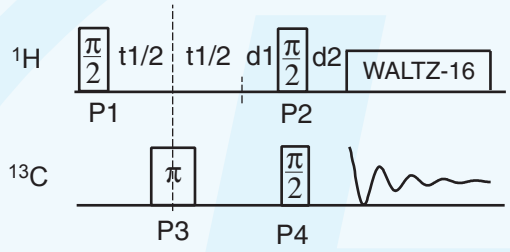
1. Introduction

A two dimensional HETeronuclear COrrelation experiment (HETCOR) is described here as ^{13}C-^{1}H Correlation by Polarization Transfer. The experiment encodes the proton chemical shift information into ^{13}C signals that are observed, and yields cross signals for all protons and ^{13}C nuclei that are connected by a ^{13}C-^{1}H coupling over one bond. The assignment of one member of a spin-coupled pair immediately leads to the assignment of the other. The HETCOR experiment described here uses a phase cycling for magnitude processing. This example demonstrates the basic principle of 2D NMR data acquisition and processing on Tecmag spectrometers.

2. Pulse sequence

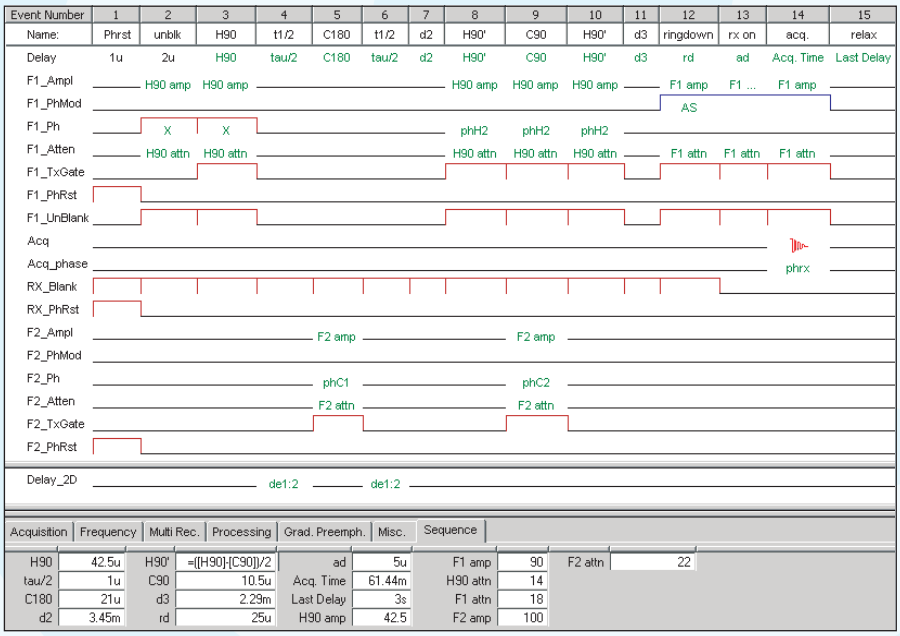
(a)



$d1 = 1/2J_{CH}$
$d2 = 1/3J_{CH}$

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

(b)



1D phase tables:

(P1):	0.
ph1(P2):	0, 2, 1, 3.
ph0(P3):	0, 0, 0, 0, 2, 2, 2, 2.
ph1(P4):	0, 0, 0, 0, 1, 1, 1, 1, 2, 2, 2, 2. 3, 3, 3, 3.
phrx:	$(0, 2, 1, 3)_2$, $(1, 3, 2, 0)_2$, $(2, 0, 3, 1)_2$, $(3, 1, 0, 2)_2$.

(All tables are in 4 step mode.)

2D delay table:

de1:2 (t1/2):
Auto, Every Pass.
Increment value: 200 µs,

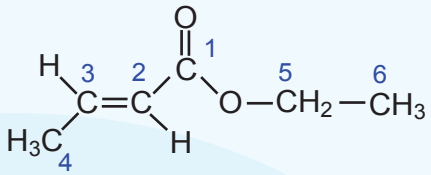
Fig. 1. (a) The pulse sequence for the HETCOR experiment. (b) The sequence in the NTNMR sequence editor with phase tables set for magnitude processing.

