop.
2. Make the PNMR program active.

There are 3 status bars at the bottom of the PNMR window. In the status bar in the lower left corner you should see the symbol $\text{H1}>$. H1 indicates that the spectrometer is set to record $^1\text{H-NMR}$ spectra. The > sign is the program's prompt. It indicates where you type keyboard commands. Start with

3. <zg>, <Enter>

*A window named **Save As** will open with the default file name pnmrfid selected.*

4. Click the Save button.

This is the file you will process with Nuts after you have collected the data. After a short delay the program displays the data as a free induction decay curve (fid). The fid is the yellow part of the image shown in Figure 1.

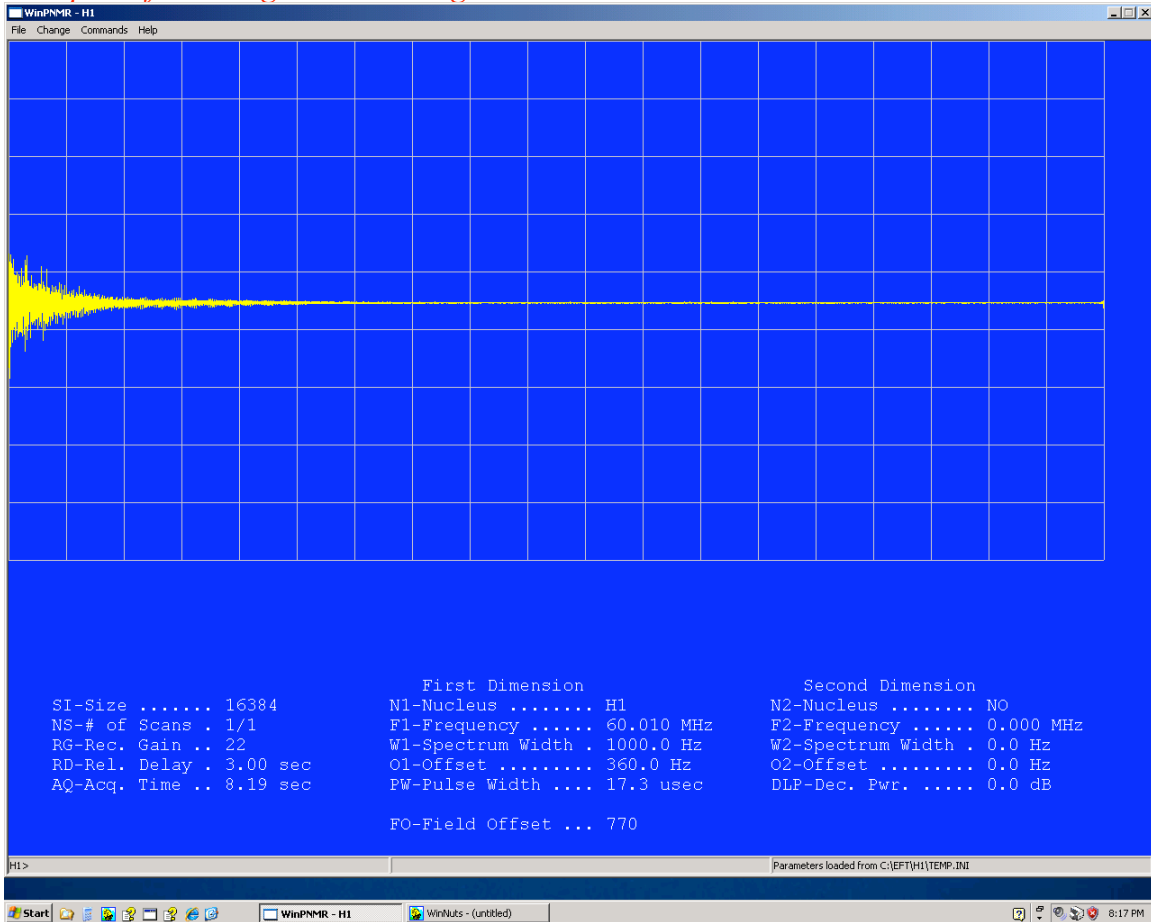


Figure 1-A free induction decay curve

5. <Alt Tab> <Ctrl F2>
6. Select the file pnmrfid and click the Open button.
7. Enter the name of your sample in the COmment field and your name in the USer field of the **Data Acquisition Parameters** window, then click OK.

You should see a spectrum that looks something like that shown in Figure 2. See Note 1 at the end of this document if it does not.

