

The DEPT Experiment

The name DEPT stands for *Distortionless Enhancement by Polarization Transfer*. The DEPT experiment is a useful 1D NMR experiment that provides information on the number of protons attached to the various ^{13}C resonances in a carbon NMR spectrum. In the DEPT experiment we use two rf transmitters, one to apply pulses to the ^1H spins and one to apply pulses to the ^{13}C spins. We acquire the ^{13}C signal during the FID time period. During the ^{13}C acquisition, the ^1H transmitter is used for broad-band (BB) decoupling to remove the splitting of ^{13}C signals by attached protons. The pulse sequence for the DEPT is given below:

^1H : D1 - 90° - tau - 180° - tau - 45° (90°, 135°) - tau - BB decouple
 ^{13}C : 90° 180° FID

It is clear that to set up the parameters for this experiment we will have to know the 90° pulse for both the ^1H and ^{13}C transmitters. For the proton channel, it is important to note that we are using the transmitter that is normally used to do decoupling and not the one that is used for routine proton acquisitions. Therefore, we need to input the 90° pulse for the decoupling channel. The 90° ^1H pulse using the decoupler transmitter is likely to be different than the 90° ^1H pulse for the observed nuclei transmitter.

Results of the DEPT experiments (quaternary carbons are null in all spectra):

DEPT 45: CH, CH₂, CH₃ positive.

DEPT 90: CH positive, CH₂, CH₃ null (often a small residual signal is seen).

DEPT 135: CH, CH₃ positive, CH₂ negative.

Some of the important parameters are:

pw 90° pulse on ^{13}C , (at indicated transmitter power - *tpwr*)
pp 90° pulse on ^1H using the decoupler transmitter (at indicated decoupler transmitter power - *pplvl*)
mult an arrayed parameter which is used to set the proper final ^1H pulse width (this pulse is often referred to as the *pw* pulse) for each of the three DEPT experiments (DEPT 45, 90, 135). **mult** = (0.5, 1, 1.5). For the DEPT 45, the final ^1H pulse is **pp** * 0.5 (the first number in the **mult** array) = 45° pulse.
d1 relaxation delay to allow system to return to equilibrium between runs through the pulse sequence.
nt number to transients (scans) per FID. Must be a multiple of 4.
tau tau is the delay between the various pulses which allow the system to *evolve* properly to achieve the desired results. This value should be equal to $1/(2*J)$, where J is the appropriate average scalar coupling constant between the protons and the ^{13}C nuclei. Typical ^1H - ^{13}C coupling constants are on the order of 140 Hz. Tau is not a parameter that is entered by the user but rather, is calculated by the computer based on the parameter **j** that is entered by the user. The default value for **j** is 140. Note that **j** = 140 may not be the optimum value for your molecule, but it should give reasonable results in most cases (e.g. for menthol, **j** = 125 is a better value).

As of 4-19-02, the 90° pulses on the two transmitters are as follows:

^{13}C : 14.5 μs (at *tpwr* = 50)

^1H (decoupler transmitter): 15.7 μs (at *pplvl* = 59)

Step-by-Step Instructions:

Note: this guide assumes the user is familiar with the running of routine ^{13}C NMR spectra.

1. Run quick carbon NMR spectrum and expand around the peaks of interest. When processing the data for this and the DEPT spectra, you may want to use quite a bit of line broadening to increase the s/n. Recommended is **lb** = 1 or 2.

2. After expanding around the peaks of interest, reset the spectral window to reflect this new window. To do so, enter the command *movesw*. Now, reacquire your spectrum with the new *sw*; phase the spectrum and set the reference peak (e.g. the chloroform triplet to 77.0p). SAVE this spectrum as your regular carbon spectrum. [Note, you do **not** have to change the spectral window if you don't want to. In that case, just run the normal ¹³C NMR spectrum and save it, as described].
3. Move the ¹³C parameters that you just used to another experiment. Enter *mp(1,2)*, if you are moving the parameters from *exp1* to *exp2*. Next, join the experiment to which the parameters were transferred; to do this enter *jexp2* (or use the appropriate buttons).
4. Load the DEPT experiment "on top of" your ¹³C parameters. Enter *dept*.
5. Check that the parameters are correct (enter *dg*; see above for a description of the important parameters). You want *d1* = 1-3*T₁ (T₁ for protons attached to a ¹³C nucleus - these are shorter than the T₁'s for protons attached to the corresponding ¹²C nuclei - about 2-3s should be fine), *nt* = number of scans required for decent s/n.
6. To check the time required for the DEPT, enter *time*. If the time required is not satisfactory, adjust *d1* or *nt*, appropriately.
7. If you would like your DEPT to be automatically saved to a file after the experiment is complete (recommended if you are going to be leaving the instrument!), you need to set the parameter *wexp*, which tells the computer what to do after the acquisition is complete.
 - a. Set *wexp* to save the file by entering: *wexp='svf(n1)'*. This tells the computer to save the fid to a file designated by the string *n1*.
 - b. Now you just need to define *n1*. You will want *n1* to include the filename for your dept, and you also need to tell the computer *exactly* where to save the data, so the whole absolute path must be specified. So, define *n1* as follows: *n1='/export/home/your account(probably advlab)/your directory/filename'*. It is very important that you type these commands accurately, with the proper "punctuation", i.e. the single quotes. If it is not all correct, the experiment will not be saved! However, the dept will still be stored in the experiment you are working in, until someone overwrites it.
8. Enter *au* to start the acquisition.

Processing the data (load and transform the DEPT spectrum in *exp2*, if it isn't already displayed):

9. Load the DEPT spectrum, if its not already loaded, and display the DEPT 45; enter *ds(1)*. Make sure this is phased (*aph*) so that all peaks are positive. You should be looking at the same spectral width you set in your ¹³C spectrum in *exp1* (if, of course, you are looking at the FULL spectrum). If you *do not* change this expansion at all now, you can plot your DEPT spectra above your full ¹³C spectrum in a stacked plot and they will all line up with each other. So, don't do any expansion of the DEPT plot!
10. Set *vp = 35* to leave room for the regular ¹³C spectrum to be plotted under the DEPT spectra. Adjust the vertical scale so that there will be "room" above the spectrum for the Dept 90 and Dept 135 spectra to be displayed.
11. Enter *dssa* to display the stacked plot.
12. Enter *pl('all')* to plot the DEPT spectra.
13. Enter *jexp1* to go back to the ¹³C spectrum. Type *ds* to re-display it (or load and transform it, if it isn't already in *exp1*).
14. Enter *pl* to plot this spectrum under the DEPT's. Make sure the vertical scale is set so plot fits below the DEPT spectra (*vs = 35*). Enter *pscale* to put a scale on the spectrum and *page* to print out your plots.
15. Obtain a printout of the DEPT parameters, if you want. Join exp 2 (*jexp2*) and enter the following: *printon dg da printoff*. *dg* displays the parameters and *da* displays the values of the arrayed parameter, *mult*.