3. Experiment

Sample:	20 % ethyl crotonate in $CDCl_3$
Spectrometer:	7 Tesla magnet with Tecmag HF3 Discovery
Probe:	Nalorac D300-5 OWB 5mm $^{1}H/^{13}C$ Switchable probe
^{13}C 90° pulse:	10.5 µs
^{1}H 90° pulse:	42.5 µs

3. Experiment (continued)

SW +/- (1D, ^{13}C) :	8.3 kHz (110.5 ppm)
SW 2D (+/-, ^{1}H):	1.25 kHz (4.15 ppm)
Dwell time (1D):	60 µs
Dwell_2D:	400 µs
Acq. points (and Points 1D):	1024
Points 2D:	256
Scans 1D:	32



Ethyl crotonate

Notes:

1. The sample is locked but not spun. The magnet is shimmed to ~0.8 Hz line width.
2. First, calibrate the 90° pulse widths of ^{1}H and ^{13}C using the nutation experiment (see note, "One Pulse Experiment and Pulse Calibration").
3. Set up the WALTZ sequence according to the note, "^{13}C NMR Spectra with ^{1}H WALTZ Decoupling".
4. The center of pulses P2 and P4 should be aligned. Since P2 > P4, P2 have to split into 3 pulses. The delay of P2's middle pulse equals to P4, and the delay of both sides is (P2 - P4)/2 . The middle pulse of P2 falls on the same event as P4 (and P5).

4. Results

Data processing:

In the NDFT window,
click the "1D" tab and select:
"Use 1D settings",
"Fourier Transform",
"Sine Bell, SB Shift: 90,
SB Skew: 5, SB width: 1024";
click the "2D" tab and select:
"Use 2D settings",
"Zero fill, 1",
"Sine Bell, SB Shift: 90,
SB Skew: 5, SB width: 512";
"Fourier Transform",
"Transpose: complex",
click "Do it".□

The data processing procedure is automated in an NMRScript. To use the script, click "Scripts|Processing scripts| Process 2D (Magnitude)".

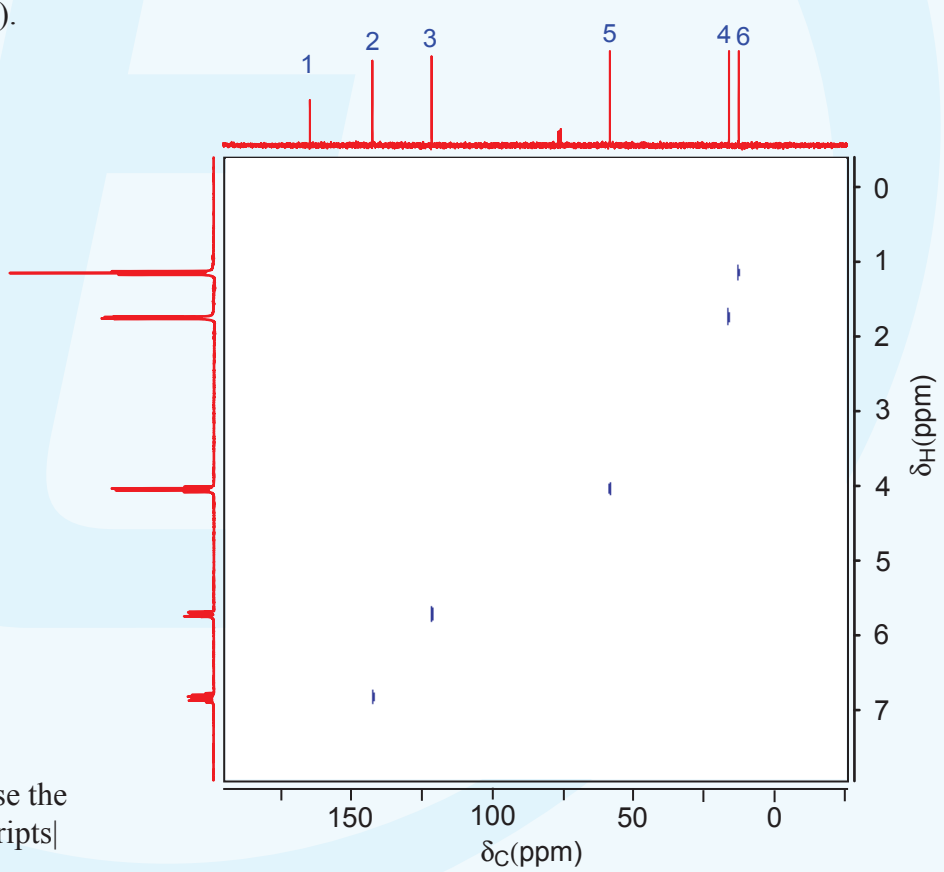


Fig. 2. Magnitude plot of the HETCOR spectrum of ethyl crotonate.

5. References

1. R. Freman, G. A. Morris, *J. Chem. Soc. Chem. Commun.*, **1978**, 684-686.

2. S. Braun, H.-O. Kalinowski, S. Berger, "150 and More Basic NMR Experiments", Wiley-VCH, 1999, 375-377.

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