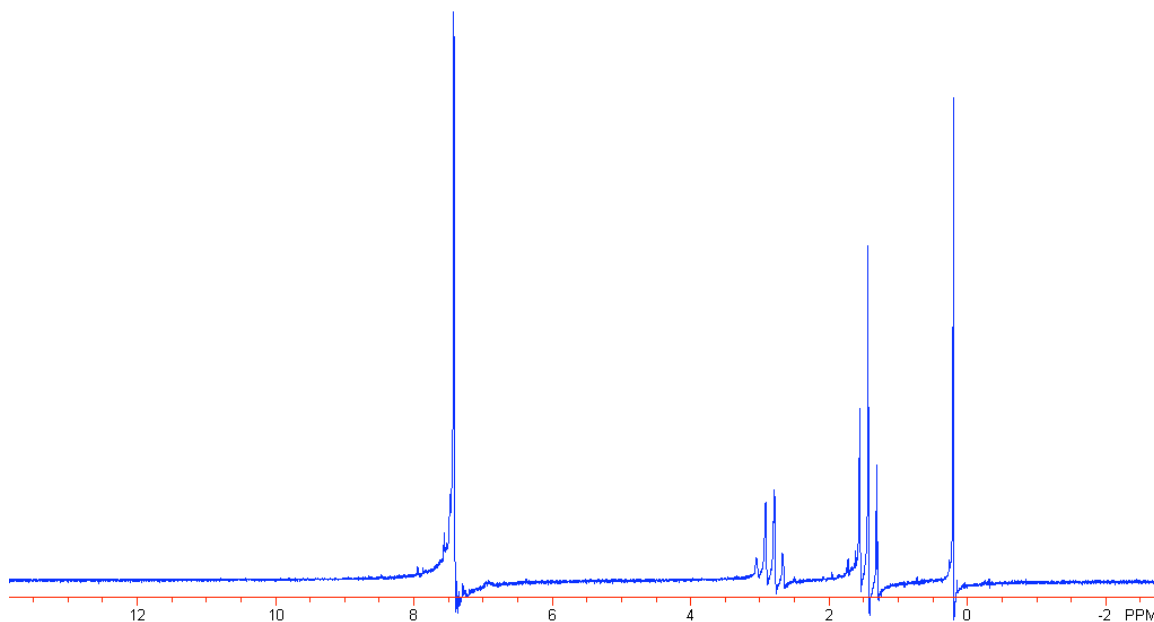


Figure 2-An example of a typical spectrum generated by the command Ctrl F2.

8. <zo>
9. Click and drag the mouse over a strong signal near the left side of the spectrum. Then type the number 1.

Now the spectrum should look something like that shown in Figure 3.

