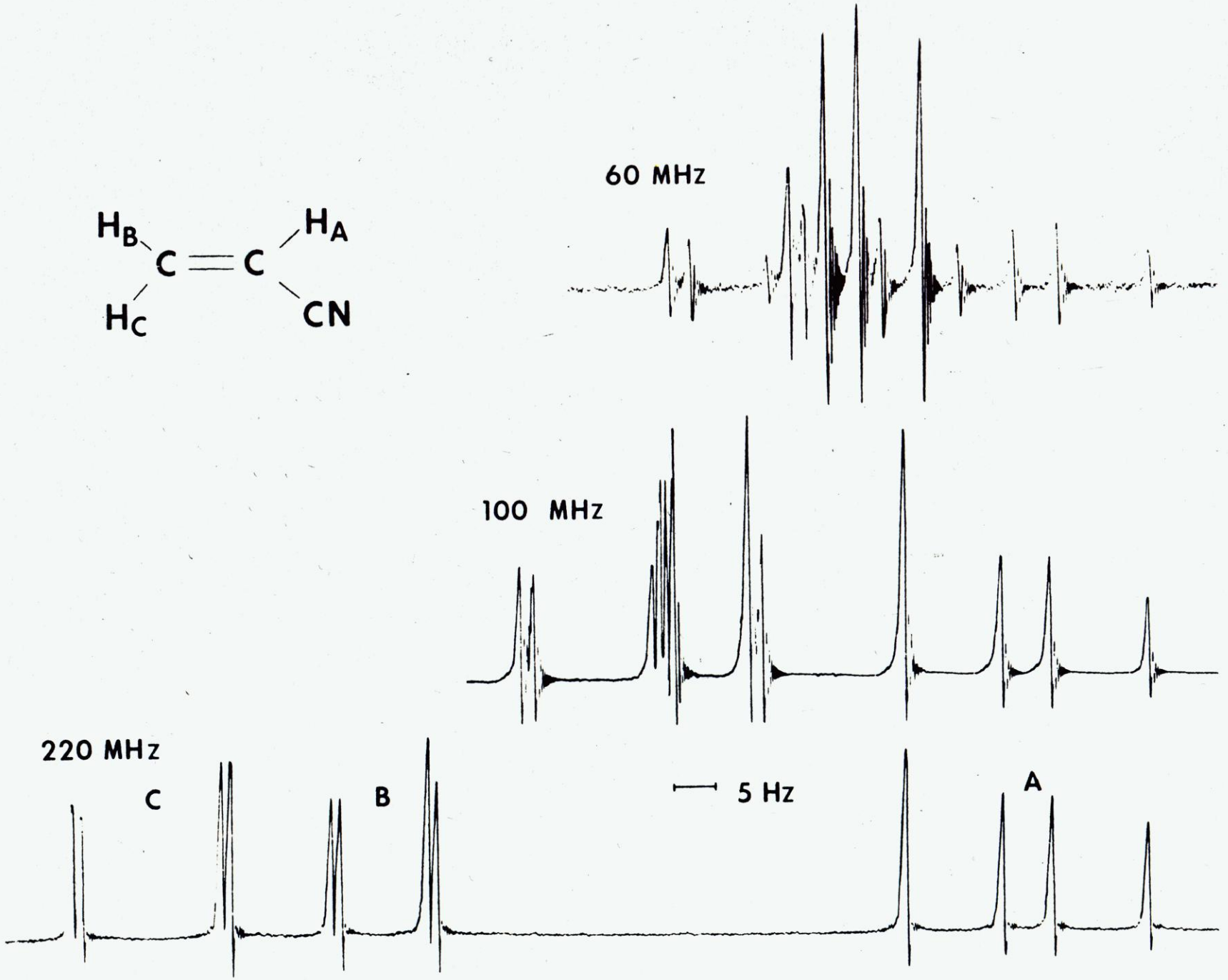
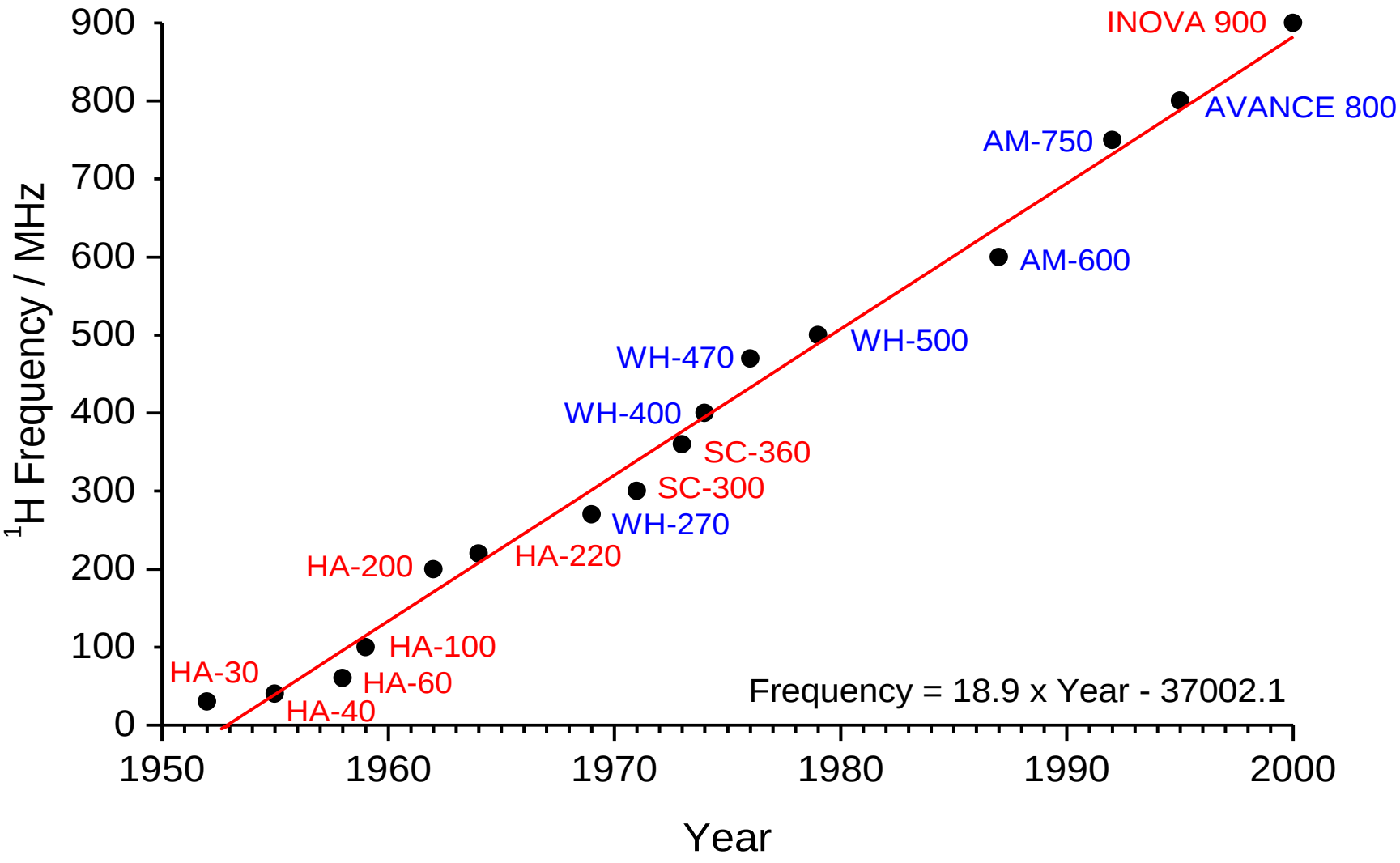
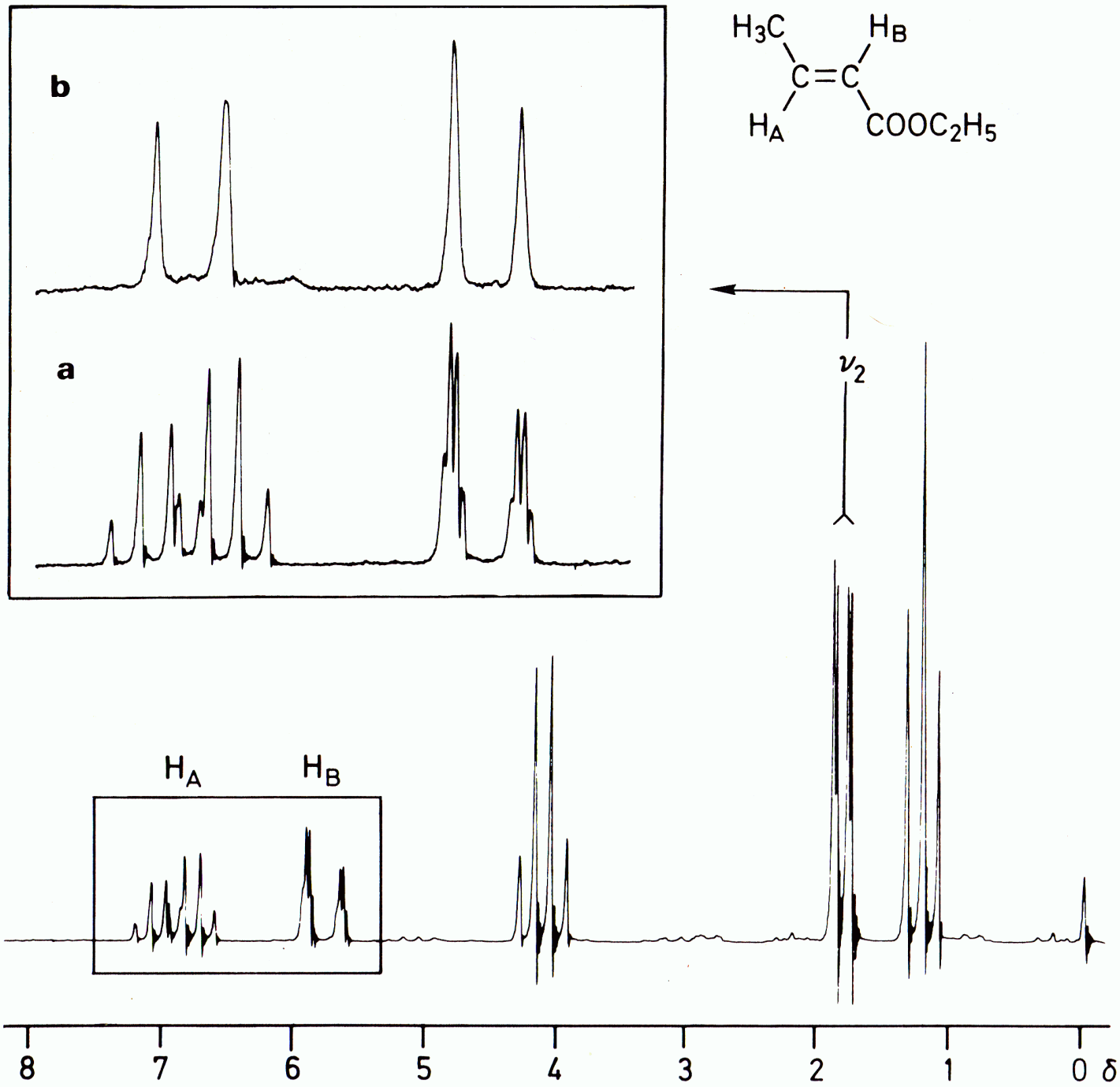
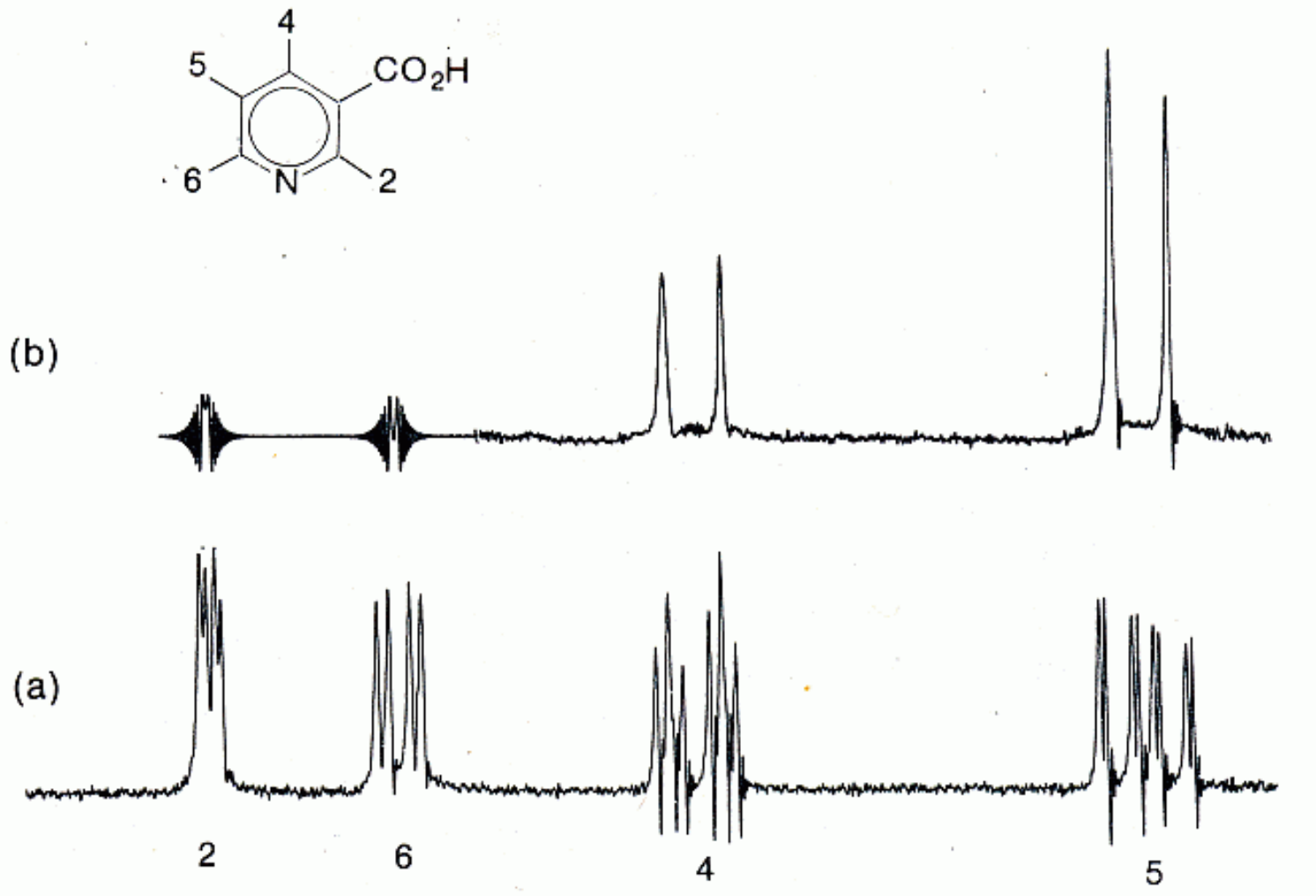
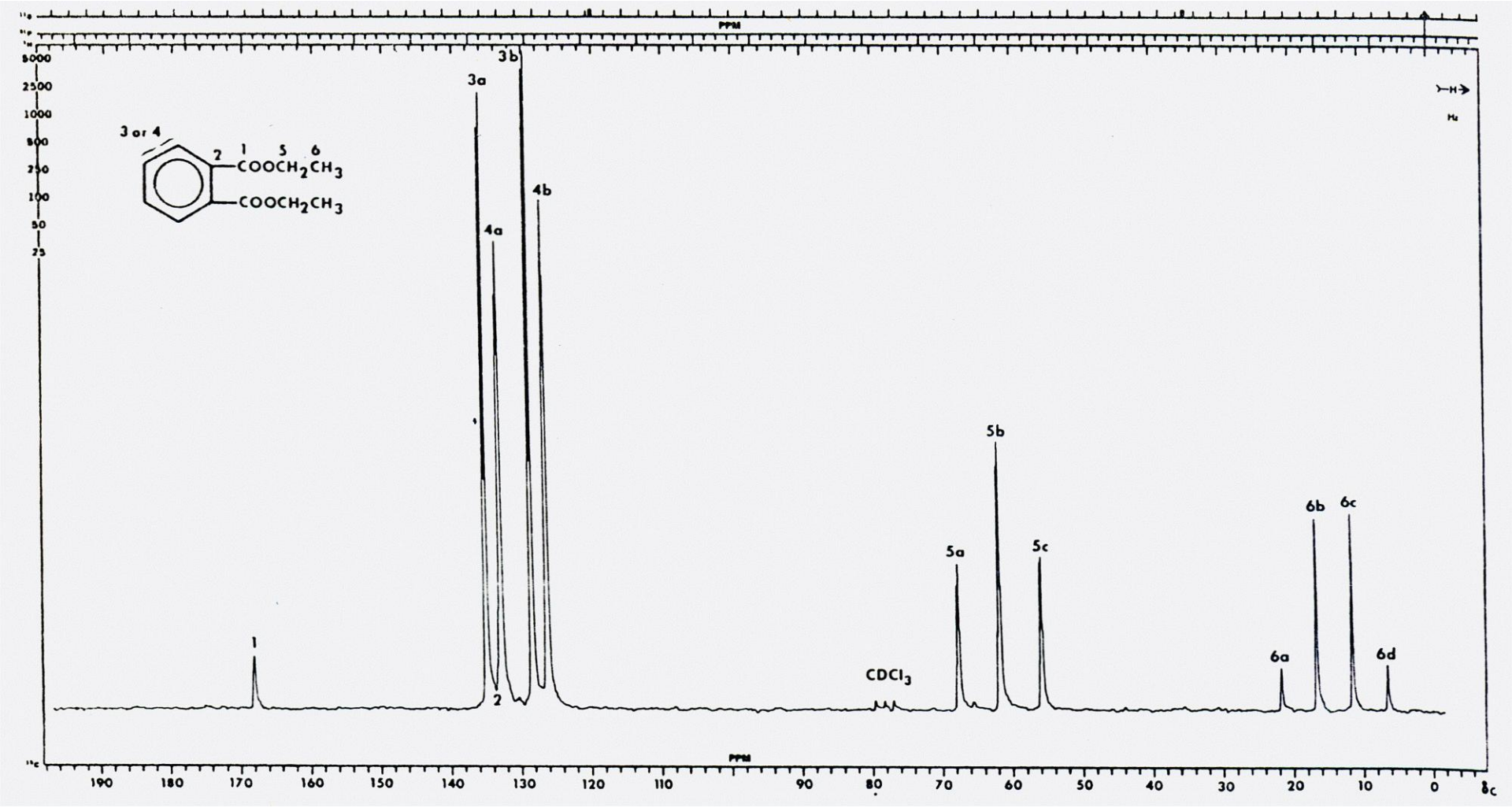
RMN PULSADA, COM TRANSFORMADA DE FOURIER.

