^{13}C DEPT NMR

1. Distortionless Enhancement by Polarization Transfer is a polarization transfer technique used to observe nuclei with a small gyromagnetic ratio, which are J-coupled to ^{1}H. DEPT makes use of this polarization transfer to differentiate different types of ^{13}C signals [methyl (CH_3), methylene (CH_2), and methine (CH)]. Quaternary carbons are missing from DEPT spectra because the one-bond heteronuclear J-coupling ($^{1}J_{XH}$) is used for polarization transfer.

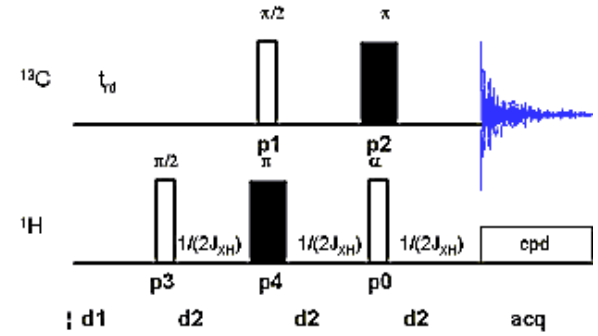
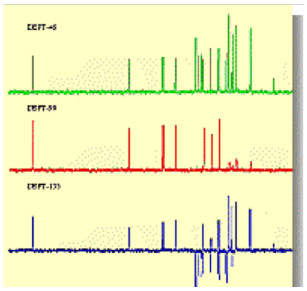


Figure 1: DEPT Pulse Sequence

2. DEPT may be run with or without ^{1}H-decoupling and it is relatively insensitive to the precise matching of delays with coupling constants, and so is much easier to use than the closely related INEPT sequence. DEPT, on the other hand, is more sensitive to pulse imperfections than INEPT.



DEPT45 CH, CH_2, CH_3 appear positive

DEPT90 only CH signals (appear positive)

DEPT135 CH and CH_3 appear positive. CH_2 appears negative

Figure 2: DEPT of cholesteryl acetate in $CDCl_3$.

3. The DEPT pulse sequence is shown in Figure 1. The final ^{1}H pulse with flip angle θ selects for the CH_3, CH_2 or CH signals. This angle is set to 45° in the DEPT-45 sequence, which yields spectra with positive CH, CH_2, and CH_3 signals; to 90° in DEPT-90, which yields spectra with only CH signals; and to 135° in DEPT-135, which yields spectra with positive CH and CH_3 signals and negative CH_2 signals. If you compare the standard ^{1}H-decoupled ^{13}C, and DEPT-45, -90, and -135 spectra, it is possible to determine which signals arise from primary, secondary, tertiary, and quaternary ^{13}C's (Figure 2).

4. The full DEPT experiment involves all three: DEPT45, DEPT90, & DEPT135. If the multiplicities of some of the signals are already known from previous structural information, one can in principal acquire only the DEPT135 and correctly phase the known signals. The unknown signals should then be either positive or negative depending on their multiplicities. While this is not complete information, one can usually distinguish CH's from CH_3's on the basis of the chemical shifts.

<u>^{13}C DEPT Data Acquisition</u>:

1) If you haven't already acquired a standard ^{13}C NMR spectrum, acquire one. Drag and drop the 13C dataset into the TopSpin window and type **iexpno** to create a new experiment with the same parameters as the carbon. You will need to change the

pulprog to the DEPT experiment you wish to run. The names of the pulse sequences are as follows:

a. **dept45** – DEPT-45 experiment
b. **dept90** – DEPT-90 experiment
c. **dept135** – DEPT-135 experiment

2) Calibrate your proton 90 degree pulse length. You can use **pulsecal**.

3) You may want to edit some acquisition parameters in **ased**.
   a. **cnst2 = 145** (an "average" value for a one-bond C-H J-coupling constant).
   b. **p3** = the 90 degree pulse you calibrated in step 2.
   c. Change **ds** = 8.
   d. The DEPT experiment enhances your NMR signal, so you can use fewer scans **ns** than you did for your standard ^{13}C NMR.

4) Make sure that you are tuned to both proton and carbon. Type **zg** to start your data acquisition. If you set up three experiments in a row (e.g., EXPNO 3-5 are DEPT45, 90, and 135), you can use the command **multizg 3** in the active window of the first experiment.

^{13}C DEPT Data Processing:

1) Process with your data and plot the spectra so the negative peaks can be seen. The DEPT45 can be used to determine the phase to be used for the rest of the DEPT spectra. The phase constants of the DEPT45 are applied to the DEPT90 and DEPT135 spectra with the pk command (usually in the combined efp command).
   a. For DEPT45: you can use **ef; apk; abs n**. Note the **phc0** and **phc1** values.
   b. For DEPT90 & DEPT135: set **phc0** and **phc1** to the same value as in your DEPT45 experiment. Then use the **efp** and **abs n** commands.

2) If the multiplicities of some of the signals are already known from previous structural information, you can in principal acquire only the DEPT135 spectrum and correctly phase the known signals. The unknown signals should then be either positive or negative depending on their multiplicities. While this is not complete information, one can usually distinguish CH's from CH_3's on the basis of the chemical shifts.