CHEM / BCMB 4190/6190/8189

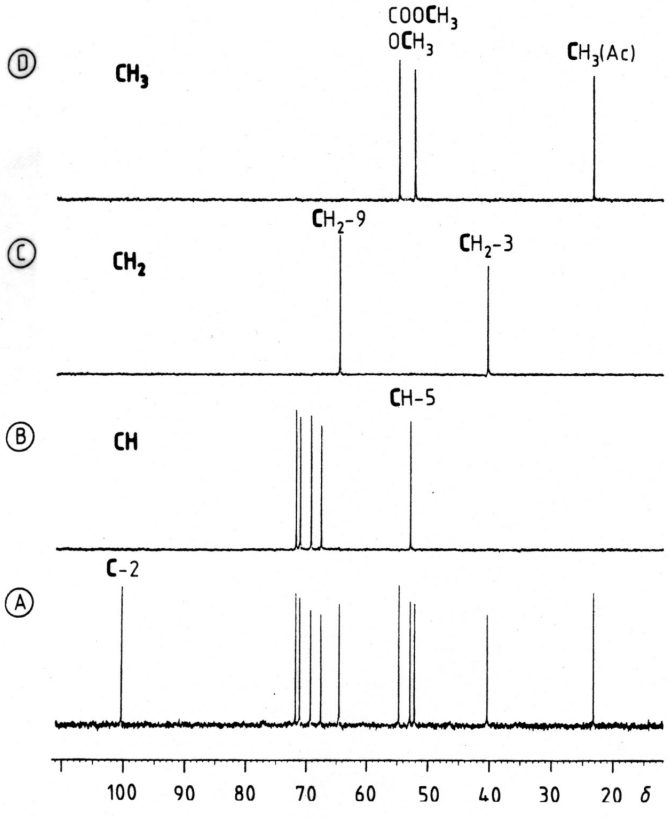
Introductory NMR

Lecture 13

The DEPT Experiment

Interpreting ^{13}C NMR spectra:

- **Very useful to know which signals belong to quaternary carbons, CH, CH2, and CH3**
- **J-modulated spin-echo experiment and refocused INEPT are useful but don't provide all the necessary information**
- **The DEPT experiment (Distorsionless Enhancement by Polarization Transfer) is now one of the most important techniques available for interpreting 1D ^{13}C NMR spectra**



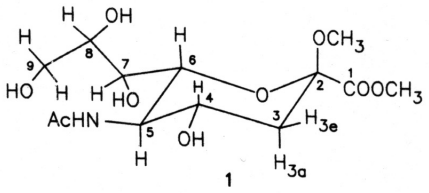


Figure 8-22.
The DEPT experiment; for pulse sequence see text.
A: 100.6 MHz ^{13}C NMR spectrum of the neuraminic acid derivative **1** with ^{1}H BB decoupling (δ = 10 to 110 region only).
B: CH sub-spectrum: DEPT(90).
C: CH_2 sub-spectrum: DEPT(45) − DEPT (135).
D: CH_3 sub-spectrum: DEPT(45) + DEPT(135) − 0.707 DEPT(90).
(Experimental conditions: 167 mg of the compound in 2.3 ml D_2O; 10 mm sample tube; 16 K data points; 32 FIDs for $\Theta_1 = 45°$ and $\Theta_3 = 135°$, 64 FIDs for $\Theta_2 = 90°$; τ = 3.57 ms; total time approx. 12 min.)

Pulse Sequence

^{1}H channel:

$$90^{\circ}_{x'} - \tau - 180^{\circ}_{x'} - \tau - \Theta_{y'} - \tau - \text{BB decoupling}$$

^{13}C channel:

$$90^{\circ}_{x'} - \tau - 180^{\circ} - \tau - \text{FID } (t_2)$$

- **τ is 1/2J_{CH} with J_{CH} = 140 Hz**
- **is chosen to be $\Theta = 45^{\circ}$, $\Theta = 90^{\circ}$, $\Theta = 135^{\circ}$**
- **Vector diagrams are not adequate to explain the effect of this pulse and to understand this pulse sequence.**
- **The following curves explain the effect of Θ on the intensities of signals**