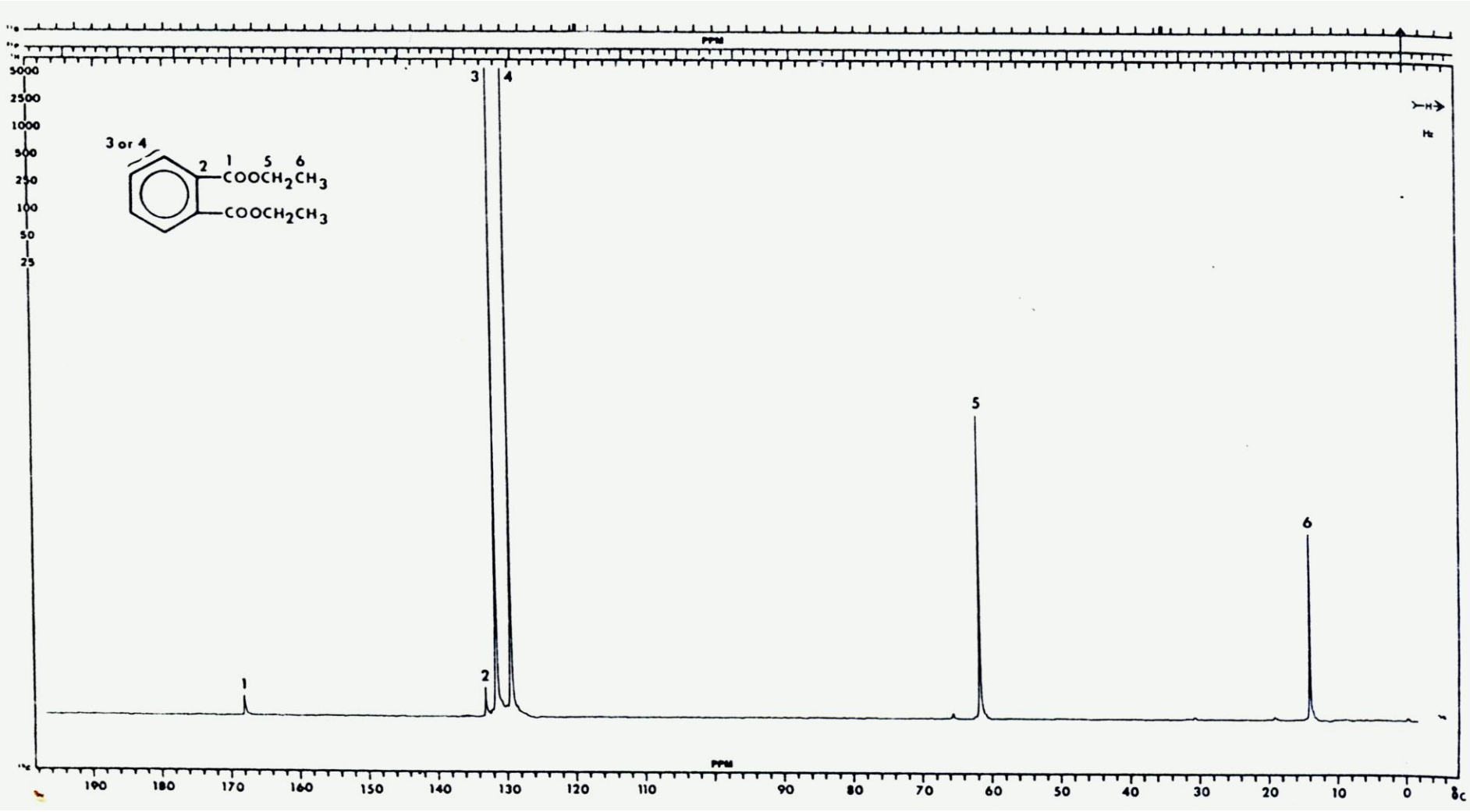

H_B
C
C
H_A
H_C
CN
60 MHz
100 MHz
220 MHz
C
B
5 Hz
A


900
800
700
600
500
400
300
200
100
0
1950
1960
1970
1980
1990
2000
Year
^{1}H Frequency / MHz
INOVA 900
AVANCE 800
AM-750
AM-600
WH-500
WH-470
WH-400
SC-360
SC-300
WH-270
HA-220
HA-200
HA-100
HA-60
HA-40
HA-30
Frequency = 18.9 x Year - 37002.1


b
a
H_3C
H_B
C=C
H_A
$COOC_2H_5$
ν_2
H_A
H_B
8
7
6
5
4
3
2
1
0 δ


4
5
CO2H
6
N
2
(b)
(a)
2
6
4
5


PPM
3 or 4
2 1 5 6
COOCH2CH3
COOCH2CH3
3a
3b
4a
4b
5a
5b
5c
6a
6b
6c
6d
1
2
CDCl3
PPM
δC


PPM
3
4
5
6
1
2
3 or 4
2 1 5 6
COOCH2CH3
COOCH2CH3
5000
2500
1000
500
250
100
50
25
Hz
190
180
170
160
150
140
130
120
110
90
80
70
60
50
40
30
20
10
0
δC