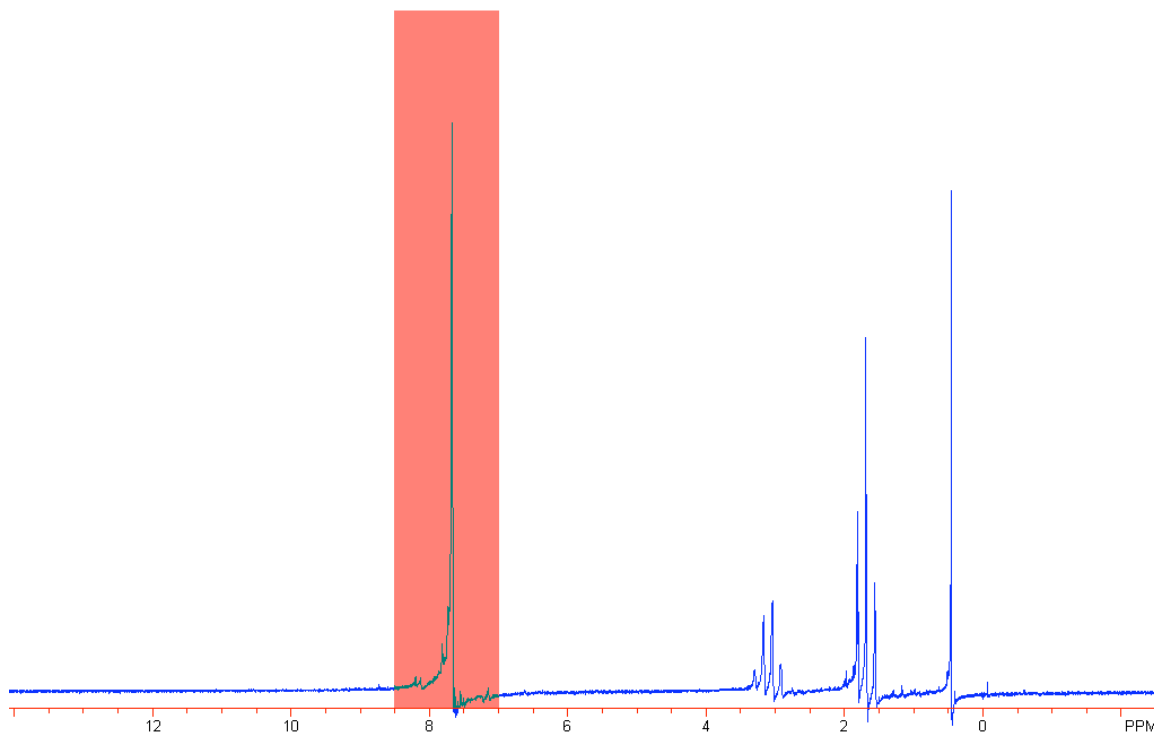


Figure 3- Defining region 1 of the zoom routine.

10. Click and drag the mouse over a strong signal near the right side of the spectrum. Then type the number 2.

Now the spectrum should look something like that shown in Figure 4.

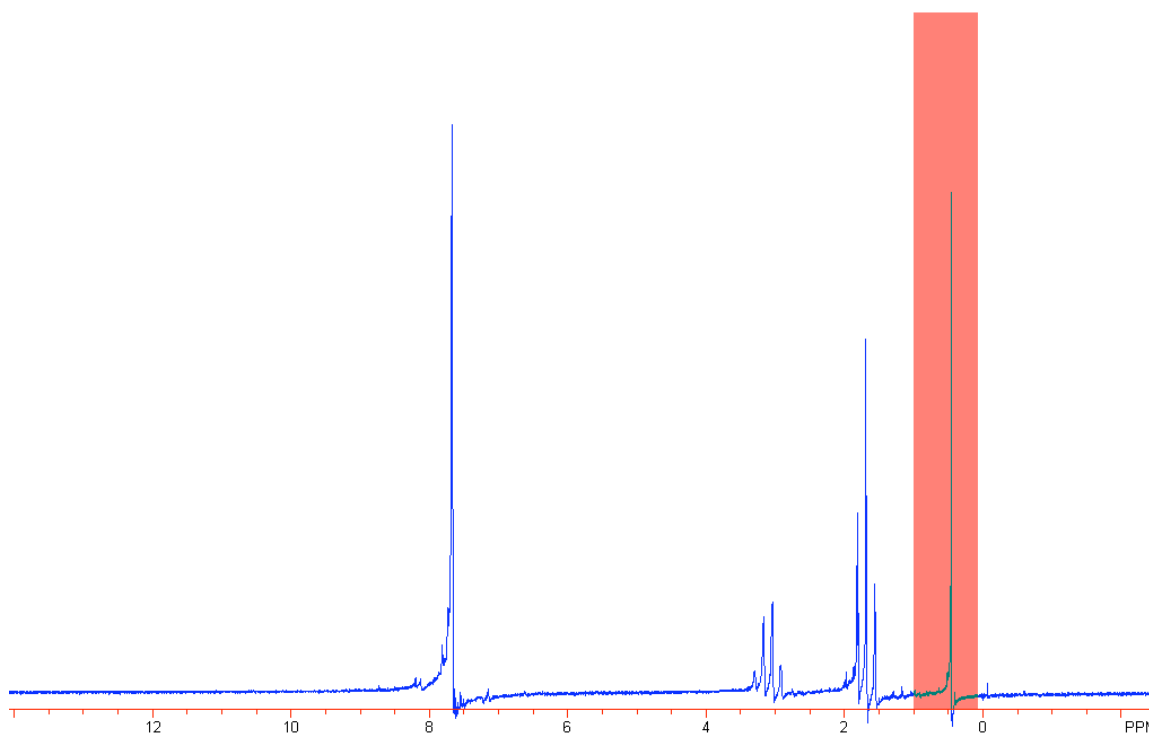


Figure 4- Defining region 2 of the zoom routine.

11. <Enter>

12. <pe>

This displays an expanded view of region 1 as shown in Figure 5. Note that the level of the baseline on the left side of the signal is higher than it is on the right side. The relative levels of the baseline are referred to as the phase of the spectrum. It needs to be adjusted.

