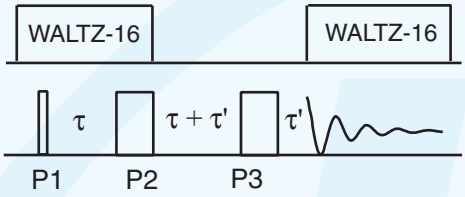
1. Introduction

The Attached Proton Test (APT) experiment is a common way to assign C-H multiplicities in ^{13}C NMR spectra. It provides the information on all sorts of carbons within one experiment. Depending on the number of hydrogens bound to a carbon atom, n, CH_n spin vectors evolve differently after the initial pulse. If the delay τ is set to 1/J, CH and CH_3 vectors have opposite phases compared to C and CH_2. Therefore, the phase of CH and CH_3 peaks is 180^o different from C and CH_2 peaks. This example demonstrates the basic procedure of double resonance 1D NMR data acquisition and processing on Tecmag spectrometers.

2. Pulse sequence

(a)



WALTZ-16 = $R\bar{R}\bar{R}\bar{R}\ \bar{R}RRR\ R\bar{R}\bar{R}R\ \bar{R}\bar{R}RR$,

$R = 90_x180_{-x}270_x$

$\tau = 1/J$

$\tau' = ad + rd;$

(b) Pulse width and phase cycle:

P1 (C45^o): ph0 = 0, 0, 2, 2, 1, 1, 3, 3.
P2 (C180^o): ph1 = 1, 1, 3, 3.
P3 (C180^o): ph2 = 1, 3, 1, 3.
Receiver: ph0

(All entries are in 4 step mode.)

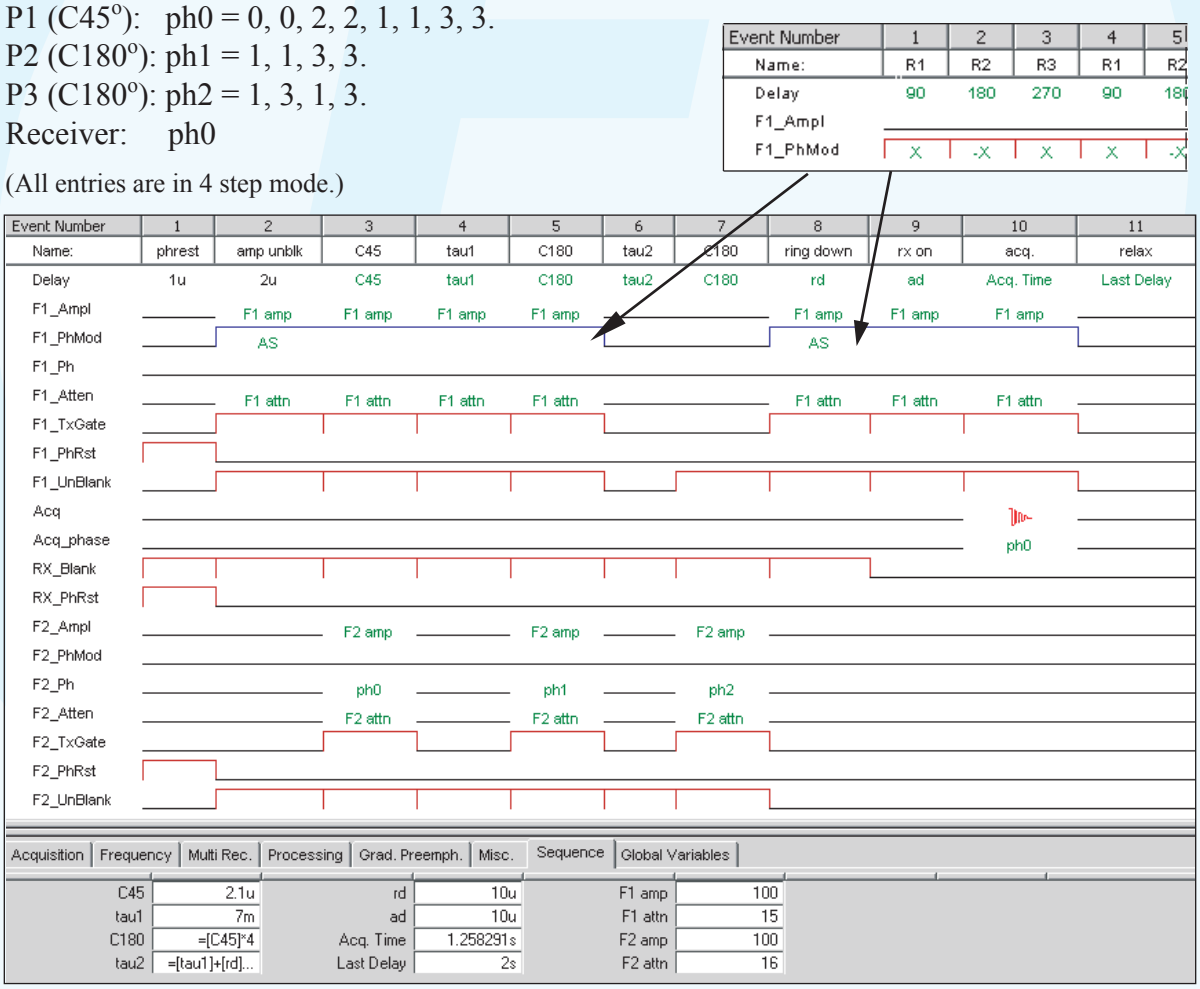
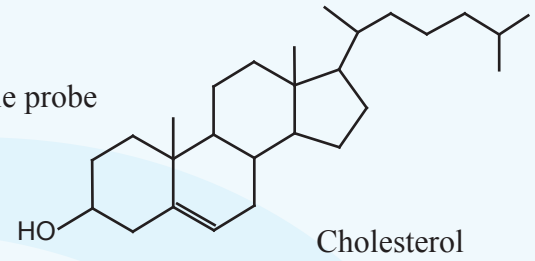