EXPERIMENTAL PARAMETERS

Instr.	HA–100
Mode	CW
Time	15 min
Solv.	Dioxane
Conc.	.5ml/1ml soln

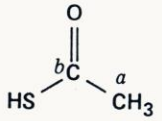
ASSIGNMENTS

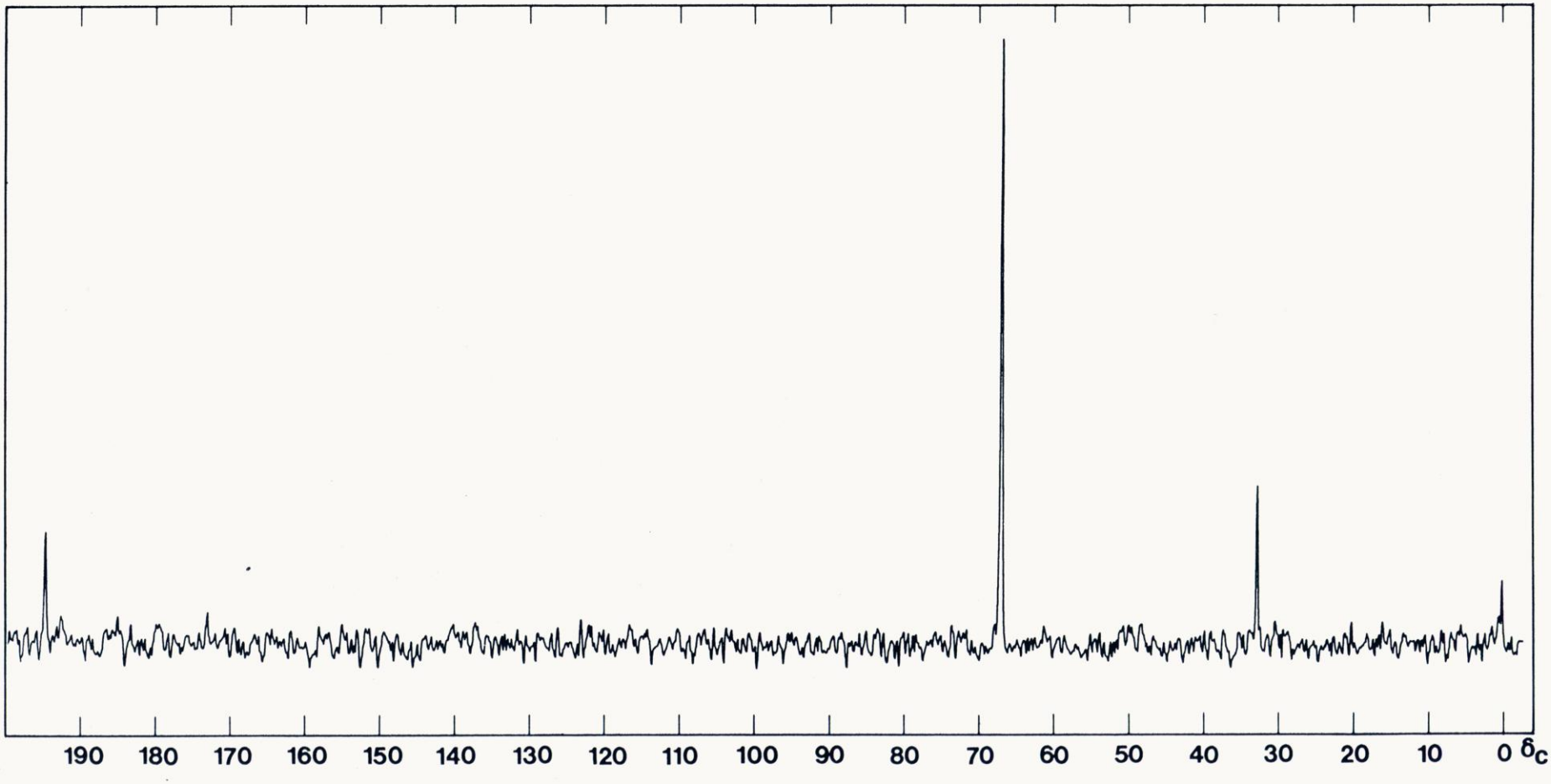
a	32.6
b	194.5

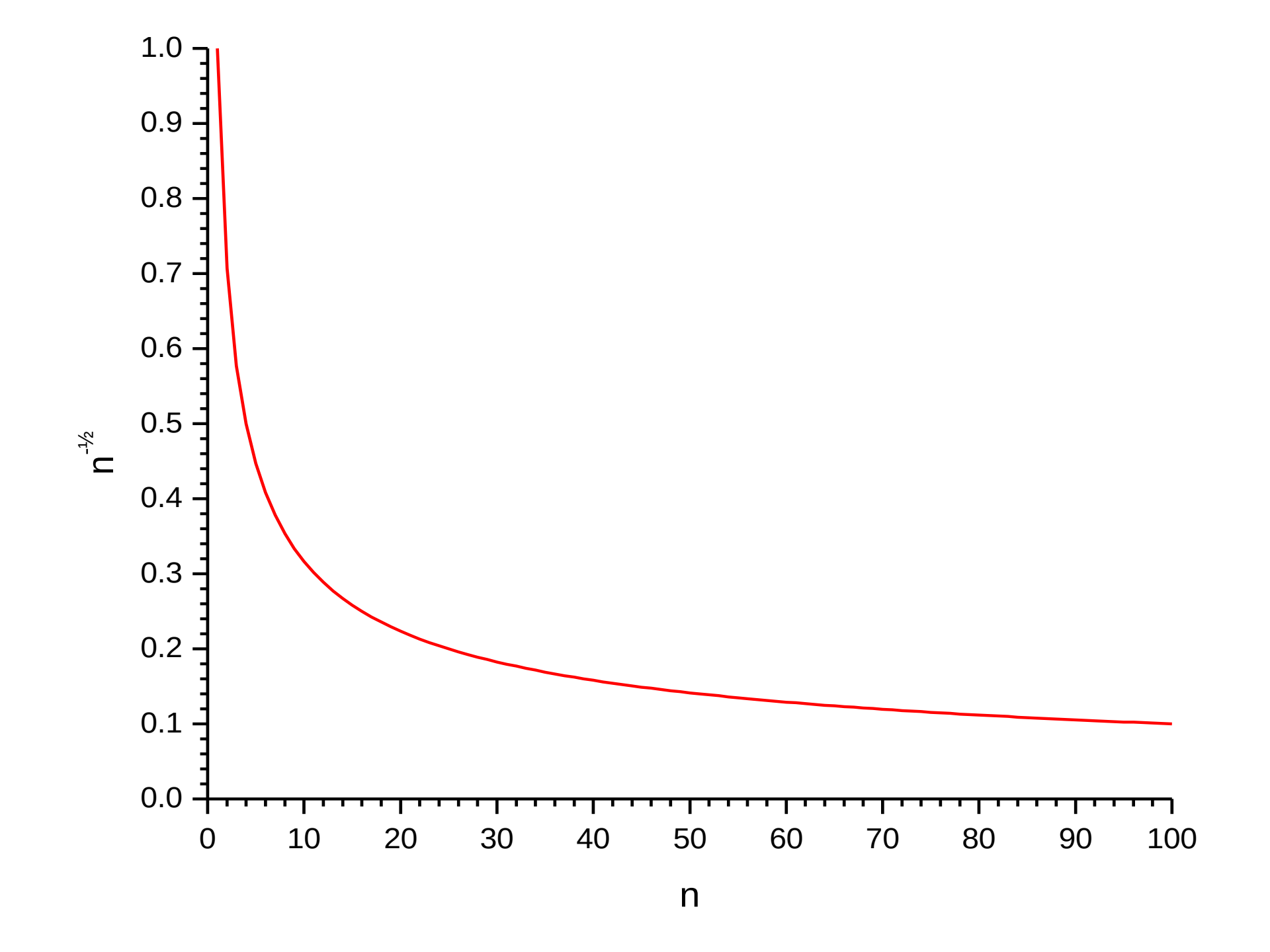
thioacetic acid

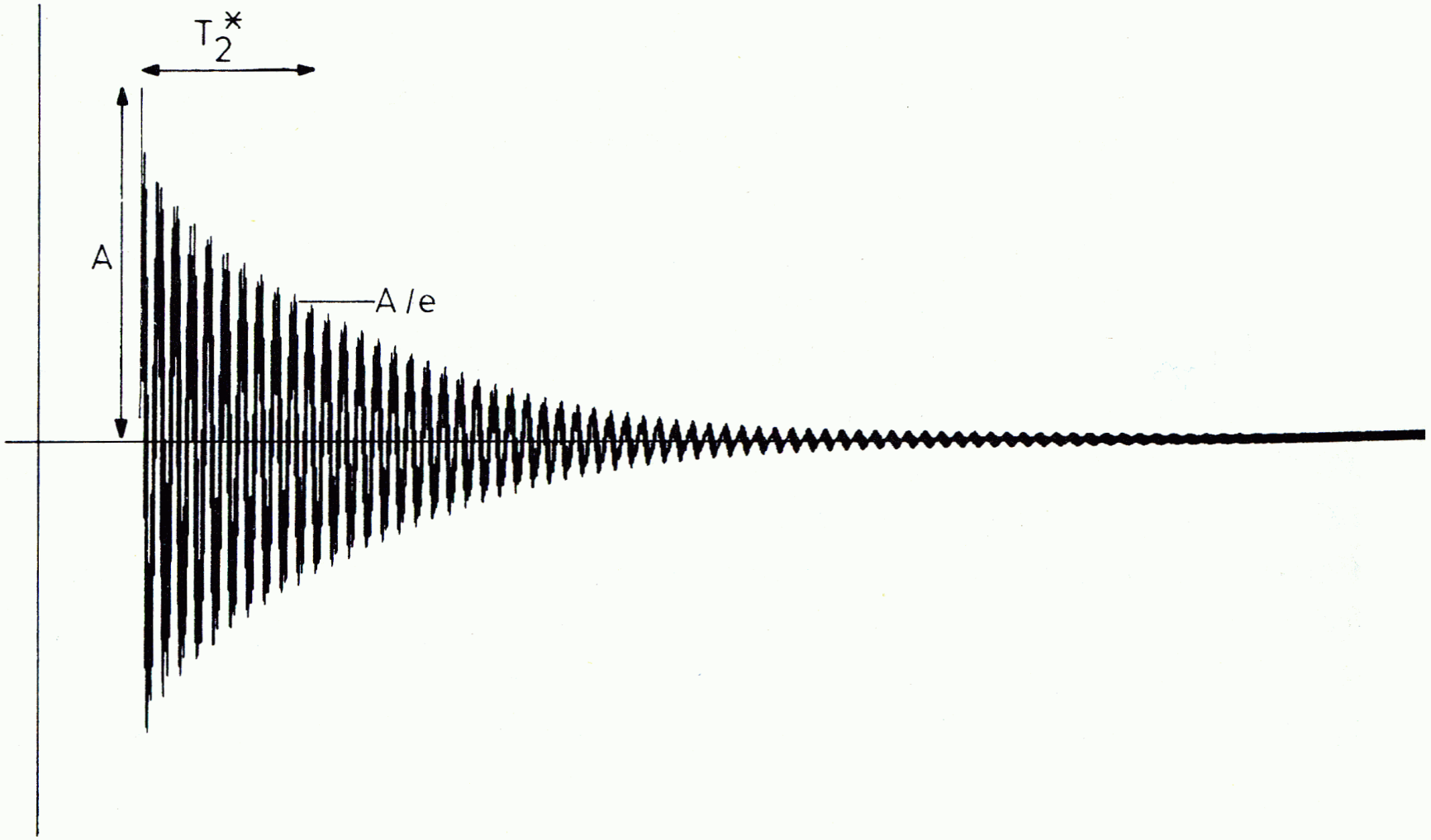
6

C_2H_4OS

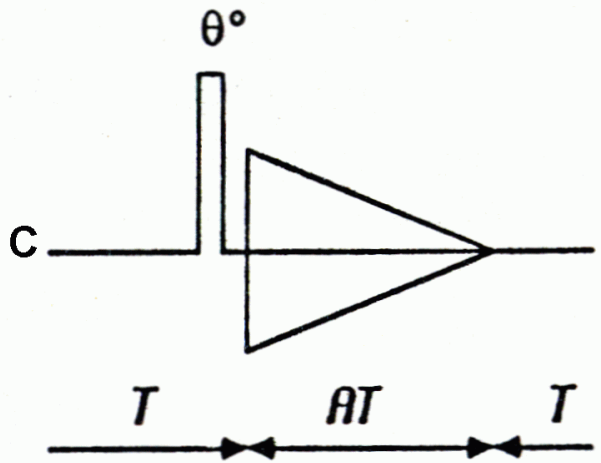




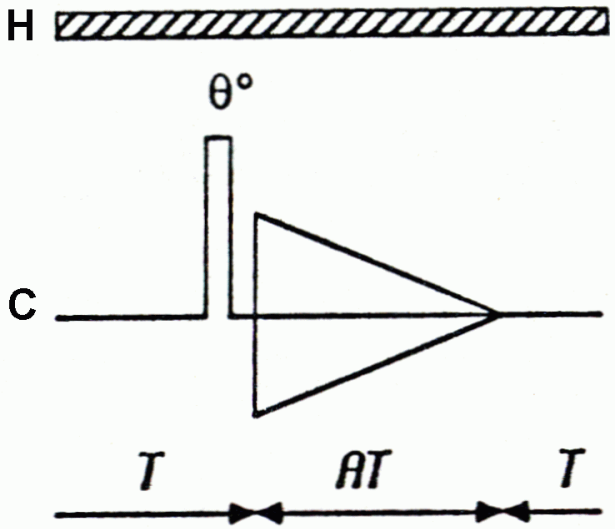

1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
n-½
0
10
20
30
40
50
60
70
80
90
100
n


