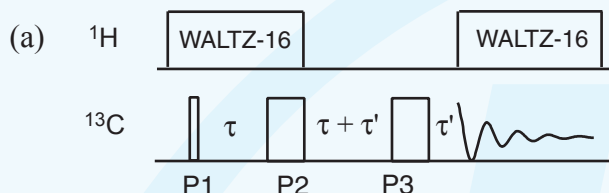


1. Introduction

The Attached Proton Test (APT) experiment is a common way to assign C-H multiplicities in ¹³C 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 CH₃ vectors have opposite phases compared to C and CH₂. Therefore, the phase of CH and CH₃ peaks is 180° different from C and CH₂ peaks. This example demonstrates the basic procedure of double resonance 1D NMR data acquisition and processing on Tecmag spectrometers.

2. Pulse sequence



WALTZ-16 = RR $\bar{R}\bar{R}$ \bar{R} RR \bar{R} R $\bar{R}\bar{R}$ \bar{R} RRR
 $R = 90_x 180_{-x} 270_x$

$\tau = 1/J$
 $\tau' = ad + rd$

(b) Pulse width and phase cycle:

P1 (C45°): ph0 = 0, 0, 2, 2, 1, 1, 3, 3.

P2 (C180°): ph1 = 1, 1, 3, 3.

P3 (C180°): ph2 = 1, 3, 1, 3.

Receiver: ph0

(All entries are in 4 step mode.)

Event Number	1	2	3	4	5
Name:	R1	R2	R3	R1	R2
Delay	90	180	270	90	180
F1_Ampl					
F1_PhMod	X	-X	X	X	-X

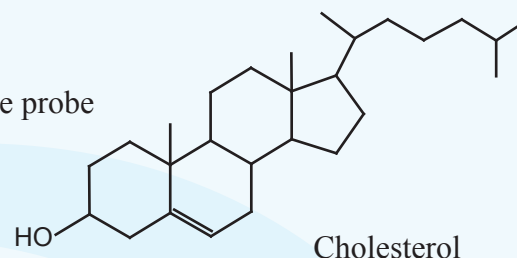
Event Number	1	2	3	4	5	6	7	8	9	10	11
Name:	phrest	amp unblk	C45	tau1	C180	tau2	C180	ring down	rx on	acq.	relax
Delay	1u	2u	C45	tau1	C180	tau2	C180	rd	ad	Acq. Time	Last Delay
F1_Ampl		F1 amp	F1 amp	F1 amp	F1 amp			F1 amp	F1 amp	F1 amp	
F1_PhMod		AS						AS			
F1_Ph											
F1_Attn		F1 attn	F1 attn	F1 attn	F1 attn			F1 attn	F1 attn	F1 attn	
F1_TxGate											
F1_PhRst											
F1_UnBlank											
Acq											
Acq_phase										ph0	
RX_Blank											
RX_PhRst											
F2_Ampl			F2 amp		F2 amp		F2 amp				
F2_PhMod											
F2_Ph			ph0		ph1		ph2				
F2_Attn			F2 attn		F2 attn		F2 attn				
F2_TxGate											
F2_PhRst											
F2_UnBlank											

Acquisition	Frequency	Multi Rec.	Processing	Grad. Preemph.	Misc.	Sequence	Global Variables
C45		2.1u		rd	10u	F1 amp	100
tau1		7m		ad	10u	F1 attn	15
C180		=[C45]*4		Acq. Time	1.258291s	F2 amp	100
tau2		=[tau1]+[rd]...		Last Delay	2s	F2 attn	15

Fig. 1. (a) The ¹³C APT sequence with WALTZ-16 ¹H 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:	Nalocac 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



Notes:

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

4. Results

Figure 2 shows the ¹³C APT spectrum of cholesterol in CDCl₃ acquired with the sequence introduced in Fig. 1. Note that the signals of CH and CH₃ are positive, but CH₂ and quaternary carbons including the solvent carbon are negative.

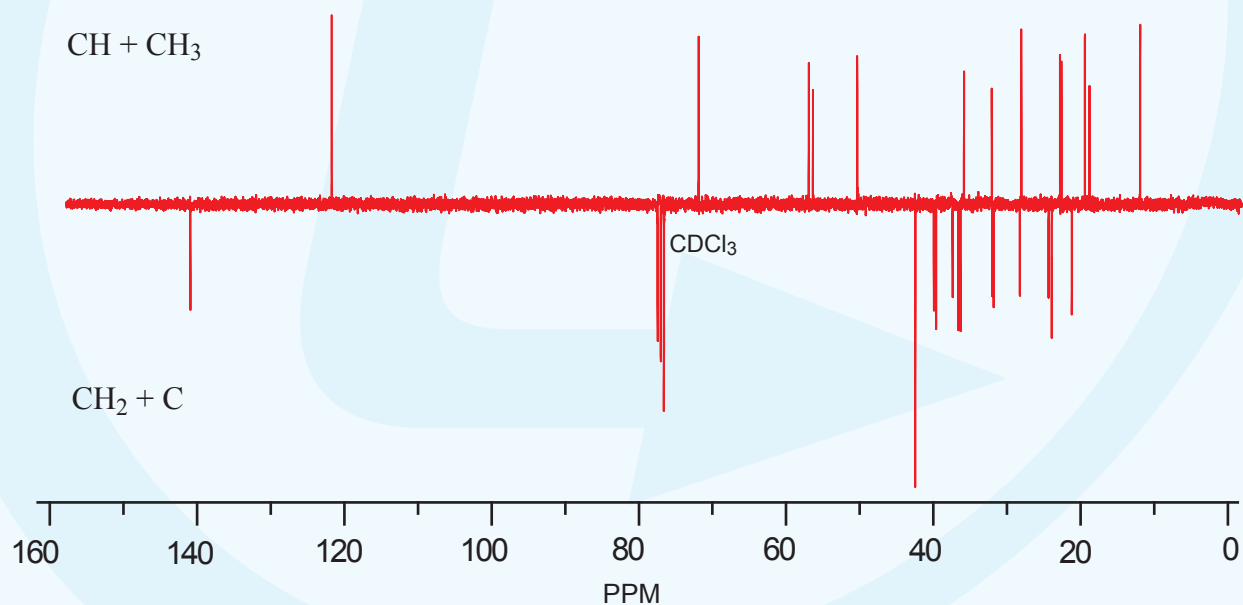


Fig. 2. The ¹³C APT spectrum of cholesterol in CDCl₃ using the sequence shown in Fig. 1.

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.