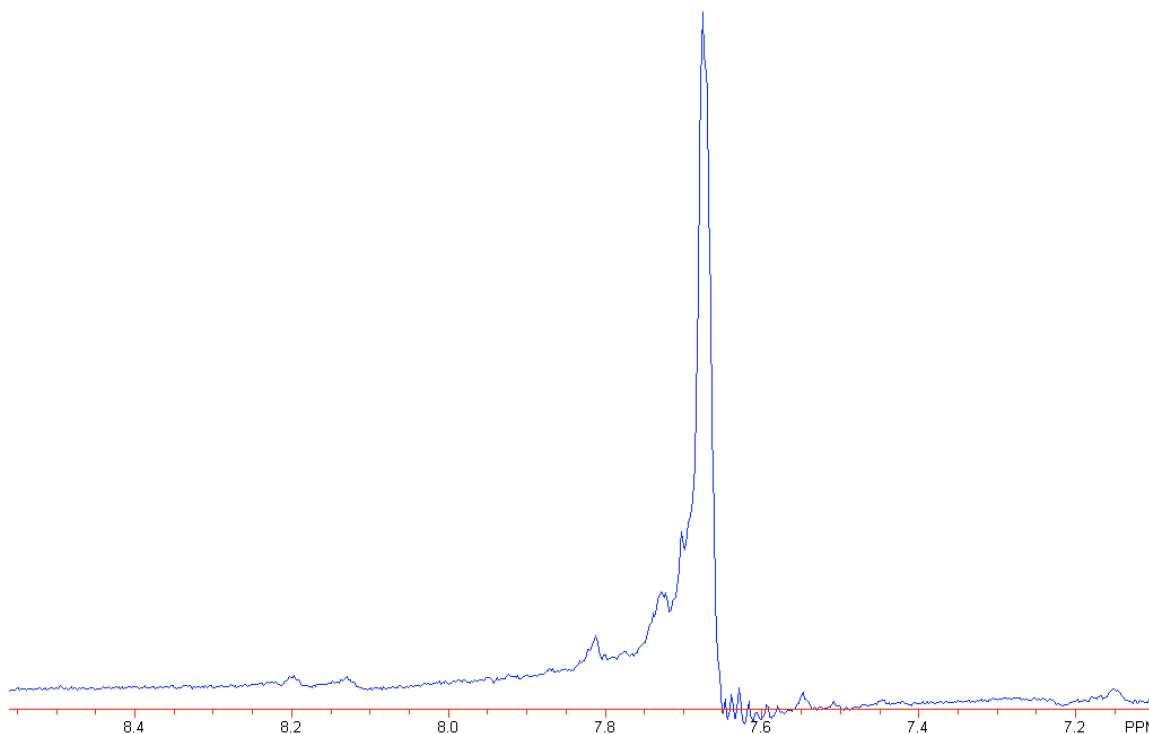


Figure 5- An expanded view of the improperly phased signal in region 1 of the zoom routine.

13. Click/drag the left mouse button horizontally until the level of the baseline on the left side of the peak is the same as that on the right side. You may have to repeat this motion several times.
14. Click/drag the right mouse button horizontally until the level of the baseline on the left side of the peak in region 2 is the same as that on the right.
15. <Enter>

Now your spectrum should look similar to the one shown in Figure 6. Note that the chemical shift of the TMS peak is approximately 0.5 ppm. It should be zero. Step 16 indicates how to set the chemical shift of the TMS peak to zero.