T_2^*
A
A /e

ACOPLADO



DESACOPLADO



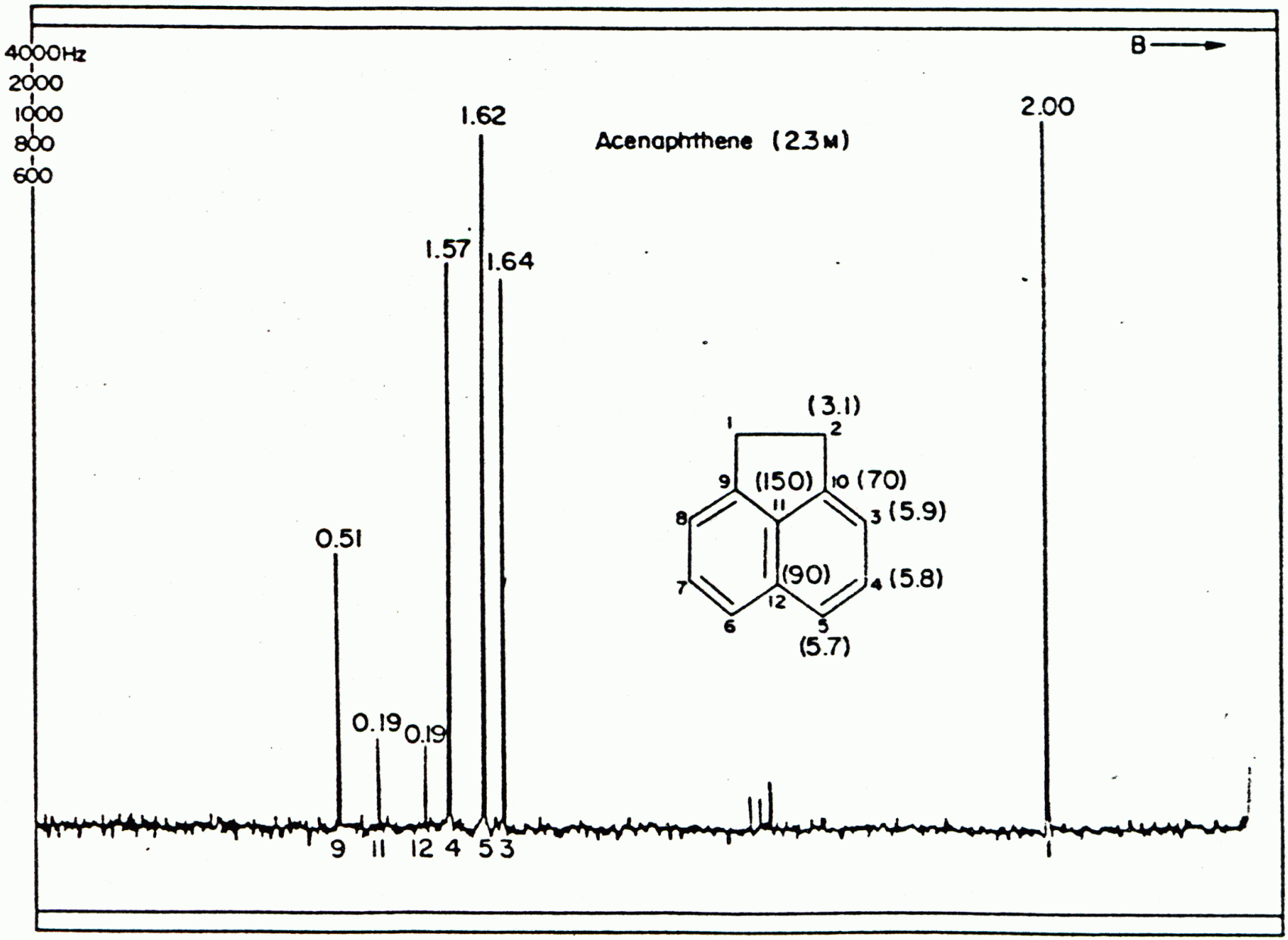
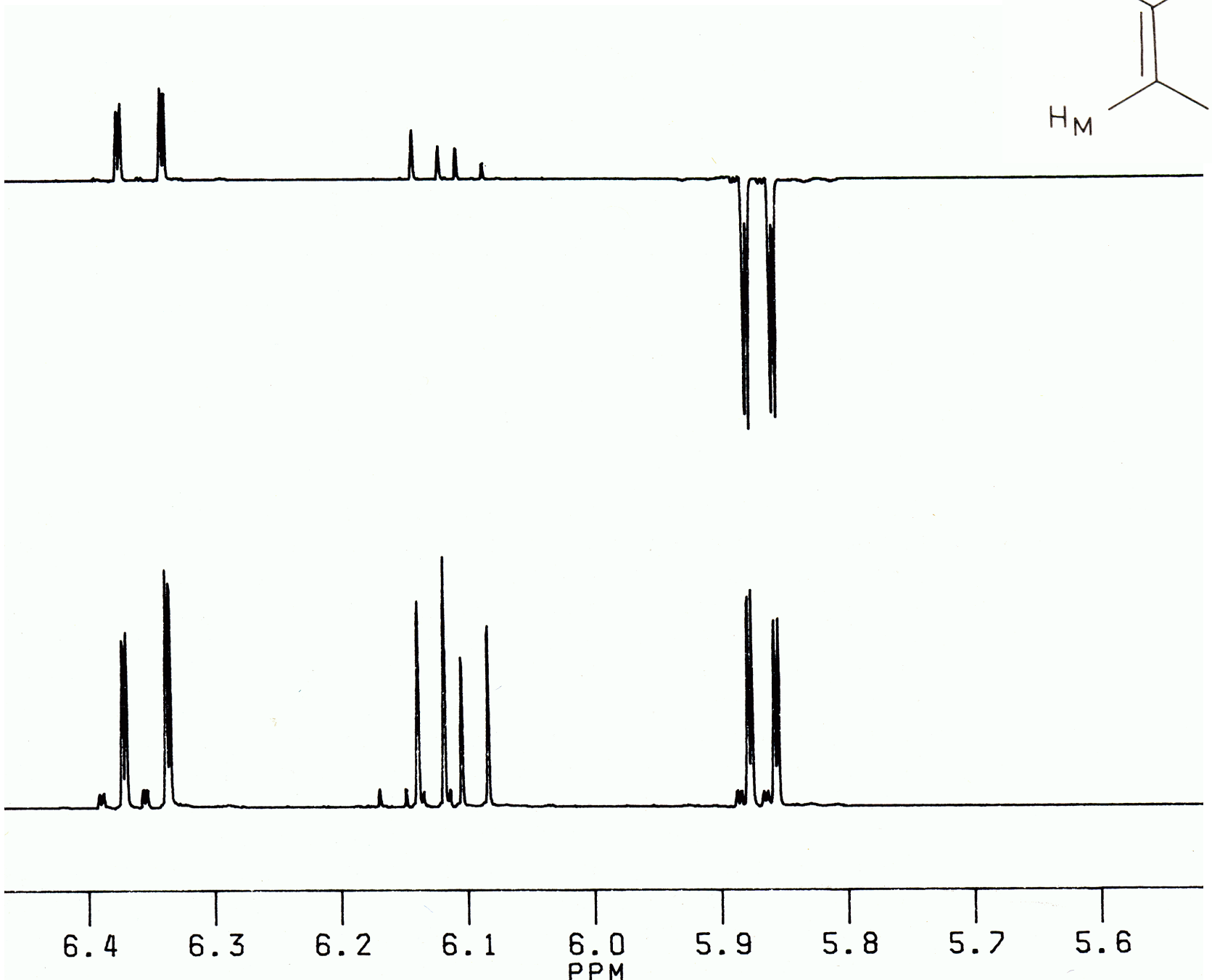
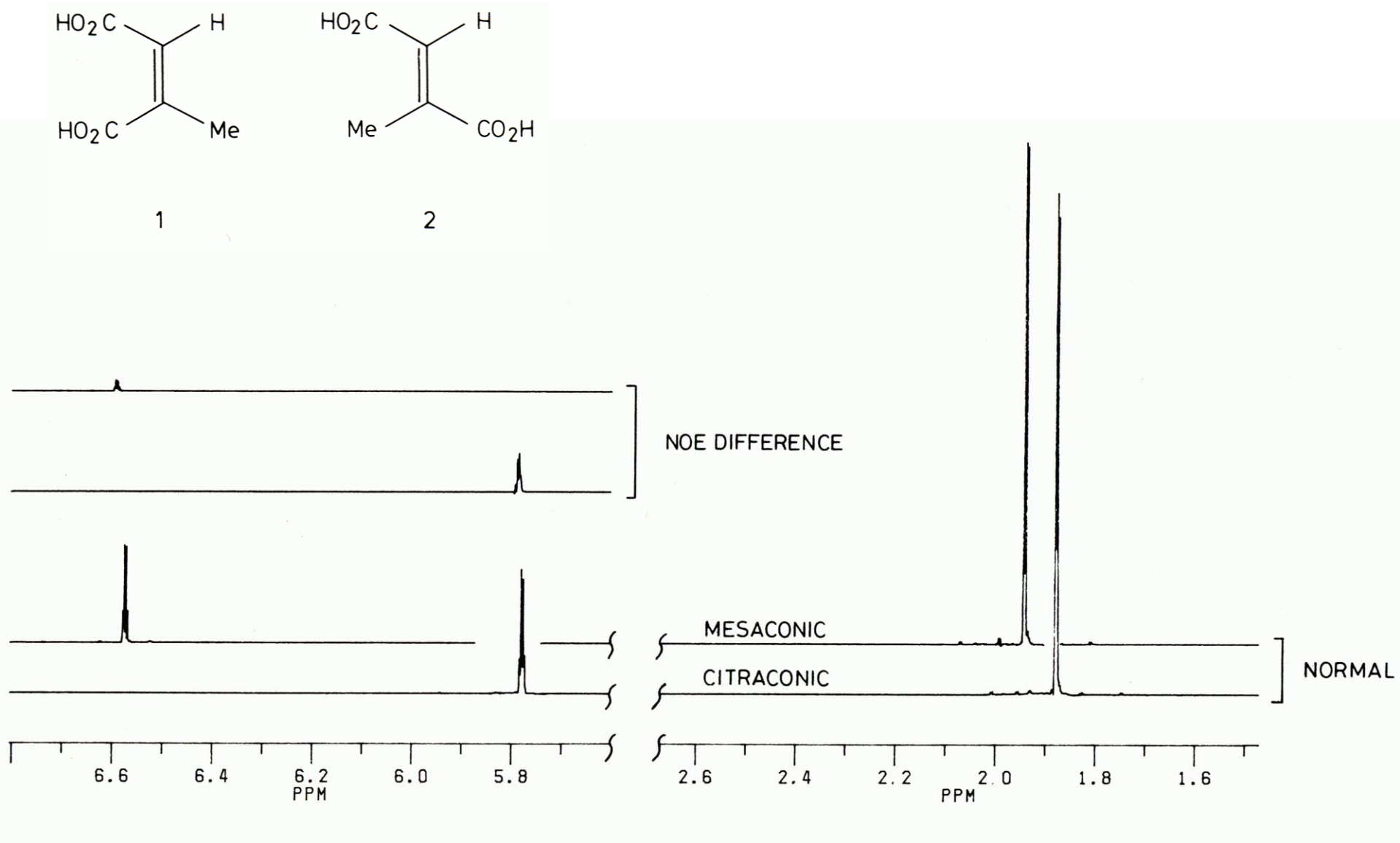
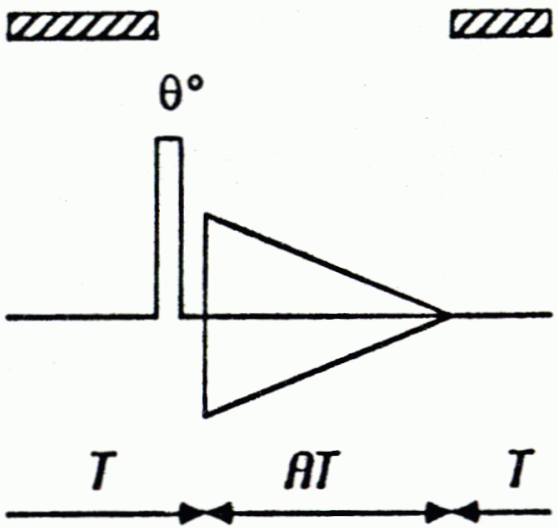


FIG. 1. Acenaphthene ^{13}C spectrum run under normal conditions showing assignments and T_1 relaxation times.

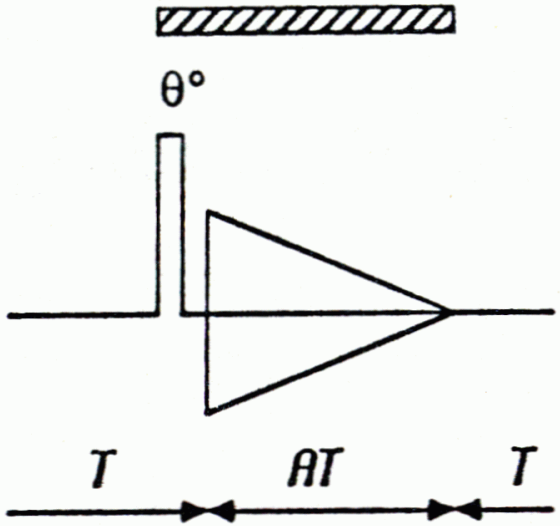

HO2C
Hx
HM
HA
6.4
6.3
6.2
6.1
6.0
5.9
5.8
5.7
5.6
PPM


HO2C
H
HO2C
Me
1
HO2C
H
Me
CO2H
2
NOE DIFFERENCE
MESACONIC
CITRACONIC
NORMAL
6.6
6.4
6.2
6.0
5.8
PPM
2.6
2.4
2.2
2.0
1.8
1.6
PPM