Fig. 1. (a) The^{13}C APT sequence with WALTZ-16 ^{1}H decoupling. (b) The sequence in the NTNMR sequence editor. The WALTZ-16 sequence is loaded using the asynchronous function.

3. Experiment

Sample:	Cholesterol in $CDCl_3$ (50mg/ml)
Spectrometer:	7 Tesla Magnet with Tecmag HF3 discovery
Probe:	Naloac D300-5 OWB 5mm $^{1}H/^{13}C$ Switchable probe
^{1}H decoupling field:	5.6 kHz (90° = 45 µs @ 800 mW)
^{13}C hard pulse:	59.5 kHz (90° = 4.2 µs @ 250 W)
τ:	7 ms (= $1/J_{C,H}$ = 140 Hz)
SW +/-:	± 6.5kHz
Last Delay:	2s
Scans 1D:	512



Cholesterol

Notes:
Before editing the sequence (Fig. 1b), you have to calibrate the 90° pulse width of ^{1}H and ^{13}C using the nutation experiment (see notes, "One Pulse Experiment and Pulse Calibration" and set up the WALTZ sequence according to the notes, "^{13}C NMR Spectra with ^{1}H WALTZ Decoupling".

4. Results

Figure 2 shows the ^{13}C APT spectrum of cholesterol in $CDCl_3$ acquired with the sequence introduced in Fig.1. Note that the signals of CH and CH_3 are positive, but CH_2 and quarternary carbons including the solvent carbon are negative.

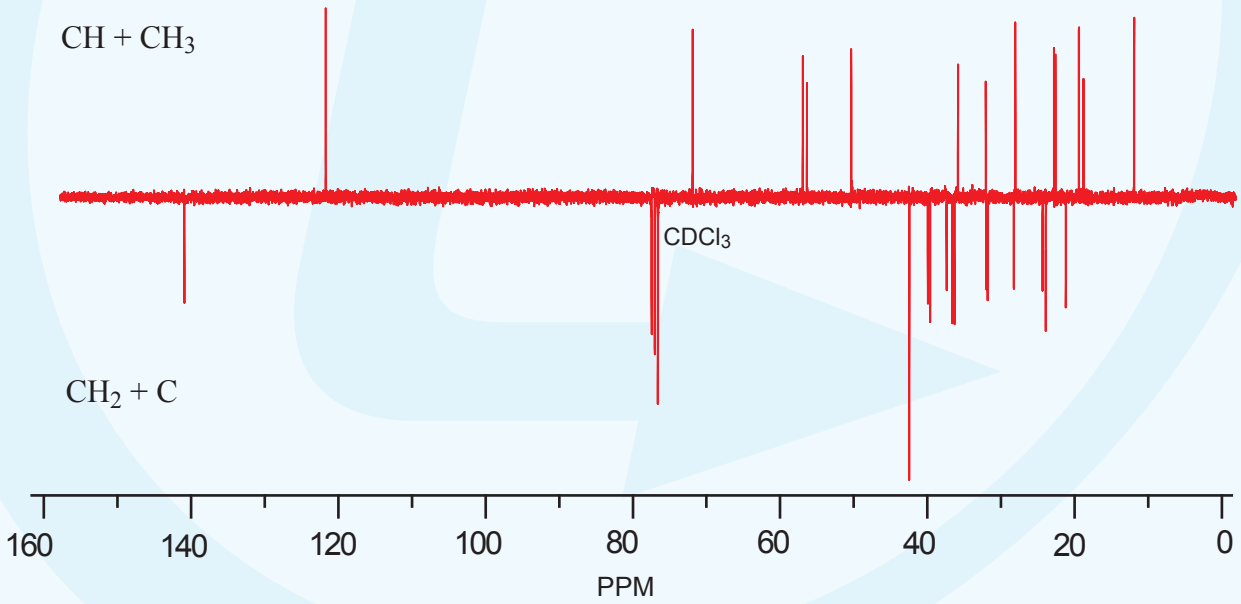


Fig. 2. The ^{13}C APT spectrum of cholesterol in $CDCl_3$ using the sequence shown in Fig.1.

5. References

1. S.L. Patt, D. Ruben and J.N. Shoolery, *J. Magn. Reson.* **1982**, *46*, 535-539.
2. H. J. Jakobsen, O.W. Sorensen, W.S. Brey and P.J. Kanyha, *J. Magn. Reson.* **1982**, *48*, 328.
2. A.M. Torres, T.T. Nakashima, and R.E.D. McClung, *J. Magn. Res. Ser. A* **1993**, *101*, 285-294.
3. S. Braun, H.-O. Kalinowski, S. Berger, "150 and More Basic NMR Experiments", Wiley-VCH, 1999, 165-167.

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