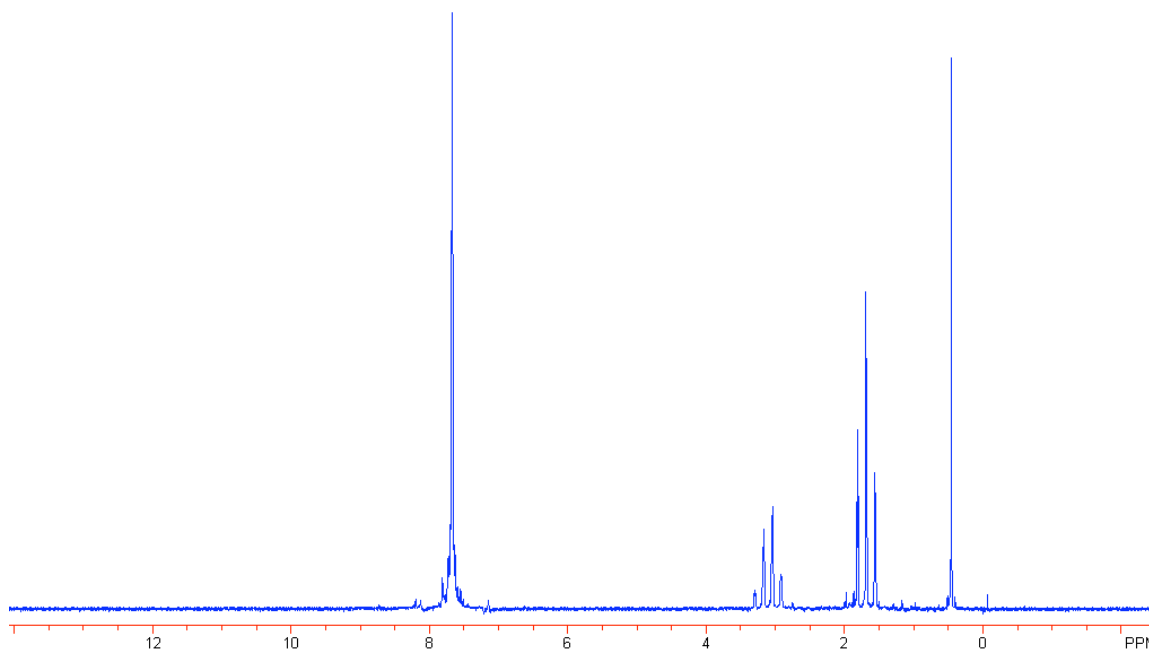


Figure 6- A properly phased spectrum of ethylbenzene.

16. Click the mouse and position the vertical line of the crosshairs exactly on top of the TMS peak. While holding the mouse down, type the letter l and enter the value 0 in the OFFSET INFORMATION dialog box that appears. Click OK.
17. <fb>, <l>, <Enter>
These commands are used to flatten the baseline prior to integration of the spectrum. The second command is the letter l.
18. <id>
Your spectrum should display a continuous integral curve something like the one shown in Figure 7. In the next step you will assign values to the integrals of the three peaks of the ethylbenzene.

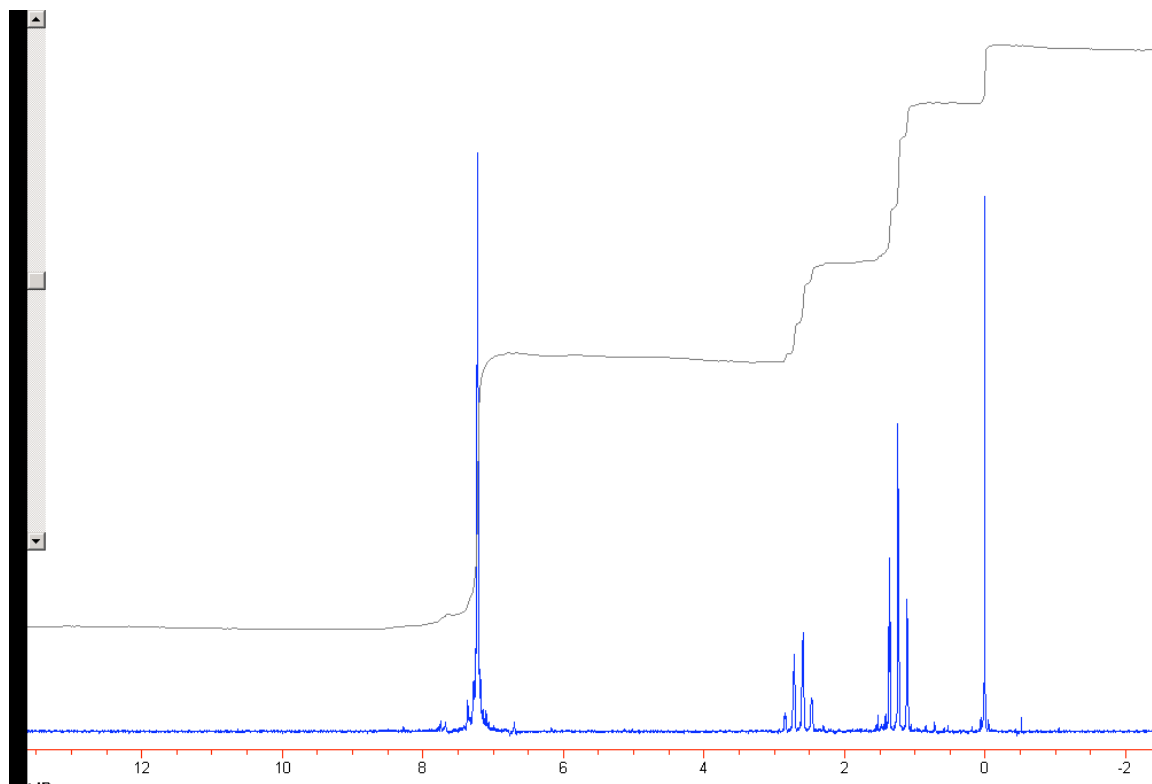


Figure 7-An integrated spectrum of ethylbenzene.

19. Position the cursor on the baseline just to the left of the signal at highest field. Click the mouse two times.

A vertical red line will appear after the first click. It will change to green after the second click.

