

EFT Summary Commands

H1 spectra

0. Place sample in spinner to measured depth. Place in the nmr and turn the air off using the elbow switch; then on. You should hear a "purr" at this point.

0. Enter the **PNMR** program.

0. Select H1 parameters. <nu h1> Commands within <> are meant to be typed in the small command line towards bottom of screen. End the command with an "enter". Parameters should be ns=1; rg= 5 for neat samples; rg=30 for 2% samples and for 1-98 ethyl benzene standard..

1. Acquire data. <zg> The computer will ask for a file name. Hit return to use the default name. The FID should be above base line and yellow. If red reduce rg. If there is no signal or very little; increase rg or run standard to find problem.

2. Enter **NUTS** program. Nut commands are just two letters in the main program with NO following enter. If you mistakenly enter a subprogram hit enter to get back to the main program. If the NUTS program is doing strange things; quit it and restart the NUTS application program.

3. Process the data. [Ctrl F2]. To use the default data file hit return when it asks for the data file name.

4. Trim the phase. [ap]

5. Integrate the spectrum [ai]

6. Plot the spectrum [pl]

7. If at any point, you have trouble run the 1-98 ethyl benzene standard to see if the problem is with the instrument or if it is with your sample. If it is the instrument problem contact JWL.

Integration tricks for EFT spectra:

If the integration scale is either too large or too small you can correct it by using the following procedure:

step	action	explanation
1.	use the command id	puts you in the integration display menu
2.	use the scroll bar on the left to adjust the scale	
3.	enter	exits the integration display submenu

If you want to put a numerical value on the integration:

step	action	explanation
1.	use the command id	puts you in the integration display menu
2.	put a cursor on one of the integrations.	
3.	use the command v	This will bring up a window where you can put the value you want on the integration of this peak. Other peaks will be give relative to this.
4	enter	exits the integration display submenu

Phasing tricks for the EFT spectra.

If the phasing of the spectrum is not good. You can manually phase the spectrum by:

step	action	explanation
1.	close the nuts window and restart a new one.	clears any bad information out.
2.	Do the basic processing the same way as usual-control F2 for proton spectra	If the spectrum looks good at this stage do not phase. Just integrate and plot.
3.	use the command PH	This will put you in a manual phase subroutine. Hold down the left mouse and drag it left and right to do a first order correction. Hold the right mouse down and drag it left and right to make a second order correction..
4	enter	exits the manual phase submenu

Clearing carry over information from your window.

step	action	explanation
1.	under the view unclick the appropriate items such as clipboard, peak labels.	

C13 Spectra

0. Place sample in spinner to measured depth. Place in the nmr and turn the air off; then on. You should hear a "purr" at this point.

0. Enter the **PNMR** program.

0. Select C-13 parameters. <nu C13> Commands within <> are meant to be typed in the small command line towards bottom of the screen. End each command with an "enter". Parameters should be ns=16; rg= 100 for all neat samples and for the 3-96 n-Butanol standard. {If no one is waiting for the instrument, you can enter a large number for ns and then <zg> and got to steps 2 and 3. Repeat 3 until the spectra "looks good then plot it. Hit Q in PNMR to stop data collection.} If you use an ns >64 you must wait until no one else has a sample to run. If you use ns >1000 you must wait until the evening.

1. Acquire data. <zg> The computer will ask for a file name. Hit return to use the default name. The FID should be above the base line and yellow.

2. Enter the **NUTS** program. Nut commands are just two letters in main program with NO following enter.

3. Process the data. [Ctrl F3]. To use the default data file hit return when it asks for the data file. Use line broadening of 2 when it asks. Line broadening LB=2.

4. Adjust scale by using scroll bar on right.

5. Plot the data [p]

DEPT Spectra

1. After you have gotten a good C-13 spectrum on your sample Enter the PNMR program. Type <DEPT> in the command line followed by return. The program will ask you for the Name of file. Just hit return if you wish to use the file My_dept for your data. If you want to call it something else- do so but remember what you called it.

2. The program now acquires three sets of data. So just wait. When it is done, it will put up another question box. This just gives you time to read the processing instructions. Hit return when you've read them.

3. Enter the **NUTS** program. Nut commands are just two letters in main program with NO following enter.

4. Process the data first spectrum. [a1] ("a" one)

5. Phase it **if necessary**. [ap]

6. Now put all three of the spectra of the screen. [Cntrl F11]

7. Adjust scale by using scroll bar on right

8. Plot the data [p]

Tuning the NMR

The commands on the previous page assume the NMR is "tuned" and TMS has been used to set the instrument scale to zero. You can determine if these have been done by taking the 1-98 ethyl benzene standard spectrum. A well separated quartet indicates good "tuning" or the correct shims have been set. The TMS signal at zero indicates the offset potentiometer is set correct.

If the instrument is not tuned you have two options:

1. Ask someone who is more familiar with the instrument to tune it for you. (JWL would be glad to).

2. For those of you bold enough to do so- tune it yourself. This is done by putting in the 1-98 sample (be sure to see that it is spinning well and the sample is set at the right depth. If you start to tune the instrument and at any time you see no FID or a very unusual FID stop and report the problem to JWL.

i. Type in the command <SHIM> and follow the directions. It will first take a minute or two to find the correct gain. The system will then guide you in the tuning of the **Y** and **curvature** values on the potentiometers on the tuning tray. Follow the directions carefully. (If at any time you make a mistake in setting the pots; keep going and it will eventually correct your mistake but each mistake adds a couple of minutes to the time it takes very time you make a mistake.) The whole process takes about 2-7 minutes and the shim program will stop when you have tuned the instrument.

ii. Set the TMS zero by taking a H1 spectrum 1-98. Process through NUTS in the usual way. Note where the TMS line is in ppm. Go back to PNMR and use the command <fo>. It will ask for the TMS reading in ppm; then where you want it set (put in 0); then it will tell you where to set the **offset** potentiometer on the tuning tray. Make this adjustment and the next spectrum you take and process should have ppm scale set right.