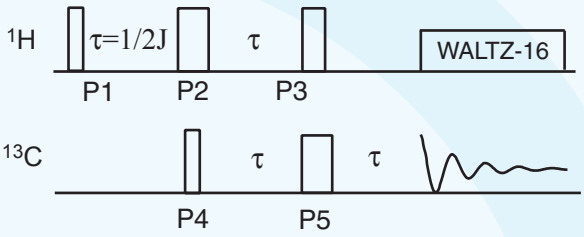
Editing ^{13}C NMR Spectra with DEPT

1. Introduction

The Distortionless Enhancement by Polarization Transfer (DEPT) sequence uses a polarization transfer from protons to a nucleus X over one chemical bond to increase the signal strength. The experiment is also used for distinguishing CH_3, CH_2 and CH groups. For molecules containing a large number of carbon atoms it desirable to generate separate subspectra for CH_3, CH_2 and CH groups. Described here is the sequence editing procedure to obtain three subspectra. This example demonstrates the basic procedure of double resonance 1D NMR data acquisition and processing on Tecmag spectrometers.

2. Pulse sequence

(i)

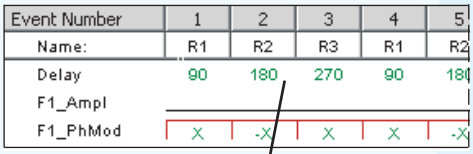


WALTZ-16 = $RR\bar{R}\bar{R}\ \bar{R}RR\bar{R}\ RR\bar{R}\bar{R}\ R\bar{R}\bar{R}R$,
$R = 90_x180_{-x}270_x$

(ii)

Pulse width and phase cycle:

P1 (H90°): 0
P2 (H180°): phH2 = 0, 2, 1, 3.
P3 (H45°/90°/135°): phH3 = $(1)_4$, $(3)_4$.
P4 (C90°): phC1 = $(0)_8$, $(1)_8$, $(2)_8$, $(3)_8$.
P5 (C180°): phC2 = $(0, 2)_4$, $(1, 3)_4$.
Receiver: phrx = $(1)_2$, $(3)_4$, $(1)_2$,$(2)_2$, $(0)_4$, $(2)_2$, $(3)_2$, $(1)_4$, $(3)_2$, $(0)_2$, $(2)_4$, $(0)_2$.
(phH2, phH3, phC1, phC2 phRX are 1D phase tables. All tables are in 4 step mode.)



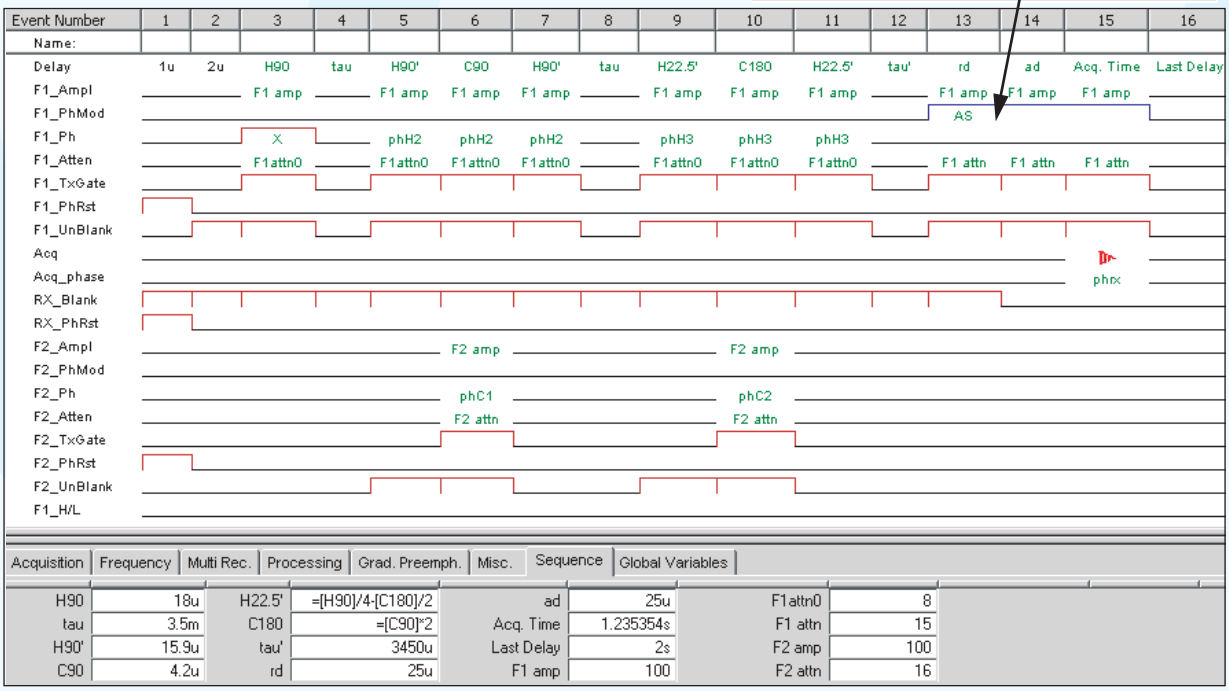


Fig. 1. (i) The editing ^{13}C DEPT sequence with WALTZ-16 sequence for ^{1}H decoupling. (ii) The sequence in the NTNMR sequence editor. Three different pulse widths for P3 (45°, 90° or 135°) yield in three spectra: a) P3 = 45°, all peaks are positive; b) P3 = 90°, only the peaks of CH groups appear; c) P3 = 135°, the peaks of CH and CH_3 are positive, and those of CH_2 are negative.