20. Position the cursor just to the right of the peak and click the mouse again.

Your spectrum should look similar to the one shown in Figure 8.

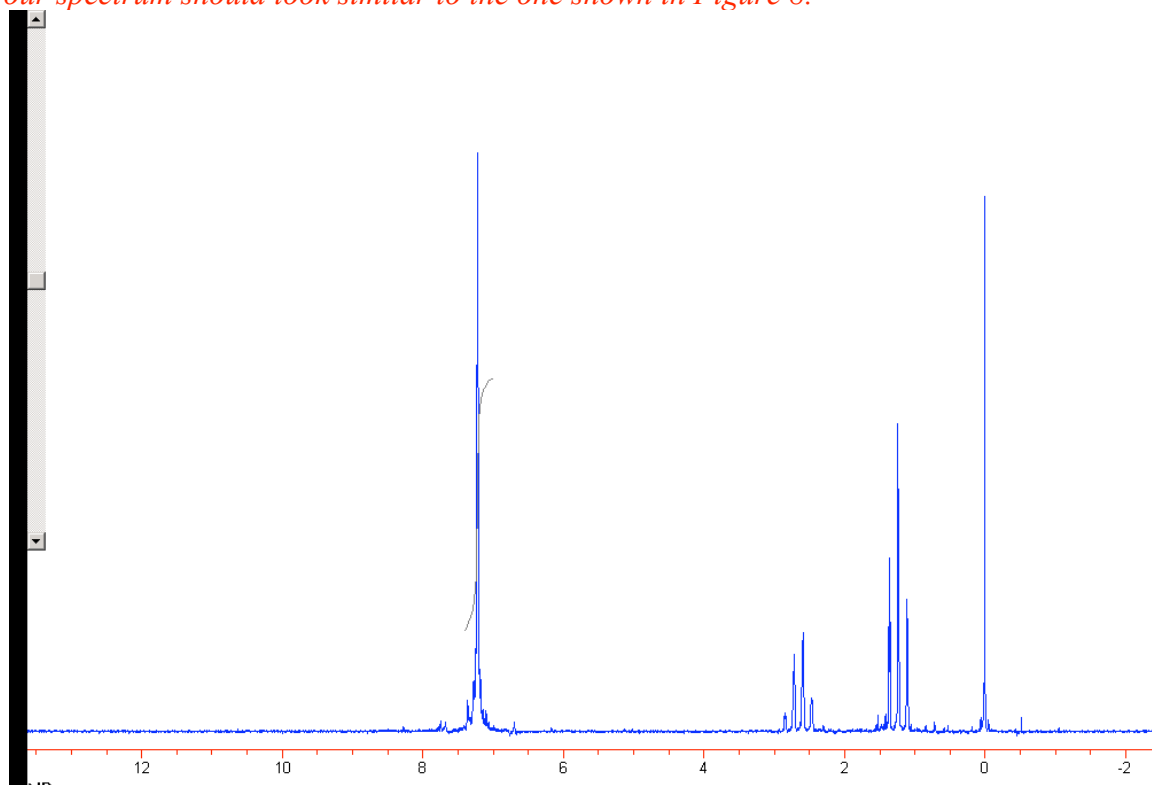


Figure 8-Integration of the signal for the aromatic protons in ethylbenzene.

21. Repeat steps 19 and 20 for the quartet and the triplet in your spectrum.

Your spectrum should contain three separate integration curves like those shown in Figure 9. There is no need to integrate the TMS peak since it is not part of your compound.