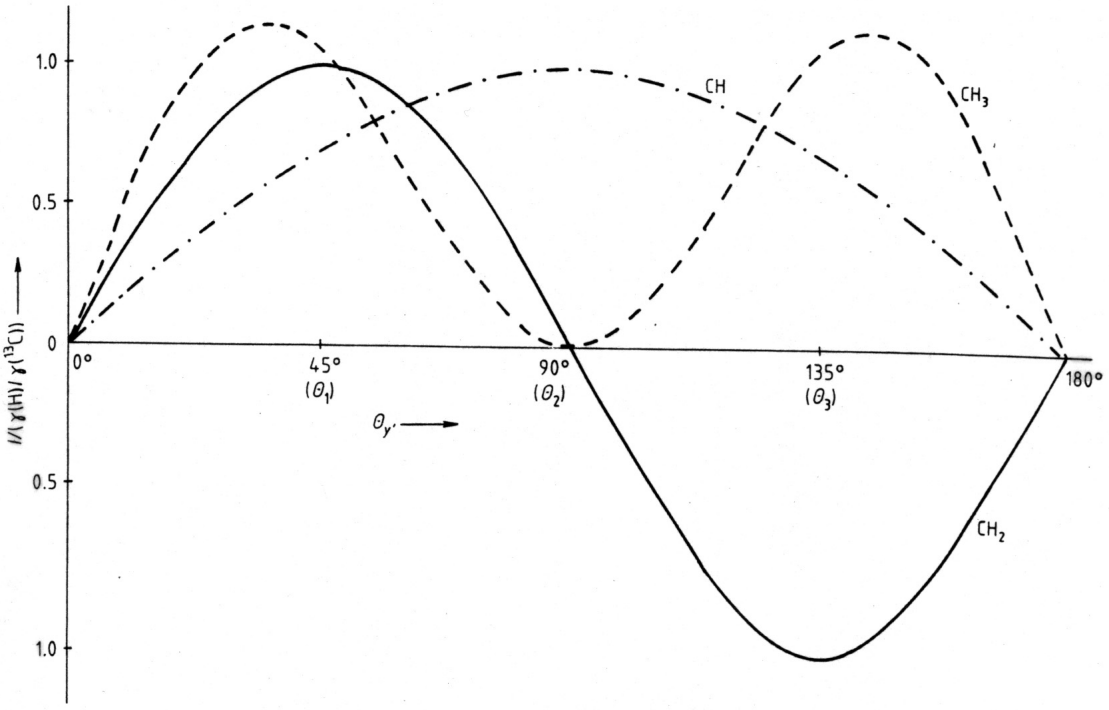


Figure 8-23.
DEPT experiment. Curves calculated from Equation (8-6) for the intensities of CH, CH_2 and CH_3 signals as functions of the pulse angle $\Theta_{y'}$; CH ·–·–·–·–, CH_2: ————, CH_3: – – – – – –.

- **CH sub-spectrum: $\Theta 2 = 90^{\circ}$ or DEPT (90)**
- **CH_2 sub-spectrum: DEPT(45) - DEPT(135)**
- **CH3 sub-spectrum: DEPT(45) + DEPT(135) - 0.707DEPT(90)**
- **To compare absolute intensities the DEPT(90) must be collected with twice as many scans as for the other two DEPT experiments**

Practically, it is sufficient to carry out two experiments, DEPT(90) and DEPT(135)

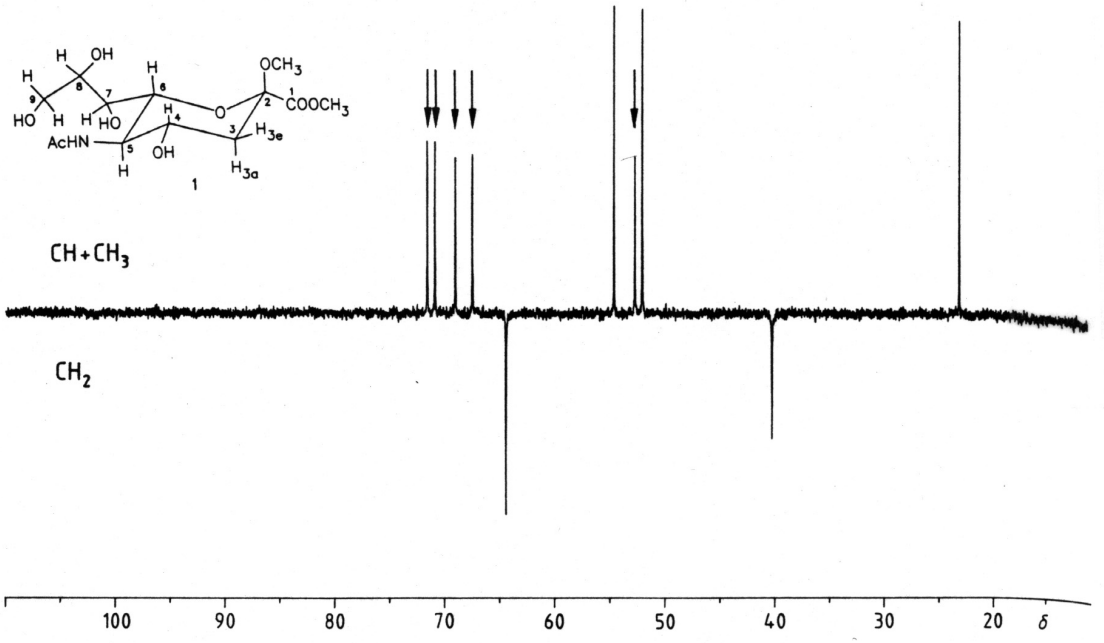


Figure 8-24.
DEPT(135) spectrum of the neuraminic acid derivative **1**, recorded using the pulse sequence given in the text, with $\Theta_{y'} = 135°$. The signals of the five CH groups, identified with the help of the DEPT(90) spectrum ($\Theta_{y'} = 90°$), are marked by arrows. The other three positive signals arise from CH_3 groups, and the two negative signals from CH_2 groups.
(Experimental conditions:
20 mg of the compound in 0.5 ml D_2O; 5 mm sample tube; 32 K data points; 300 FIDs; $\tau = 3.57$ ms; total time approx. 20 min.)

Table 8-2.
Partial assignment of the ^{13}C NMR signals of **1** from the results of the DEPT experiment.

δ	CH_3	CH_2	CH	C	Assignment
23.2	×				CH_3 (Ac)
40.31		×			C-3
52.12	×				
52.83			×		C-5
54.65	×				
64.50		×			C-9
67.51			×		
69.18			×		
70.98			×		
71.67			×		
100.32				×	C-2
171.50[a)]				×	
175.93[a)]				×	

[a)] Values from the complete spectrum (Fig. 8-12 A).