GATEDEC



INVGATE



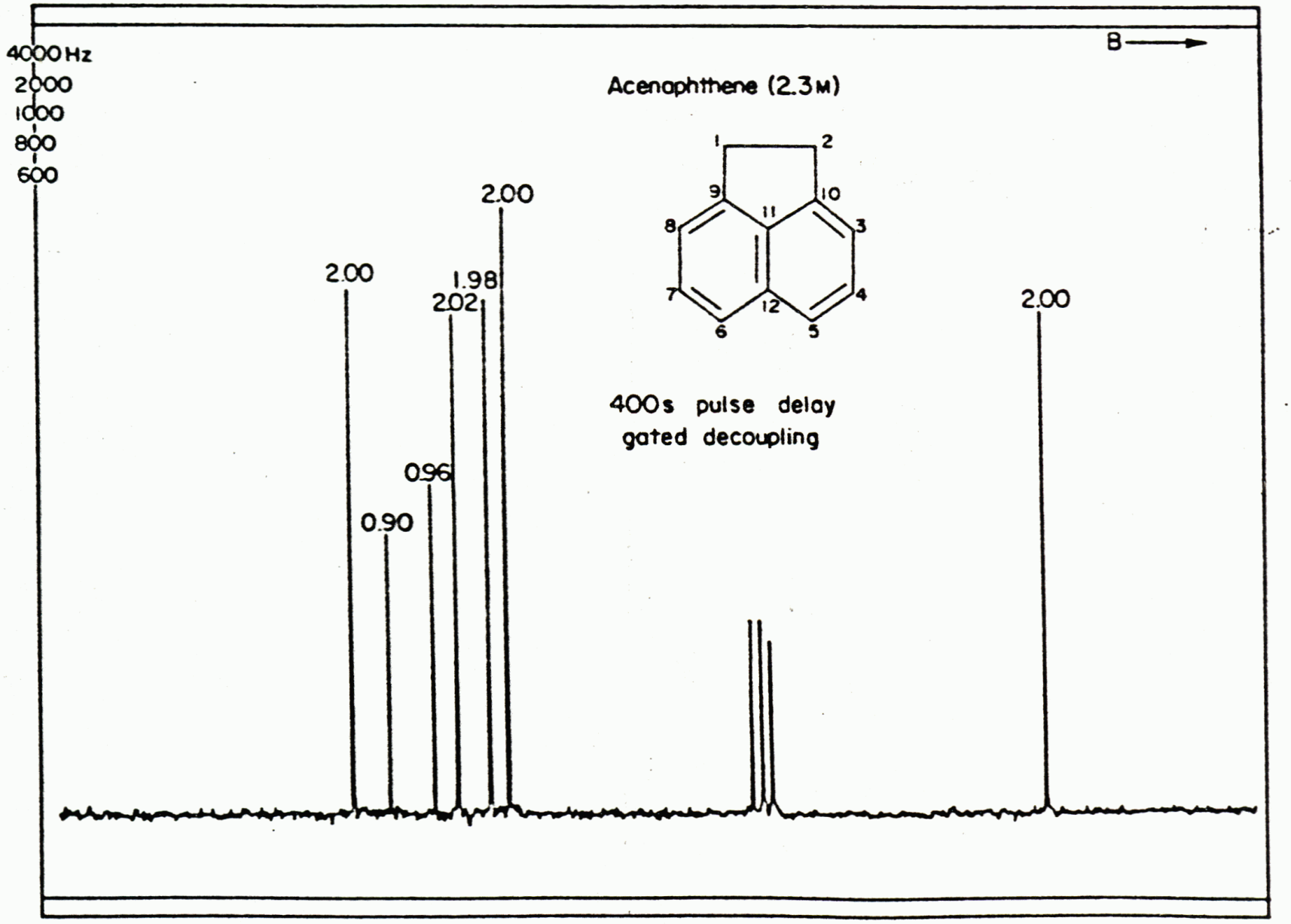
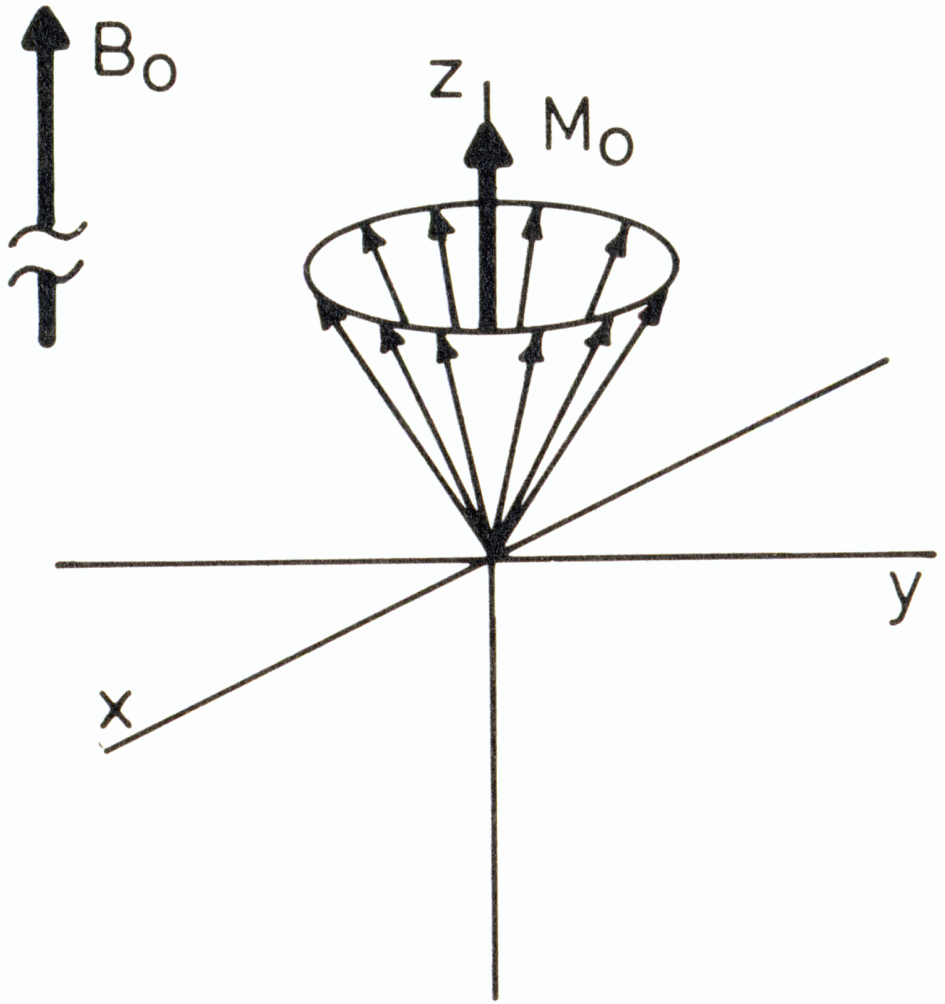
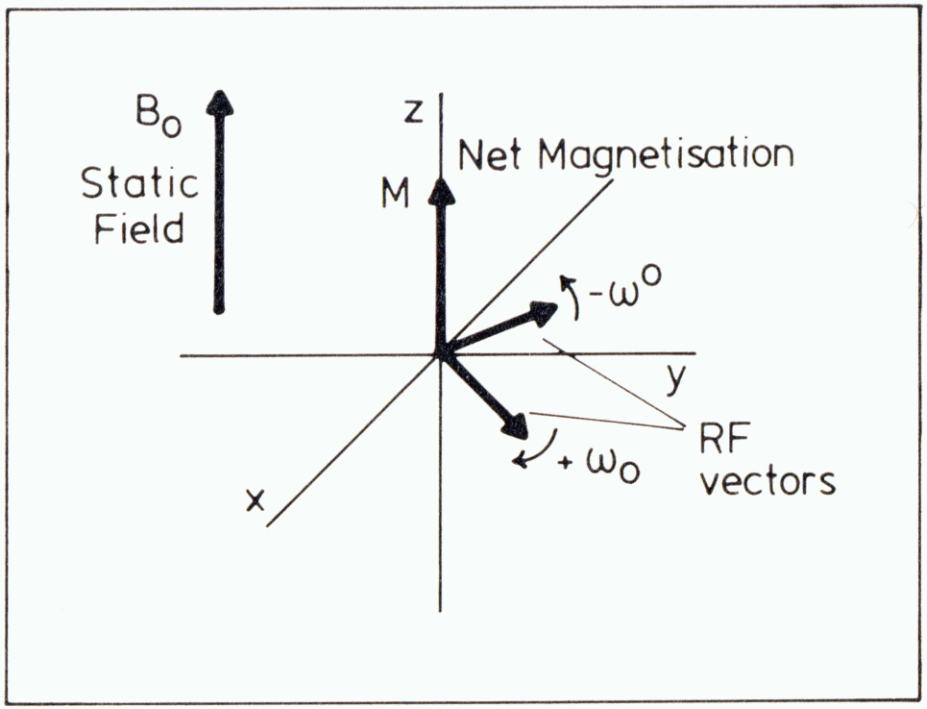
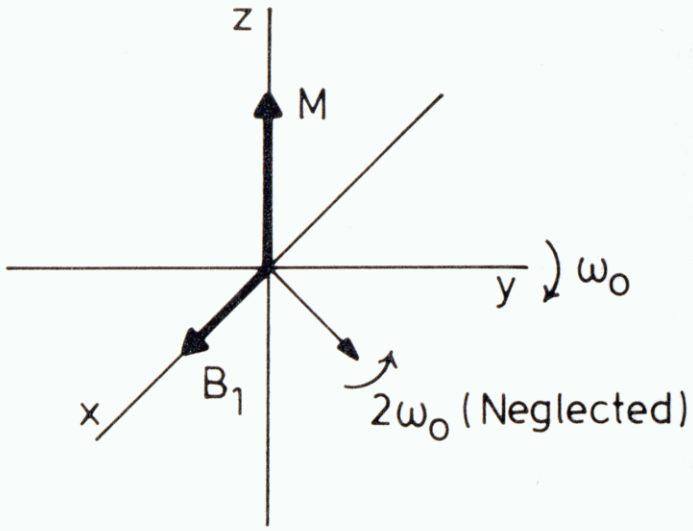


FIG. 3. Acenaphthene ^{13}C spectrum with 400 s pulse delay and decoupler gated 'off' during delay period.