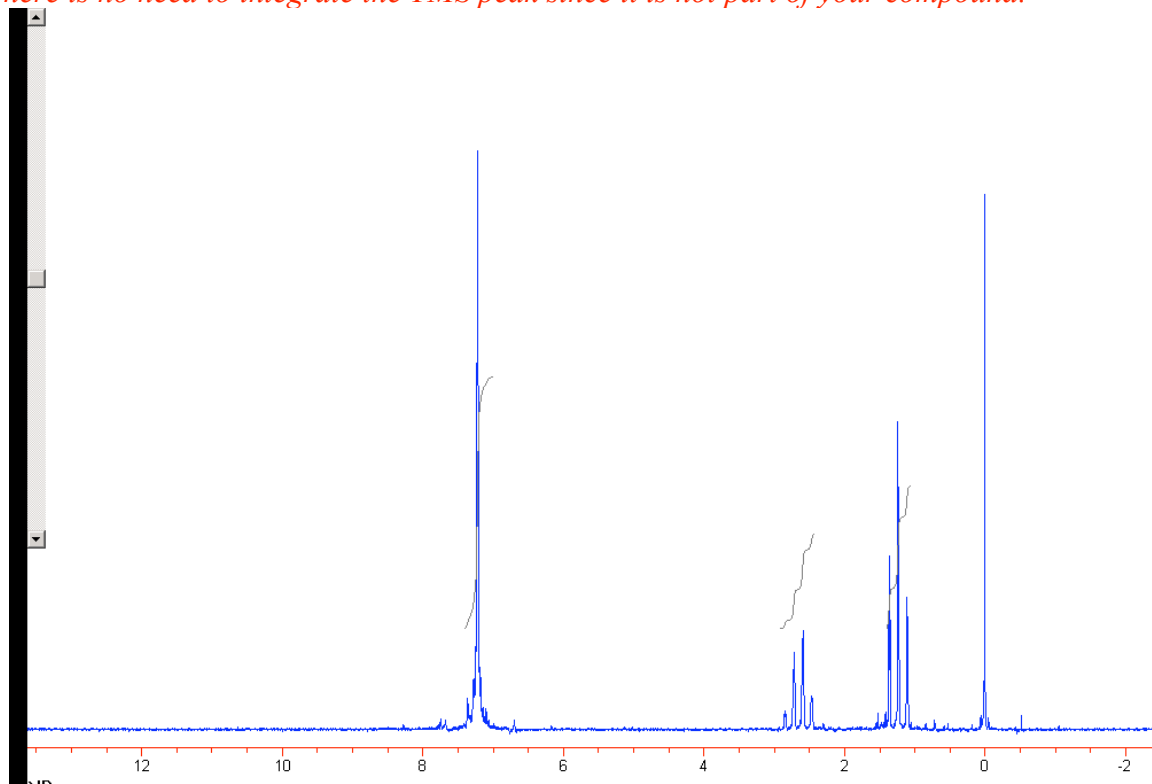


Figure 9-Separate integration curves for the three peaks in the spectrum of ethylbenzene.

22. Place the cursor over the quartet in your spectrum. Click the mouse. Type the letter v. Enter the number 2. Click OK.

This sets the value of the integral for the quartet at 2. The values of the other integrals are assigned relative to that value. The theoretical values are 3 for the triplet and 5 for the signal from the aromatic protons. In Figure 10 the values are 3.00 and 5.31, respectively. There is an inherent error in integration curves of about 5-10%. Note that you could have selected the quartet and set its value to 3, or the aromatic signal and set its value to 5.