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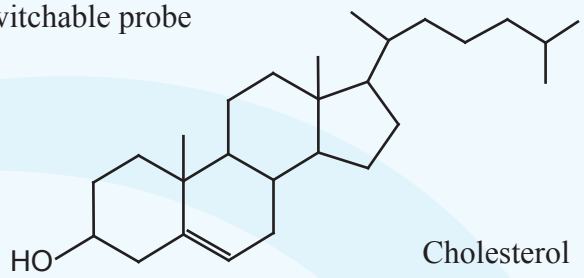
3. Experiment

Sample: Cholestrol in $CDCl_3$ (50mg/ml)
Spectrometer: 7 Tesla Magnet with Tecmag HF3 discovery
Probe: Nalorac D300-5 OWB 5mm $^1H/^{13}C$ Switchable probe
^1H hard pulse: 13.9 kHz (90^o = 18 µs @ 5W)
^1H decoupling field: 5.6 kHz (90^o = 45 µs @ 800 mW)
^{13}C hard pulse: 59.5 kHz (90^o = 4.2 µs @ 250 W)
τ: 3.5 ms (= $1/(2J_{C,H})$ = 140 Hz)
Spectrum width: ± 6.5kHz
Recycling time: 2s
Number of scans: 512



Cholesterol

Notes:

1. Before editing the sequence (Fig. 1b), calibrate the 90^o pulse widths of ^1H and ^{13}C using the nutation experiment (see note, "One Pulse Experiment and Pulse Calibration").
2. Set up the WALTZ sequence according to the note, "^{13}C NMR Spectra with ^1H WALTZ Decoupling".
3. The center of pulses P2 and P4 (also P3 and P5) should be aligned. Since P2 > P4 (and P3 > P5) P2 (and P3) have to split into 3 pulses. The delay of P2's (and P3's) middle pulse equals to P4 (and P5), and the delay of both sides is (P2 - P4)/2 [and (P3 - P5)/2]. The middle pulse of P2 (and P3) falls on the same event as P4 (and P5).

4. Results

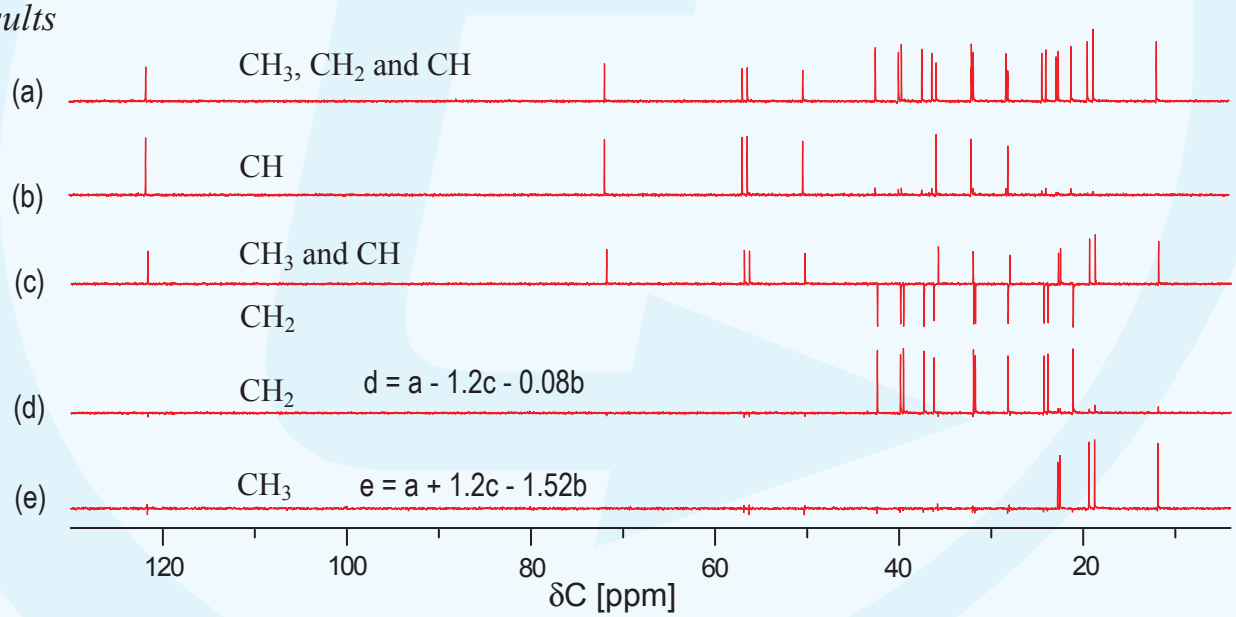


Fig. 2. The Editing DEPT ^{13}C spectra of cholestrol in $CDCl_3$. Spectra a, b, and c are correspondant to P3 = 45^o, 90^o, and 135^o in the sequence shown in Fig.1. Spectra d and e are calculated using spectra a , b and c and the equations shown in the figure. The calculation is done by a script, "Editing DEPT".

5. References

1. M.R. Bendall, D.M. Doddrell, D.T.Pegg, *J. Am. Chem. Soc.* **1981**, *103, 4603-4605.*
2. D.M. Doddrell, D.T.Pegg, M.R. Bendall, *J. Magn. Res.* **1982**, *48,* 323-327.
3. S. Braun, H.-O. Kalinowski, S. Berger, "150 and More Basic NMR Experiments", Wiley-VCH, 1999, 183-184.

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