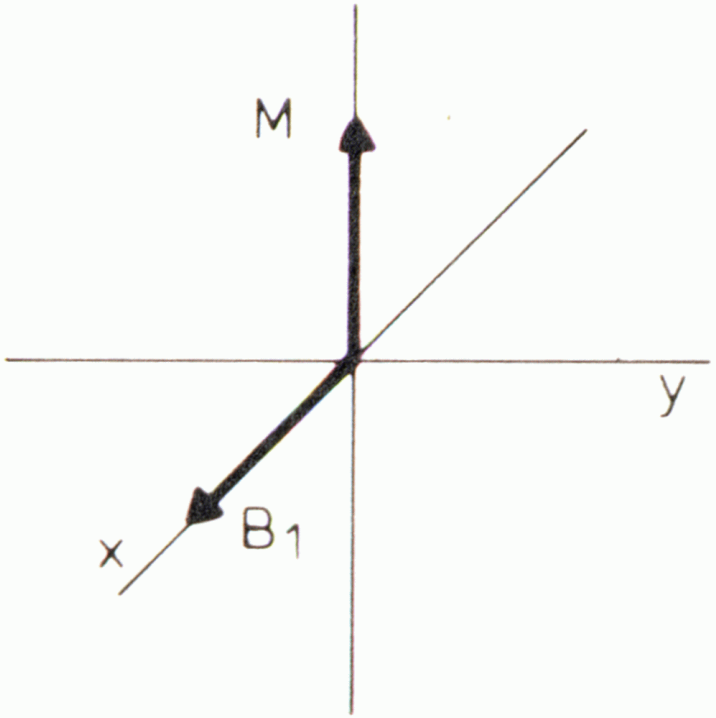

Bo
z
Mo
y
x

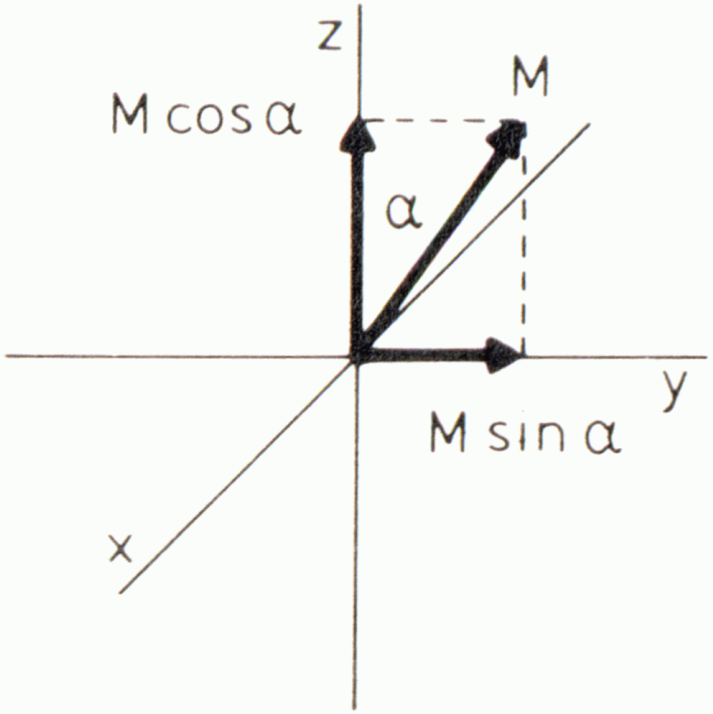


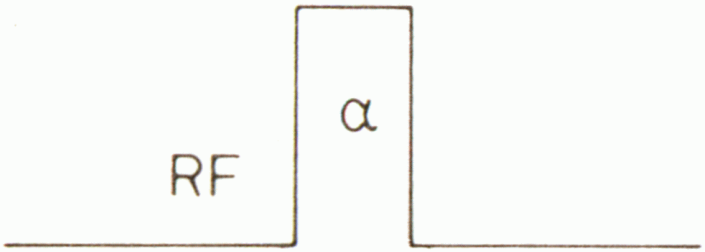
LAB. FRAME



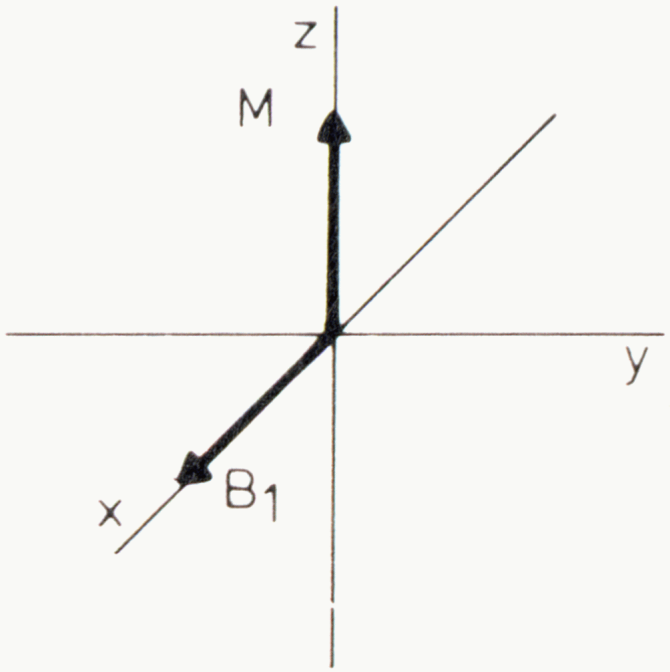
ROTATING FRAME

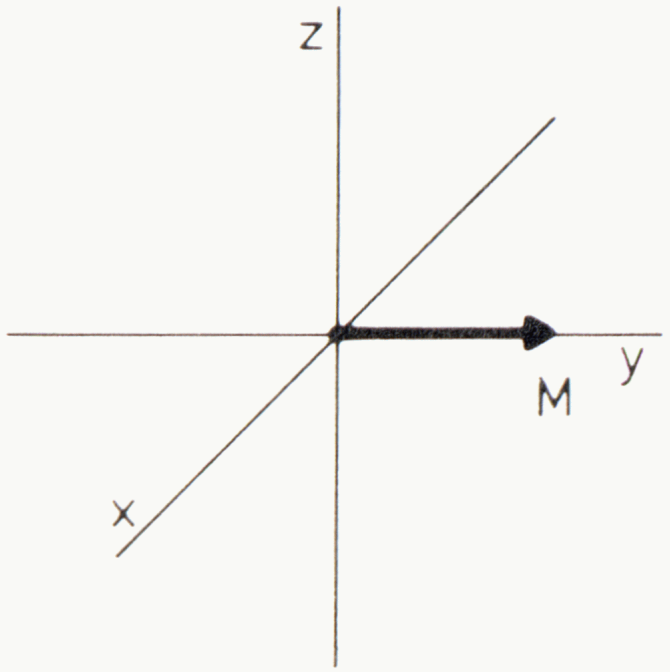


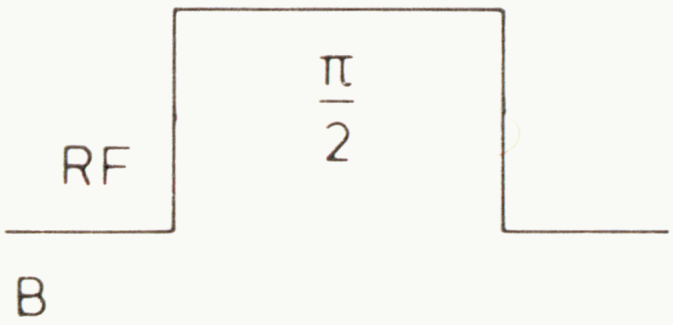


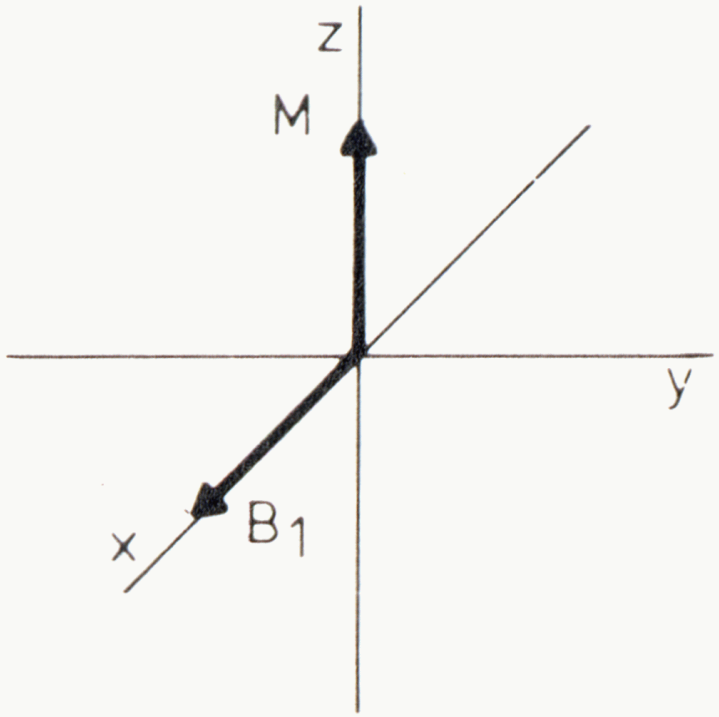


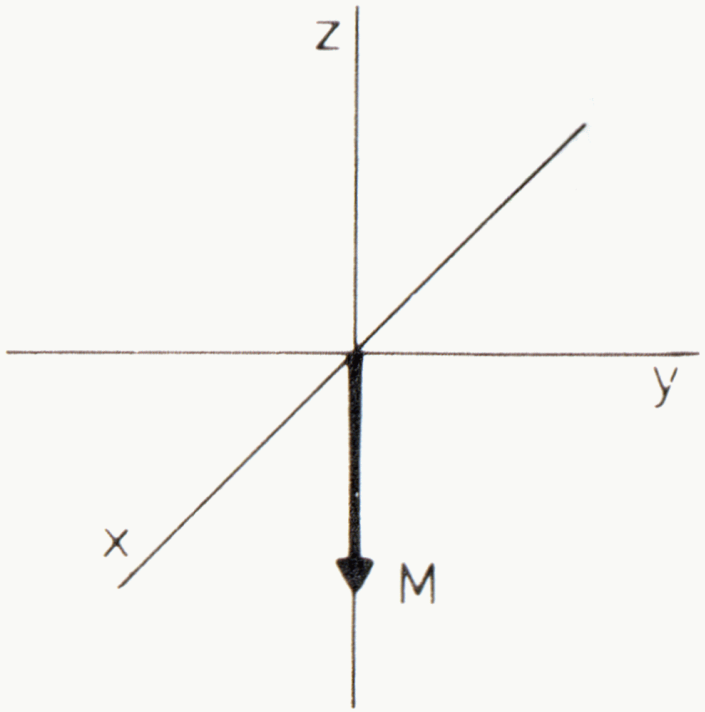
A

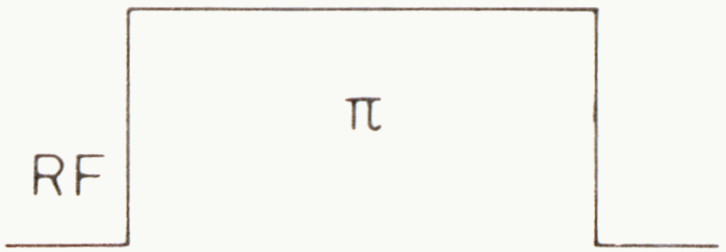

z
M
y
x
B_1


z
y
M
x


RF
π/2
B

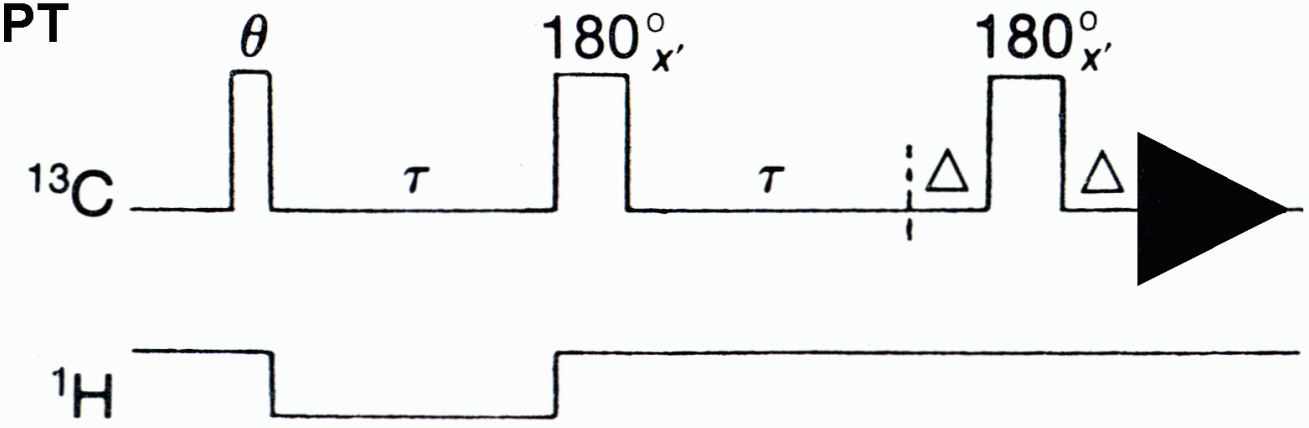


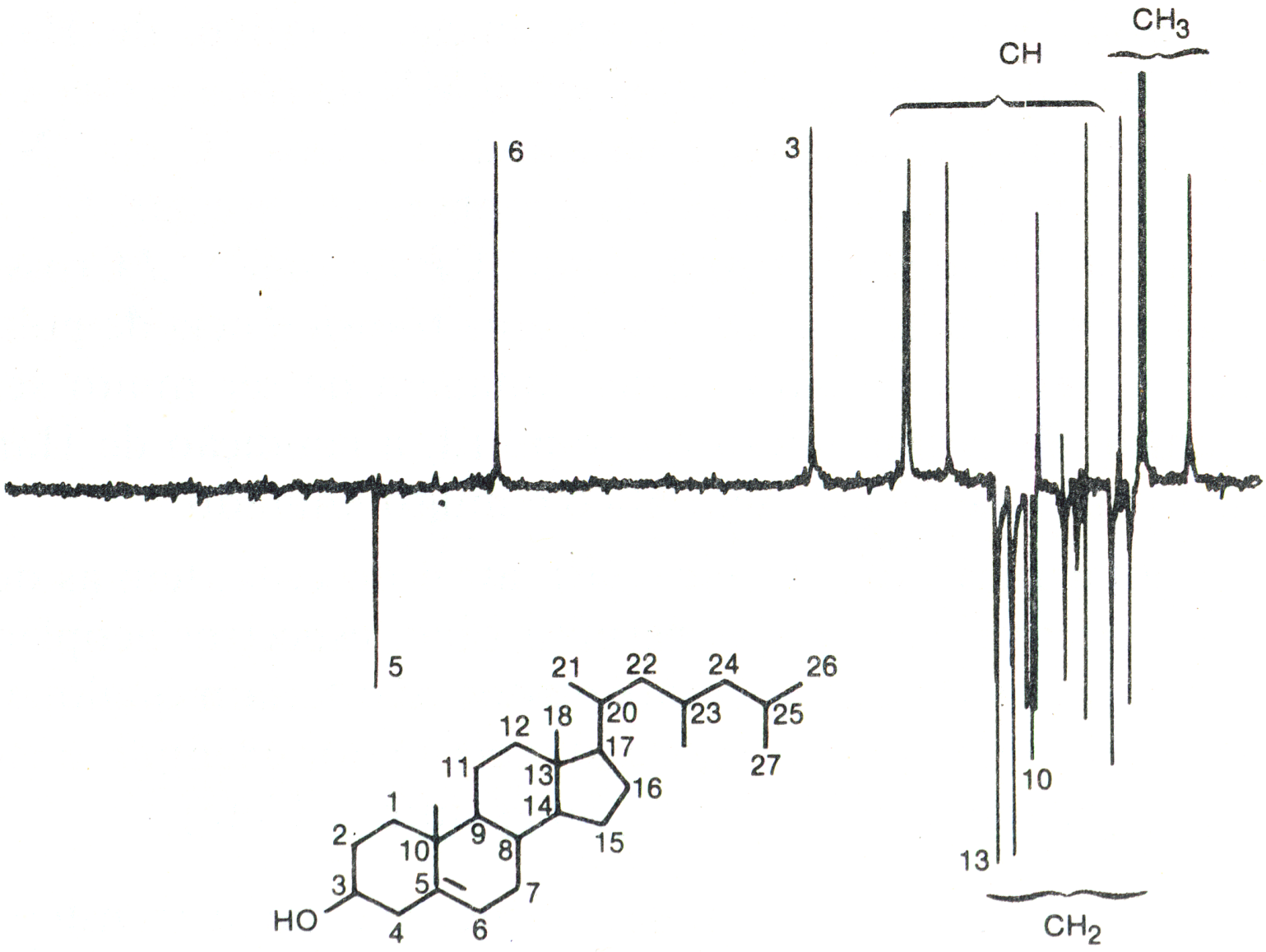


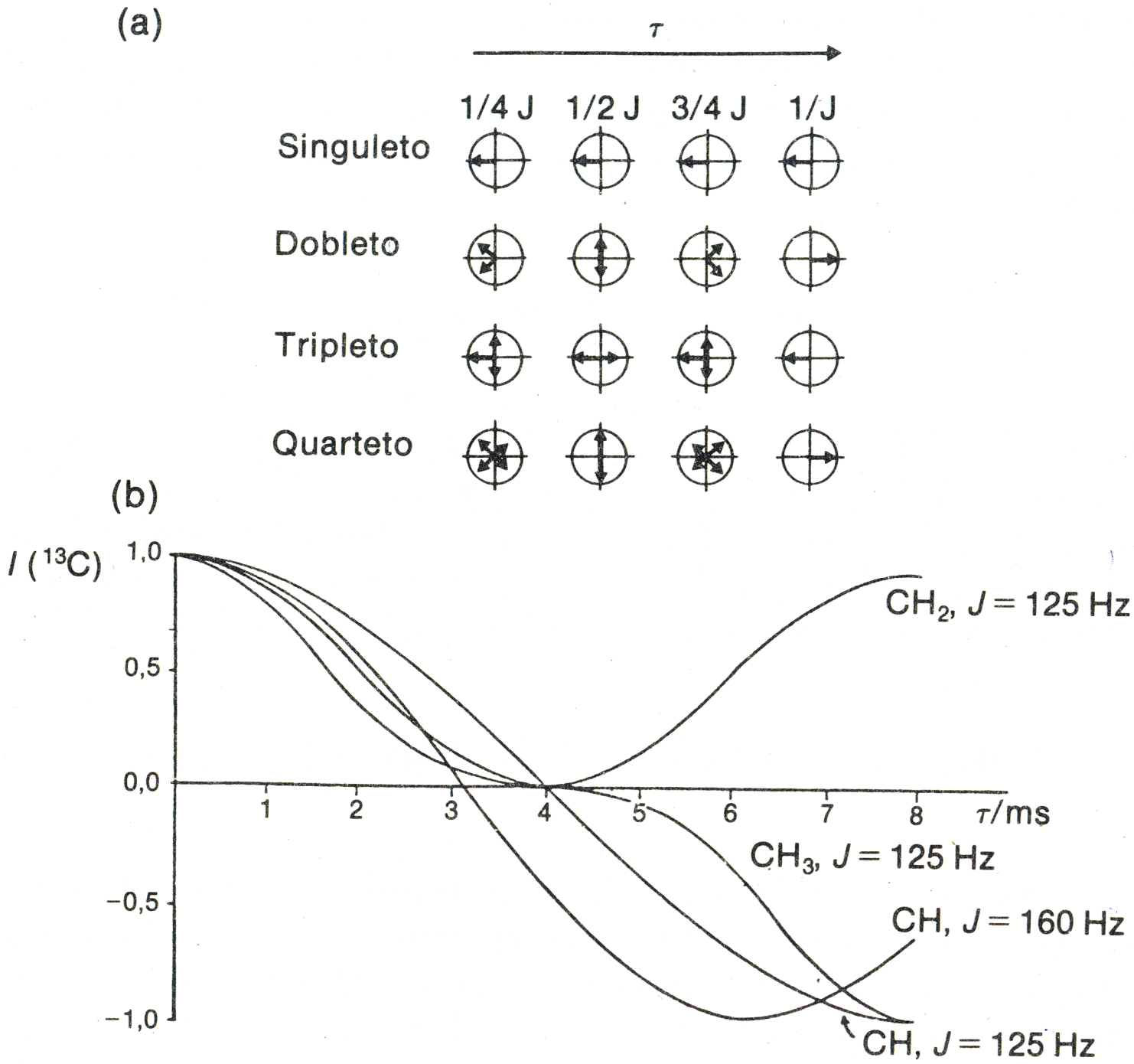


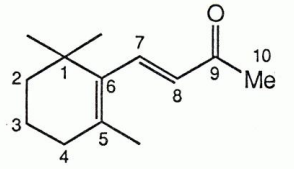
C

APT




CH
CH3
6
3
5
10
13
CH2
HO
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
20
21
22
23
24
25
26
27