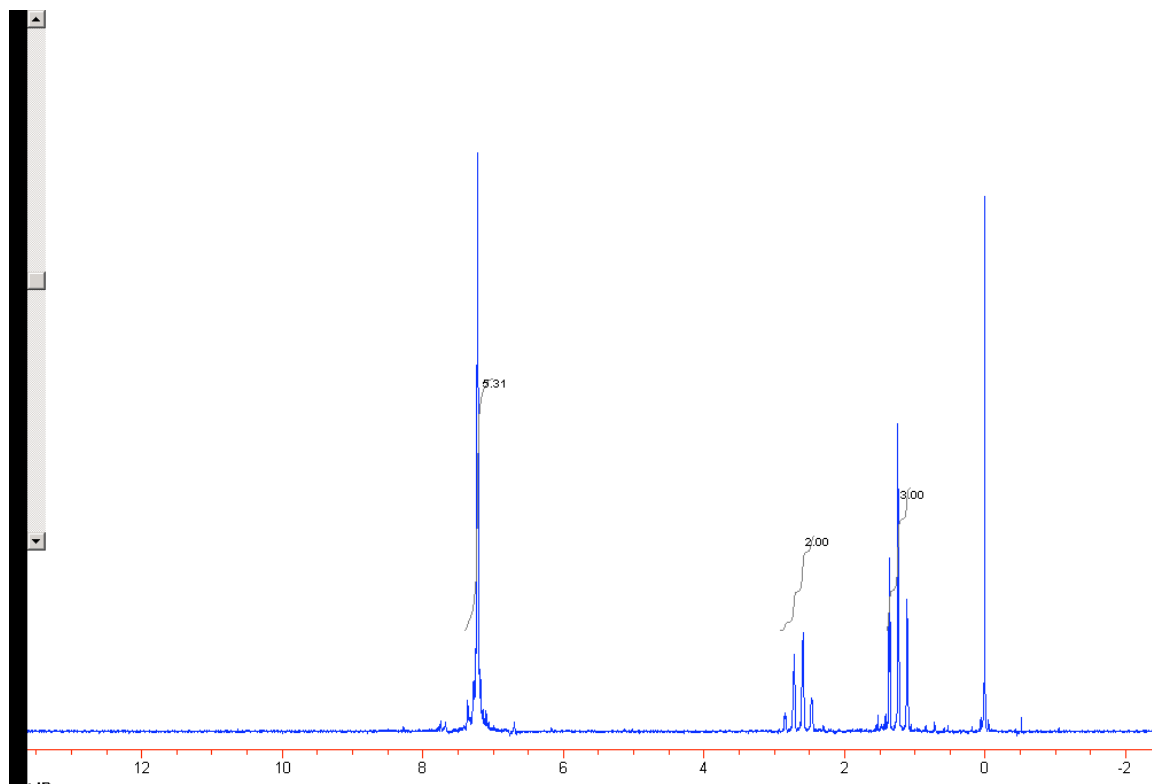


Figure 10-The NMR spectrum of ethylbenzene showing the relative integration values.

23. <Enter>, <Enter>

This takes you out of the id subroutine. You should see a question mark above the command line prompt (>) in the lower left hand corner of the Nuts window.

24. <dp>

A cross labeled DP will appear. You will use it to label the chemical shifts of the peaks in your spectrum.

25. Position the DP cross on the top of the signal for the aromatic hydrogens and click the mouse. Repeat this procedure for each peak that you want to label. <Enter>

26. From the View menu at the top of the Nuts window, select Show Integrals and Show Peak Labels.

Your spectrum should look like the one in Figure 11.

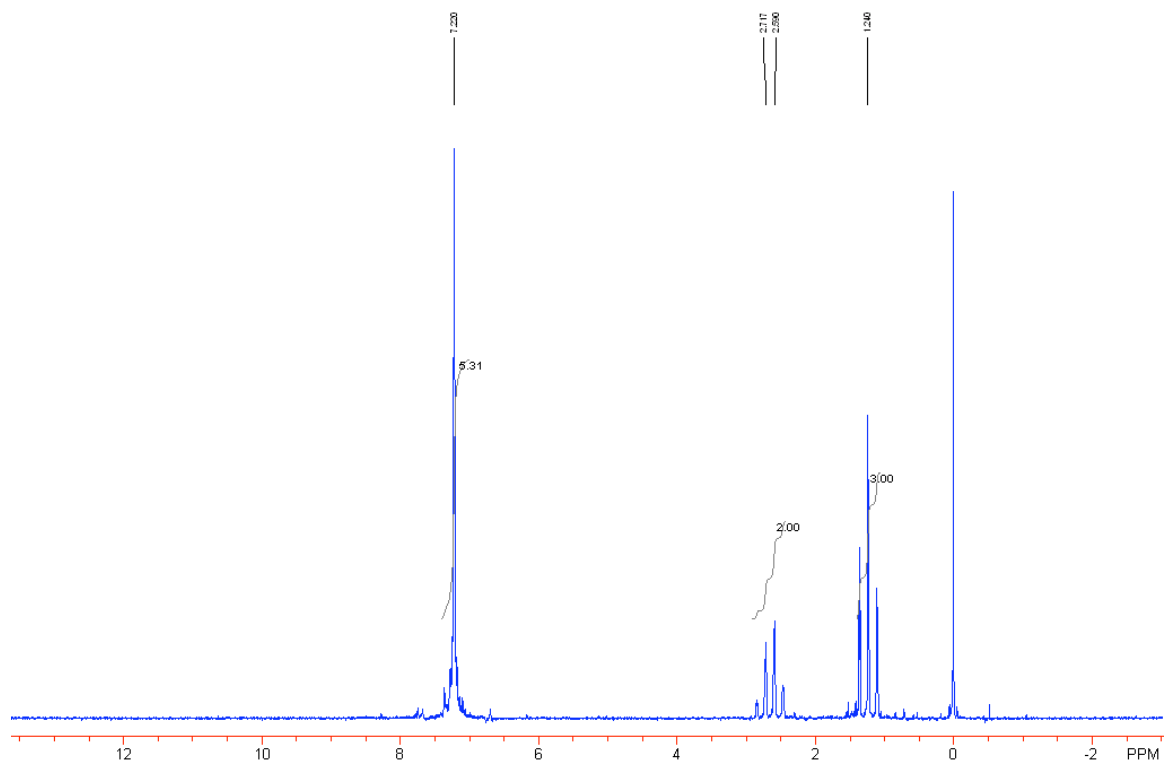


Figure 11-A complete $^1\text{H-NMR}$ spectrum of ethylbenzene.

27. Save your file on your flash drive.
28. Select Print from the File menu if you want a copy of your spectrum.