

Due at the *beginning* of lab, the week of 4 Nov 2008

Use Athena (AC-300) only for this lab.

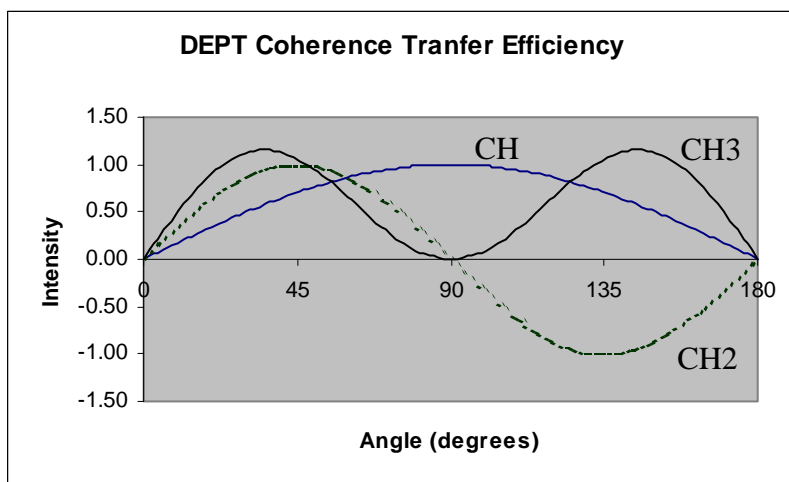
Reading and reference materials for this lab:

- **Chap 16 of BUG: “Polarization Transfer Experiments (INEPT and DEPT)”**
- Review **GO** statement from page 1 **Chap 2 of BUG “Bruker AM/AC Acquisition Basics”**
- **Section 4.4 of Claridge**

In this lab, you will be guided through the normal setup and acquisition sequences for DEPT and INEPT experiments.

- DEPT generates a less distortion when a number of different J -couplings exist in the sample; this situation is common in organic compounds, where aliphatic carbons will have a one-bond $^1J_{\text{CH}} \sim 110\text{-}130\text{Hz}$ for aliphatic carbons, and $^1J_{\text{CH}} \sim 145\text{-}160\text{Hz}$ for aromatic carbons.
- INEPT can provide somewhat better sensitivity for experiments where a single J -coupling value is known; e.g., ^{29}Si spectra of a compound with one silicon nucleus. Otherwise, the DEPT and INEPT experiments are quite similar:
- DEPT is the preferred experiment for ^{13}C NMR in all routine situations.
- INEPT is used when J -couplings are small, or when MW or viscosity (e.g., temp is close to freezing point of solvent) are large.

Both experiments (but most commonly DEPT) can be used for “spectrum editing” or “multiplicity analysis.” The editing is based on the phase of the ^{13}C nucleus resulting from the *number* of attached protons, as shown in the figure below. Angle refers to the final *nutaton* of the magnetization, and is set with a pulse length (here **P0**) being 1/2, 1 or 3/2 times the 90° pulse in the experiment (**P1**), leading to DEPT-45, -90 or -135 spectra respectively. INEPT spectra can be run similarly, but rely on a change in delay (**D3**) rather than nutation.



See the BUG notes listed above for a good discussion about the practical aspects of setting up and running both techniques. In brief, the setup follows:

- read in a DEPT jobfile
- enter **II**
- check and adjust parameters (using **AS DEPT.AU** or **AS INEPTRD.AU**), especially:
 - **D1** = $2-3 \times T_1(^1H)$, typically ~2s for non-deoxygenated solvents and normal organic compounds
 - **D2** = $1/(2J_{CH})$ for DEPT, or $1/(4J)$ for INEPTRD (*D2 must be changed from the parameter value for INEPTRD!*)
 - **P0** = $\epsilon \times P1$ where $\epsilon = 0.5, 1, 1.5$ for DEPT-45, -90, -135, respectively, or
 - **D3** = ϕ / J_{CH} where $\phi = 1/8, 1/4, 3/8$ for the INEPT analogs of DEPT-45, -90, -135, respectively
- **RD** and **PW** must = 0 for these sequences to work correctly
- use **AU DEPT.AU** or **AU INEPTRD.AU** to acquire the data; **ZG** will initiate a GO, and will produce only noise.

Acquisition of DEPT and INEPTRD Spectra

1. Setup and acquire a standard ^{13}C spectrum with CPD decoupling of your unknown sample. Acquire research quality data: for ^{13}C spectra, this primarily involves taking sufficient scans such that all carbon peaks are unambiguously observed above the noise (i.e., have signal-to-noise ≥ 3). $NS \leq 128$ should be sufficient.
2. Acquire a DEPT-135 using the standard DEPT jobfile. The θ pulse in a DEPT experiment is set by the parameter **P0** in DEPT.AU. Make certain **P0** is set correctly for a DEPT-135 (**P0=1.5×P1**). Use the same number of scans as used for the ^{13}C spectrum. Run the experiment with **AU DEPT.AU**.
3. Change **P0** (=P1) to acquire a DEPT-90. This spectrum is often not needed in research; it is run if $>CH-$ and $-CH_3$ moieties are difficult to differentiate from one another in the DEPT-135.
4. You can optionally acquire a DEPT-45 spectrum (**P0=0.5×P1**). This is usually a superfluous spectrum, adding no information content to the $^{13}C\{^1H\}$, DEPT-135 and DEPT-90 spectra.
5. Setup and acquire an INEPT experiment (use **AS INEPTRD.AU** and **AU INEPTRD.AU**) on the same sample where $-CH_2-$ moieties are inverted compared to $-CH_3$ and $>CH-$ moieties.
6. Perform a buffers (**BU**) stack plot of the $^{13}C\{^1H\}$ spectrum plus the 2 (or 3) DEPT spectra onto a single page. List the values of **P0** used for each of the DEPT spectra.
7. Perform a dual plot of the ^{13}C standard spectrum plus the INEPT spectrum onto a single page. List the value of **D2** and **D3** used for the INEPT spectrum.

Hand in 2 plots