(a)
τ
1/4 J
1/2 J
3/4 J
1/J
Singuleto
Dobleto
Tripleto
Quarteto
(b)
I (13C)
1,0
0,5
0,0
−0,5
−1,0
1
2
3
4
5
6
7
8
τ/ms
CH2, J = 125 Hz
CH3, J = 125 Hz
CH, J = 160 Hz
CH, J = 125 Hz



C (θ=135°)

B (θ=90°)

A (θ=45°)

200 180 160 140 120 100 80 60 40 20 0 PPM

IONONE 100
SUNBOR 21DEC86

BETA-IONONE /CDCL3
DEPT 45 DEG.

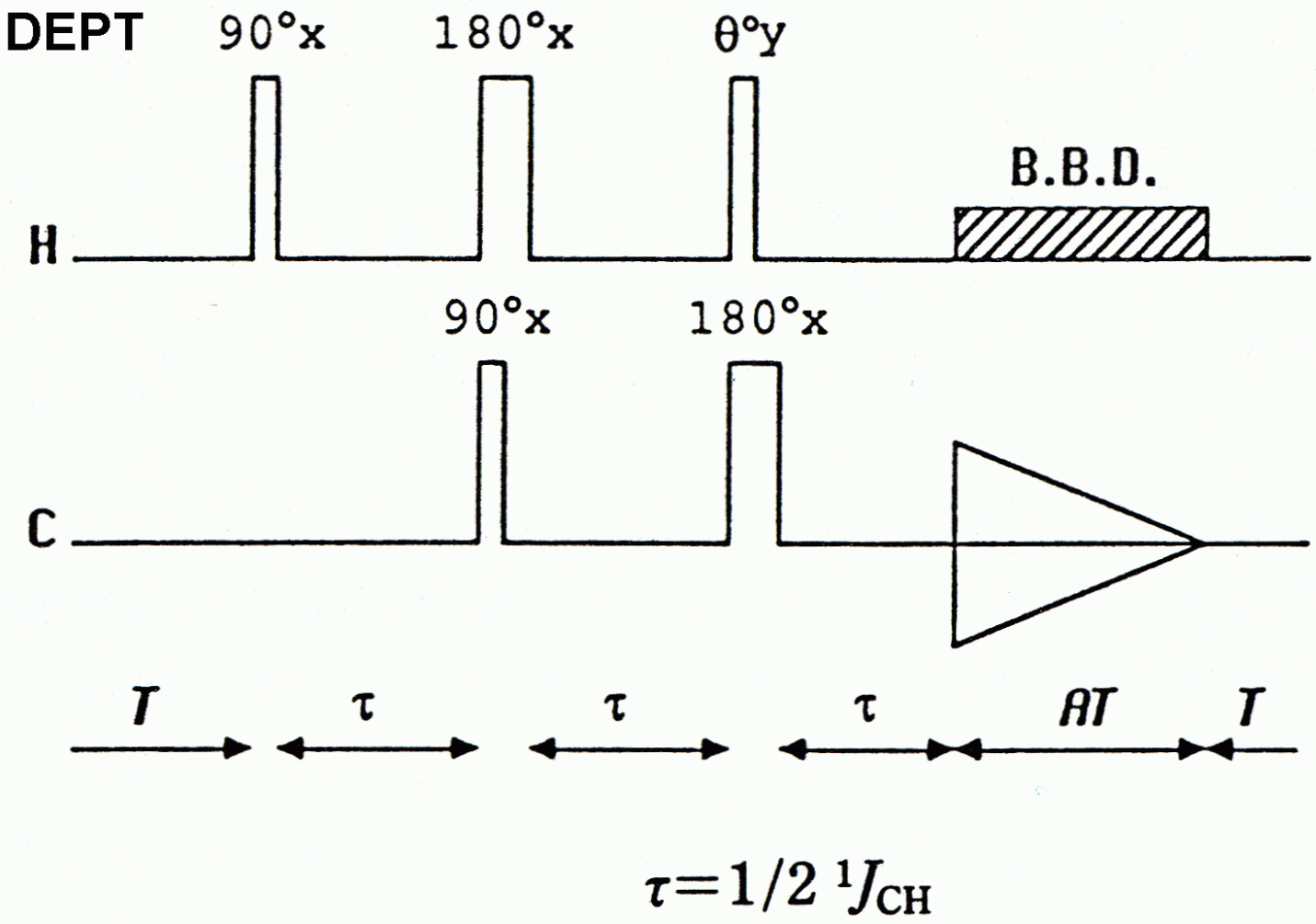
D. ENHANCEMENT BY
POLARIZATION TRANSFER

P2	=	11.20 USEC
P4	=	22.40 USEC
D2	=	19.00 USEC
D4	=	38.00 USEC
D5	=	10.00 SEC
D10	=	3.60 MSEC
L1	=	59
L2	=	72
NA	=	64
SIZE	=	32768
ADC	=	12
AI	=	8
RG	=	10
EM	=	1.00
PA	=	5.2
PB	=	-142.6
LOCK	=	7.26
OF	=	9122.41
SW	=	+/- 10000.0 HZ
DW	=	50 USEC
DE/DW	=	.50
AT	=	819.20 MSEC
SF	=	75.4689574
F2	=	300.0997336

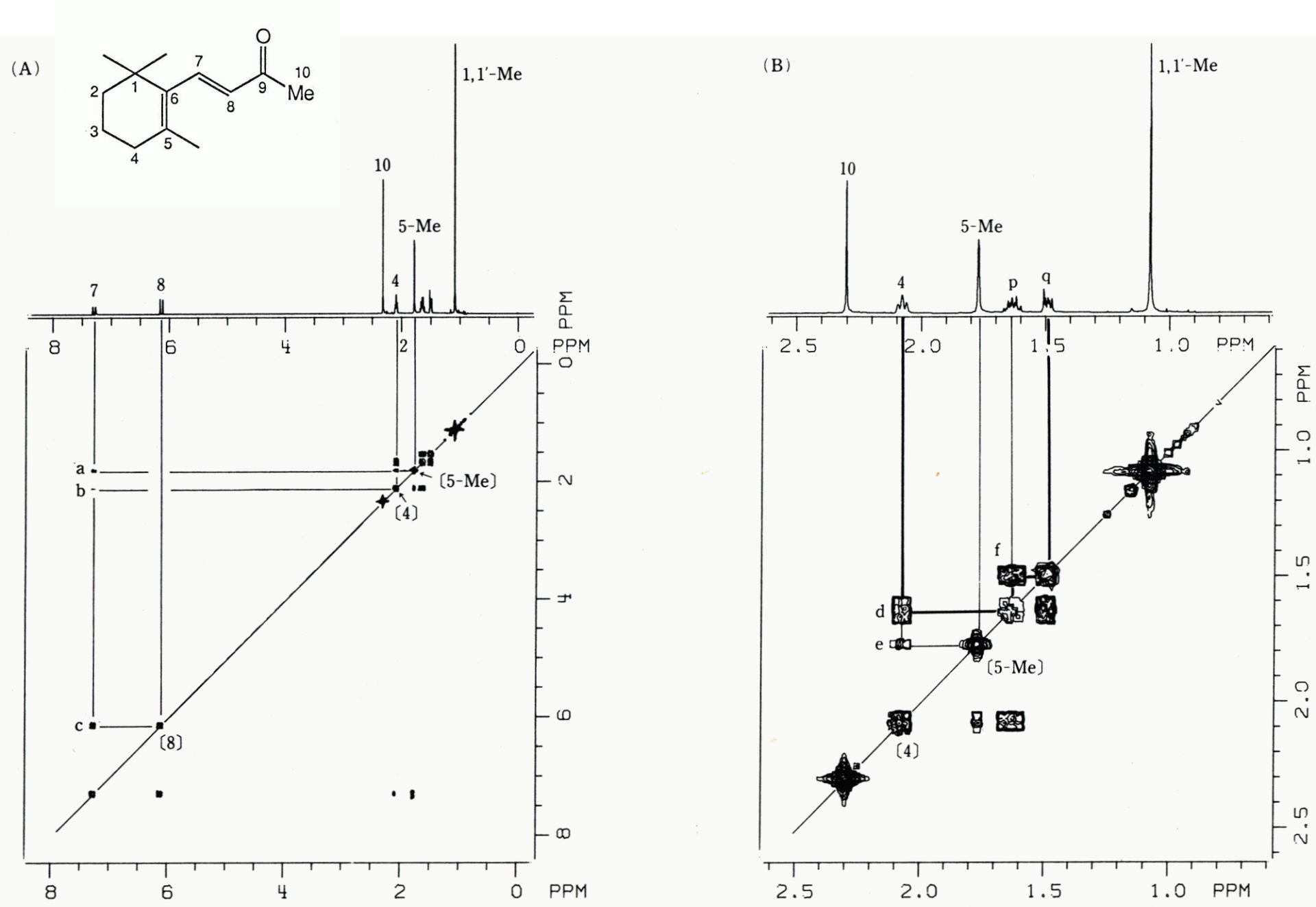
OBS HI PWR = 63
OBS LO PWR = 0
DEC PWR = 59
DEC SCHEME = 3
SCALE = 528.32 HZ/CM
= 7.0004 PPM/CM

FROM 210.00
TO 00 PPM

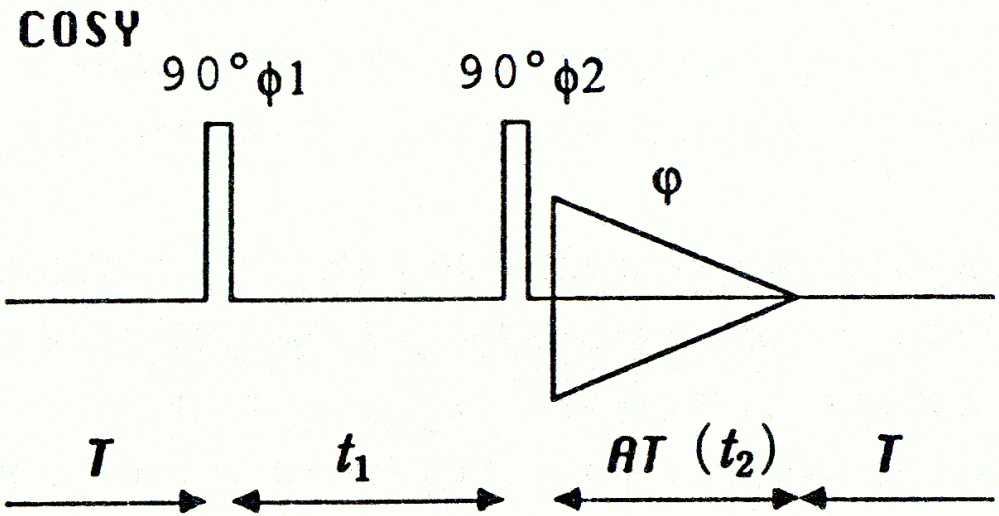
75 MHz ^{13}C-NMR ($CDCl_3$) [KM]



$$\tau = 1/2\ {}^{1}J_{CH}$$



360MHz ^{1}H-NMR ($CDCl_3$) [TI]



