

BARE BONES GUIDE TO DEPT & APT

on the Varian NMR Instruments

This guide is written assuming proficiency in basic operation of the Varian NMR instrument. You should be experienced in performing basic 1-dimensional NMR experiments before attempting to perform more advanced experiments on your own. Please ask for help the first time you perform this, to minimize your frustration (if for no other reason).

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Conventions in this manual:

Boldface text indicates commands to be typed at the computer

<angle brackets> are used to designate a key to be pressed (i.e. **<Ret>** for Return/Enter)

[square brackets] designate an icon/button in the VNMR menu to be *clicked*

Mouse Conventions: *click*, by default, refers to the Left Mouse Button.

LMB will be used to designate the Left Mouse Button

MMB will be used to designate the Middle Mouse Button

RMB will be used to designate the Right Mouse Button

Sometimes you will need to *hold*, rather than *click* the mouse button. This means that you should press and hold the button down throughout the operation.

Note: All **commands** in boldface assume that you press **<Return>** afterwards.

Considerations: The DEPT experiment can be done as a single 1D spectrum, using a *theta* pulse of 135-degrees. This so-called "DEPT-135" will generate a spectrum with CH & CH₃ signals positive, and CH₂ signals negative. Usually, this provided all necessary information. Alternatively, you can do a full, edited DEPT experiment; however, this requires that four (4) complete spectra be acquired and processed for subsequent editing. The full, edited DEPT will generate 4 spectra, one showing all protonated carbons, one CH-only, one CH₂ only, and one CH₃ only. The full edited DEPT wastes 50% of the instrument time used, but no thinking is required to interpret the results. The APT experiment is less sensitive than DEPT; however, it shows all carbons, edited by Even/Odd H-multiplicity (CH₀&CH₂ up, CH&CH₃ down).

I.: Quick, DEPT-135 Experiment:

- 1) Acquire a normal ¹³C NMR spectrum, using as many transients as needed for acceptable signal-to-noise.
- 2) Note the number of transients (NT or CT) used to acquire the normal ¹³C NMR spectrum.
- 3) With the 1D-Carbon spectrum displayed, type **DEPT <Ret>**.
 - a) Note: if you previously acquired the normal ¹³C spectrum, load the spectrum in the normal manner, and do **wft aph** to process and display the spectrum. Then type **su <Ret>** to setup the instrument for carbon-observe.
- 4) Set "nt" to ½ the value used to acquire the normal ¹³C spectrum.
- 5) Set "bs" to a small value (i.e. 16 or 32) so that you will be able to see the spectrum as the data is acquired.
- 6) Type **mult=1.5 <Ret>**.
- 7) Start the acquisition with **ga**.
- 8) After a block-size is completed, do **wft** to see the spectrum. Wait until "nt" scans are completed, or stop the acquisition with "sa" if it looks good enough.
- 9) Type **svf** to save your spectrum in your data directory. You can process/plot on a workstation, or on the instrument; however, it is always better to use the workstation for processing/plotting.
- 10) Phasing: Since some peaks will be positive and some will be negative, auto-phasing doesn't usually work.
 - a) Type **aph0** to phase only the zero-order term. This will result in the phase being close; however, the CH/CH₃ peaks might be inverted.
 - b) If the CH/CH₃ peaks are inverted (and CH₂ peaks are positive), type **rp?**, and note the value for "rp" at the top of the screen. Add or subtract 180 to the displayed value for rp, and type **rp=#** (where # is the value of rp displayed above). This will invert the entire spectrum.
 - c) Use manual-phasing (described in the Bare-Bones Guide for processing and plotting) to phase the CH/CH₃ signals positive, and CH₂ signals negative.

II.: Full, Edited DEPT:

- 1) Acquire a normal ^{13}C NMR spectrum, using as many transients as needed for acceptable signal-to-noise.
- 2) Note the number of transients (NT or CT) used to acquire the normal ^{13}C NMR spectrum.
- 3) With the 1D-Carbon spectrum displayed, *type* **DEPT <Ret>**.
 - a) Note: if you previously acquired the normal ^{13}C spectrum, load the spectrum in the normal manner, and do **wft aph** to process and display the spectrum. Then *type* **su <Ret>** to setup the instrument for carbon-observe.
- 4) Set “nt” to the ½ the value used to acquire the normal ^{13}C spectrum.
- 5) Set “bs” to a small value (i.e. 16 or 32) so that you will be able to see the spectrum as the data is acquired.
- 6) Start the acquisition with **ga**, and 4 spectra will be acquired using 4 values for the “mult” parameter (0.5, 1.0, 1.0, & 1.5). Four spectra will be acquired in the same experiment. The first contains all protonated carbon signals. The second and third are redundant, and contain CH carbon signals only. The fourth spectrum contains the “mult-1.5” data, with CH/CH₃ signals positive, CH₂ signals are negative.
- 7) Choose/Select the desired printer for output.
- 8) Type **autodept** to automatically process/plot the unedited spectra, edit the spectra, and plot the edited spectra.
- 9) To simply generate and plot the edited data, type **padept**.
- 10) Type **svf** , followed by your filename, to save the data. All 4 fids will be saved in the same filename.

III: APT

- 1) APT can be done instead of a normal ^{13}C , as all carbons are visible, but phase-edited according to multiplicity. The sensitivity is slightly less (~root-2) vs. normal 1D-carbon.
- 2) Setup for a normal 13C Observe: **[Nucleus,Solvent] su <Ret>**. Then type: **APT <Ret>**.
- 3) Set “nt” to a value appropriate for a normal ^{13}C spectrum... or pick a BIG number and just stop it when it is OK.
- 4) Be sure “bs” to a reasonable value (i.e. 16 or 32) so that you will be able to see the spectrum as the data is acquired.
- 5) Start the acquisition with **ga**.
- 6) After a block-size is completed, do **wft** to see the spectrum. Wait until “nt” scans are completed, or stop the acquisition with “sa(‘bs’)” if it looks good enough.
- 7) Follow the instructions above for DEPT-135 for processing and plotting, as you will have positive and negative peaks. (section I:(10)).

Quitting:

- a) Type **jexp1** , eject your sample, and re-insert the reference.
- b) Be sure to turn the spinner back on in **[Acqi]**.
- c) Lock and shim on the reference, and sign-out in the logbook.

-R.Shoemaker