

Instructions for Obtaining ^{13}C DEPT Spectra on the Bruker AM/AC Spectrometers

Background Information:

The DEPT technique allows one to quickly identify carbon atoms according to the number of directly attached protons. There are three DEPT experiments, DEPT-45, DEPT-90 and DEPT-135. The DEPT-45 experiment shows all of the protonated carbons with positive phase, and the DEPT-90 experiment shows only the -CH- carbons, with positive phase. The DEPT-135 experiment shows -CH₃ and -CH- carbons with positive phase and -CH₂- carbons with negative phase. Quaternary carbons do not appear in any DEPT spectrum. Therefore, a standard ^{13}C spectrum, a DEPT-90 spectrum and a DEPT-135 spectrum together will allow one to determine the types of all of the carbons in a molecule. The DEPT-45 experiment is useful because its sensitivity is about twice that of a standard ^{13}C experiment, making DEPT-45 the ^{13}C experiment of choice if only a limited amount of sample is available.

NOTE: These instructions assume the user is checked out for ^1H and ^{13}C operation on the Bruker spectrometers. The instructions below are in the same format as the basic ^1H and ^{13}C instructions, and assume that the reader is familiar with those instructions and basic spectrometer operation.

Note: = return key (orange), _ = spacebar, ^ = "CTRL" key (orange).

Acquisition of ^{13}C DEPT-135 spectrum.

1. Acquire a proton spectrum in job 1.
2. Acquire a carbon-13 spectrum in job 2.

It is important to check the proton spectrum before investing time in any carbon-13 spectra.

Usually, any obvious sample problems will be apparent after running a proton spectrum and this can save one wasting time running carbon-13 and/or DEPT spectra on a bad sample.

3. 3 (go to job 3 to run DEPT)
4. RJ_DEPTDUAL.XXXX (read DEPT-135 acquisition and automation parameters)
where XXXX = ACET, CDCL, D2O, DMSO, METH
5. PJ (read processing and plotting parameters)
6. AS_DEPT.AU (load up DEPT pulse program - wait until green text appears)
7. ^Q (quit pulse program listing - wait until parameter list starts)

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|-----|--------|---|
| 8. | ^Q | on LCD display)
(quit parameter input -
screens should look normal now) |
| 9. | II | (initialize interfaces) |
| 10. | NS 256 | (change number of scans, if
necessary; use a multiple of 16) |
| 11. | AU | (start DEPT acquisition - this will
give a DEPT-135 spectrum [CH,
CH ₃ up, CH ₂ down] by default) |

Acquisition of ¹³C-DEPT-45,90 spectra.

Use the same steps as above, except that prior to step 11, the value of P0 must be changed. In pulse-programming parlance, P0 is a pulse applied at the proton frequency that must be of the duration necessary to effect a 45, 90, or 135 degree rotation of the proton magnetization, to produce the corresponding ¹³C DEPT spectrum. The necessary pulse duration (in microseconds) for a 90 degree rotation (a "90-degree decoupler pulse") is given on the chart located on the console by the keyboard.

On the example shown for the AM-400, the 90-degree decoupler pulse is 12.0 microseconds (us). Therefore, to run a DEPT-90 on the AM-400, P0 needs to be set to 12 us, i.e.,

10a. P0 12.0u (no)

before typing AU . For a DEPT-45 P0 should be set to 6.0u, and to 18.0u for a DEPT-135. Note that for the AC-200 different values will be needed. DO NOT just type in 45, 90, or 135 for P0 - it will not work! Note that P0 is P-zero, NOT PO.

Processing the FID.

Processing is the same as for normal carbon-13 spectra, except that the DEPT-135 spectrum will have both positive and negative peaks - don't try to phase them all positive!

Plotting the spectrum.

Again, plotting is the same as for normal carbon-13 spectra, except that for DEPT-135 the spectrum baseline has to be moved to the center of the page to show both positive and negative peaks. This is done by default in the DEPTDUAL parameter sets by setting CY to 10 cm and the offset parameter in the DPO (display plotter options) routine to -10. If you run a DEPT-45 or DEPT-90 starting from the DEPTDUAL

parameter set, you should change CY to 20 before acquiring data (e.g. CY 20) and set the plot offset in DPO to the normal value of -2 (e.g. DPO -2 ^Q).

Transferring FID's to observe spectrum during acquisition.

DEPT FID's can be transferred just like normal carbon-13 FID's. BUT, when you transfer to a new job, the old data in the new job is lost.

When you are finished acquiring spectra.

Finish up as usual. Remember to re-initialize (II) back to proton in job 1.