(1) Absolute value mode

ϕ1	x	x	x	x
ϕ2	x	y	-x	-y
φ	x	-x	x	-x

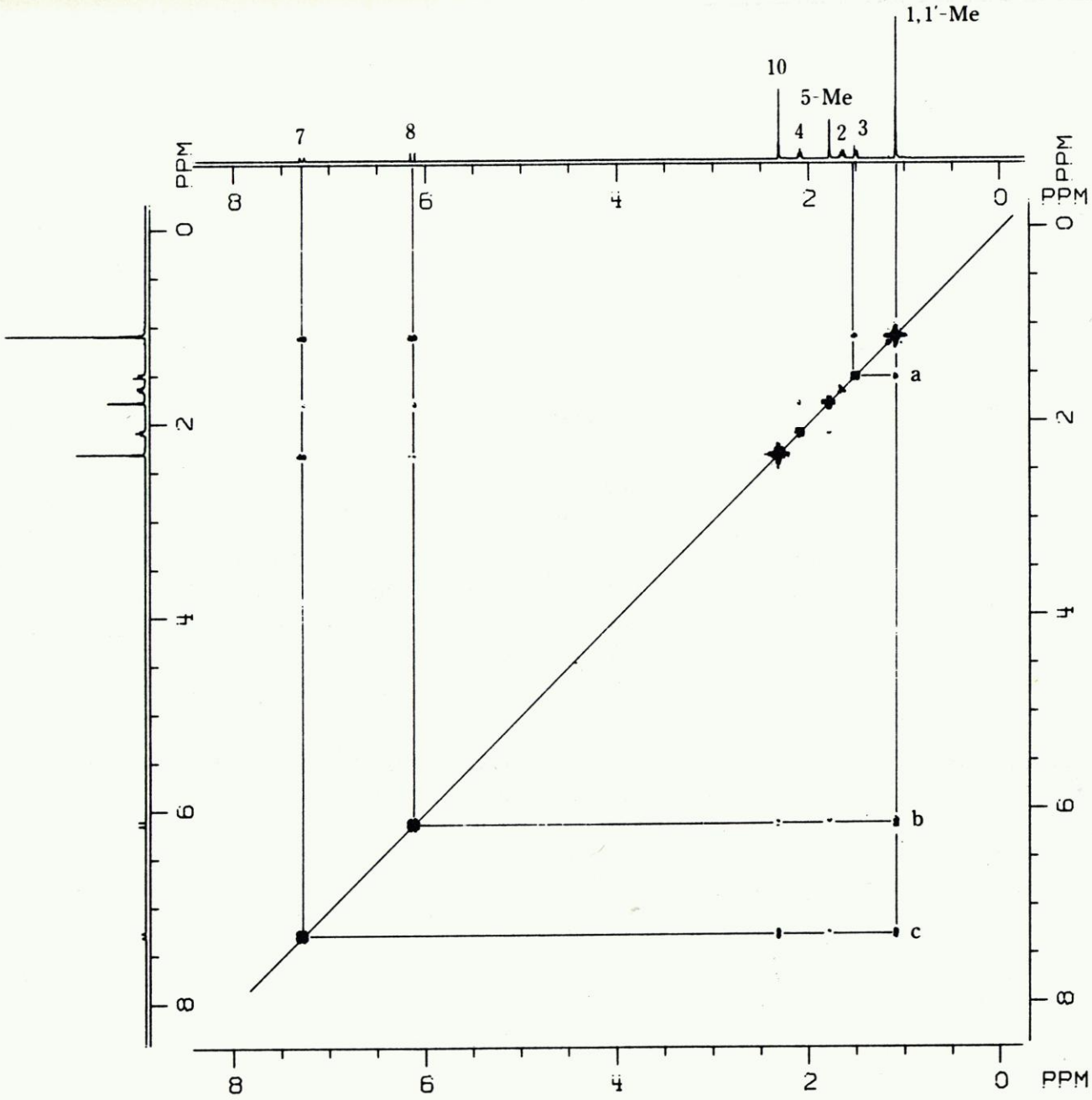
(2) Phase-sensitive mode

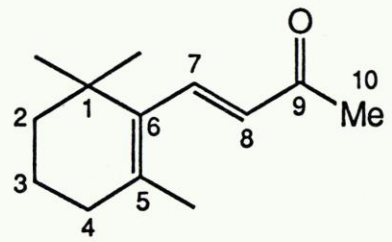
Real t_1

ϕ1	x	x
ϕ2	x	-x
φ	x	x

Imaginary t_1

ϕ1	x	x
ϕ2	y	-y
φ	y	y



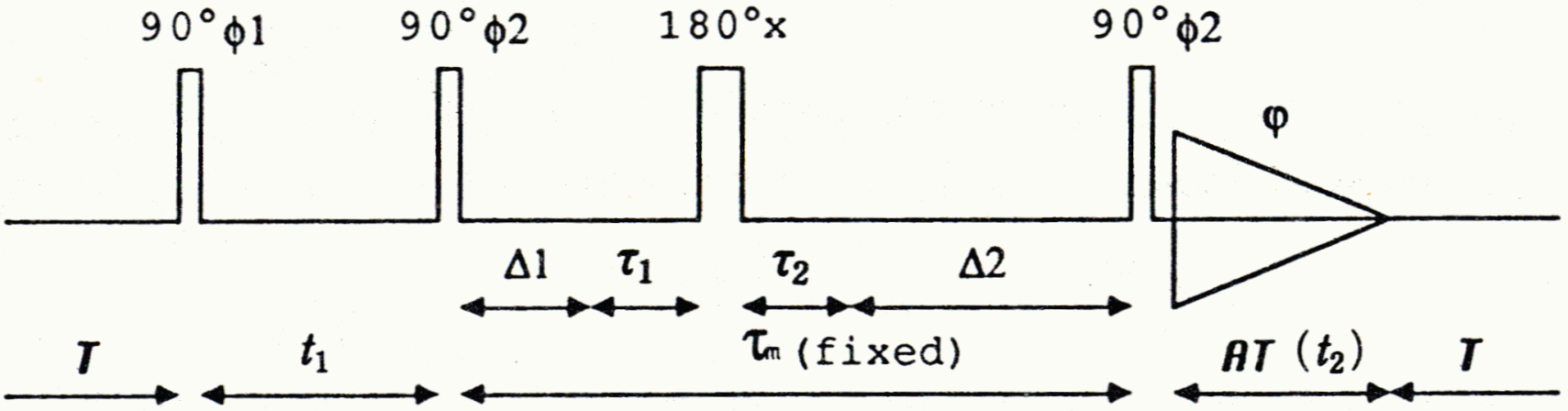



```
2DNOE
MIN NA=16.DP=0.AB=-
DB=DW/4.D6=TM/2
SYM
 P1=        11.00 USEC
 P2=         7.00 USEC
 D5=         1.00  SEC
 D6=         2.00  SEC
 D8=        80.00 USEC

I8=        80.00 USEC

NA =          16
SIZE =      1024
AT =        183.84 MSEC
QPD ON =       0
ABC OFF
BUTTERWORTH FILTER ON
DB ATT.=       3
ADC =   12 BITS
AI =           7
SW = +/- 1562.50
DW =         320
RG =          10 USEC
DE=       320 USEC
TL HIGH POWER ON
OF=     1488.87
SF=     360.055455

SCALE      156.25 HZ/CM
       =    .4338 PPM/CM
CONTOUR LEVEL=16
(Y5=13680)
```

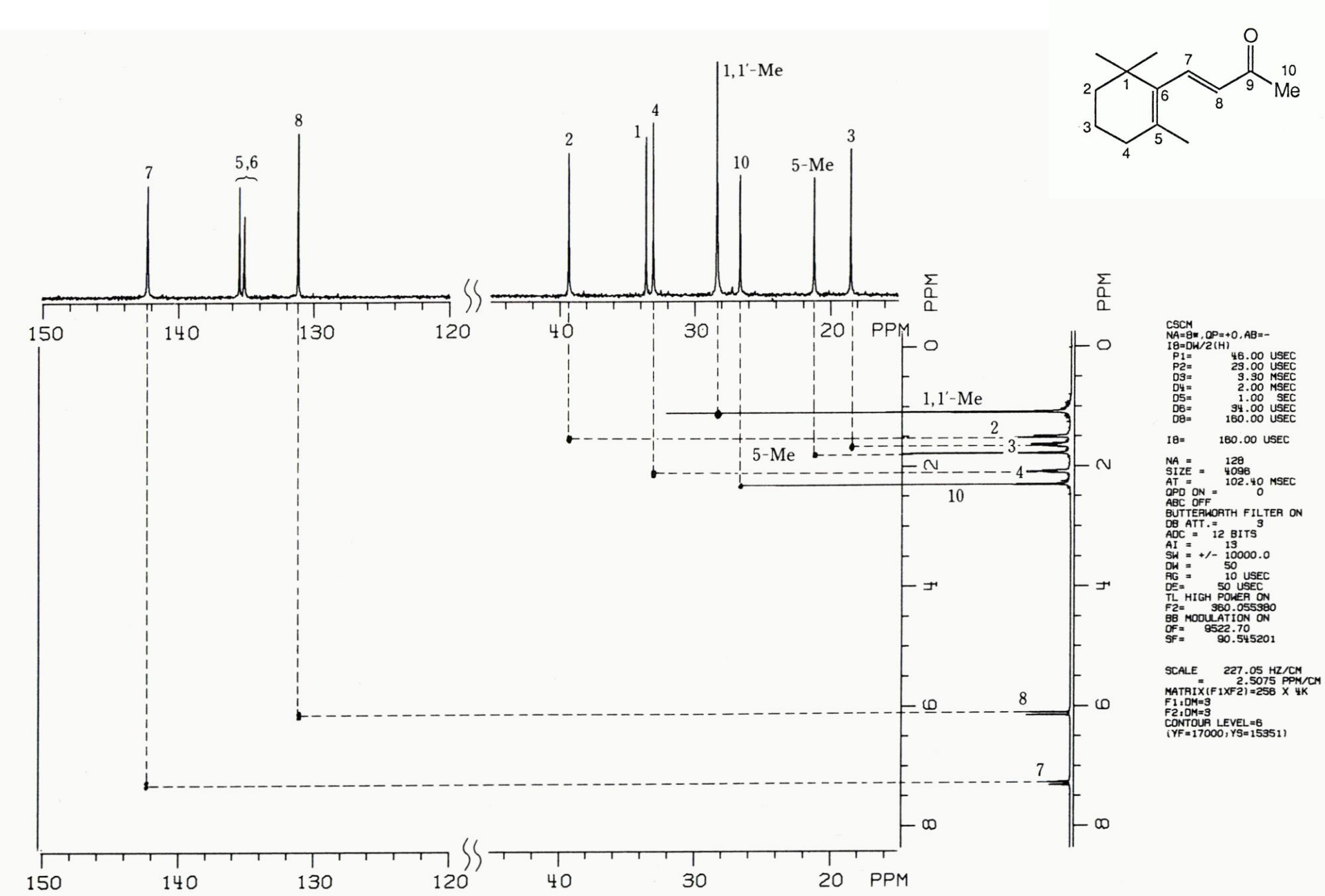
360MHz ^{1}H-NMR ($CDCl_3$) [TI]

NOESY

$\phi 1$	x	x	x	x
$\phi 2$	x	y	-x	-y
φ	x	-x	x	-x

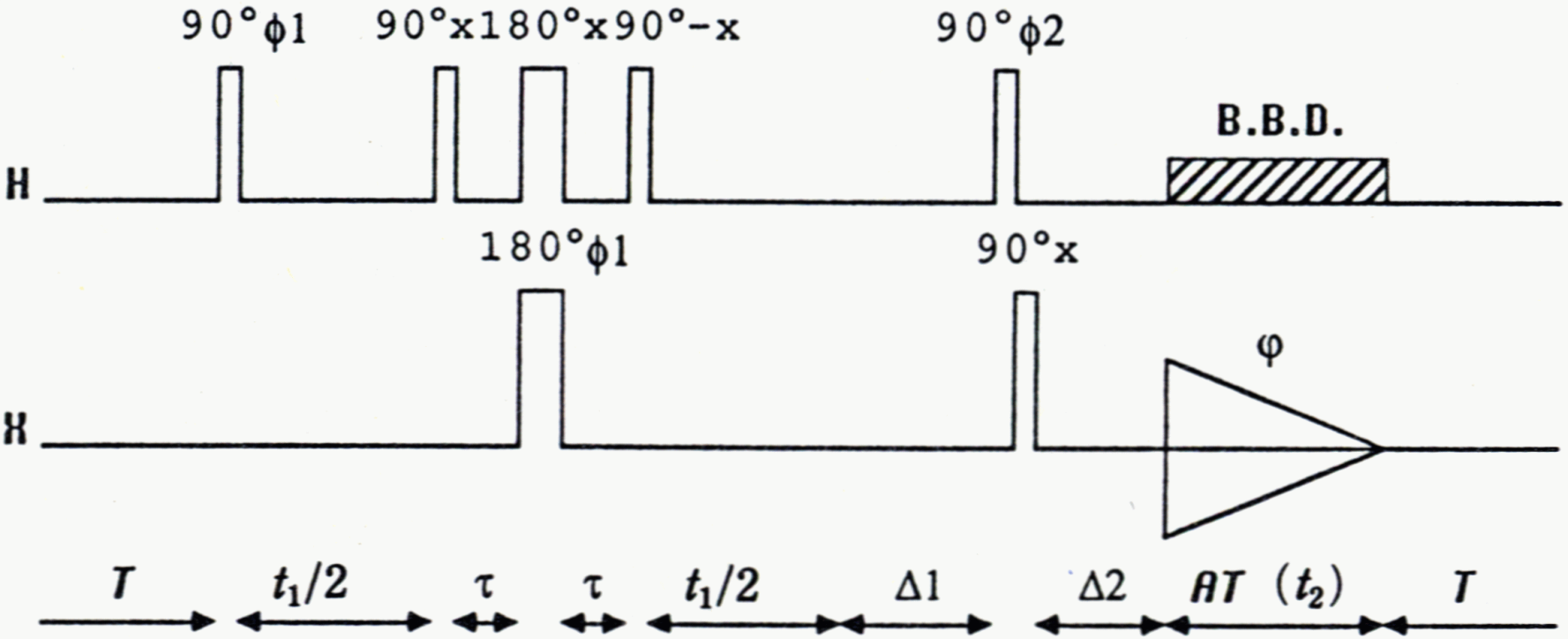


$\tau_1 = t_1/k$, $\tau_2 = \Delta - t_1/k$ or $\tau_1 = \Delta'$ (random), $\tau_2 = \Delta''$ (random)



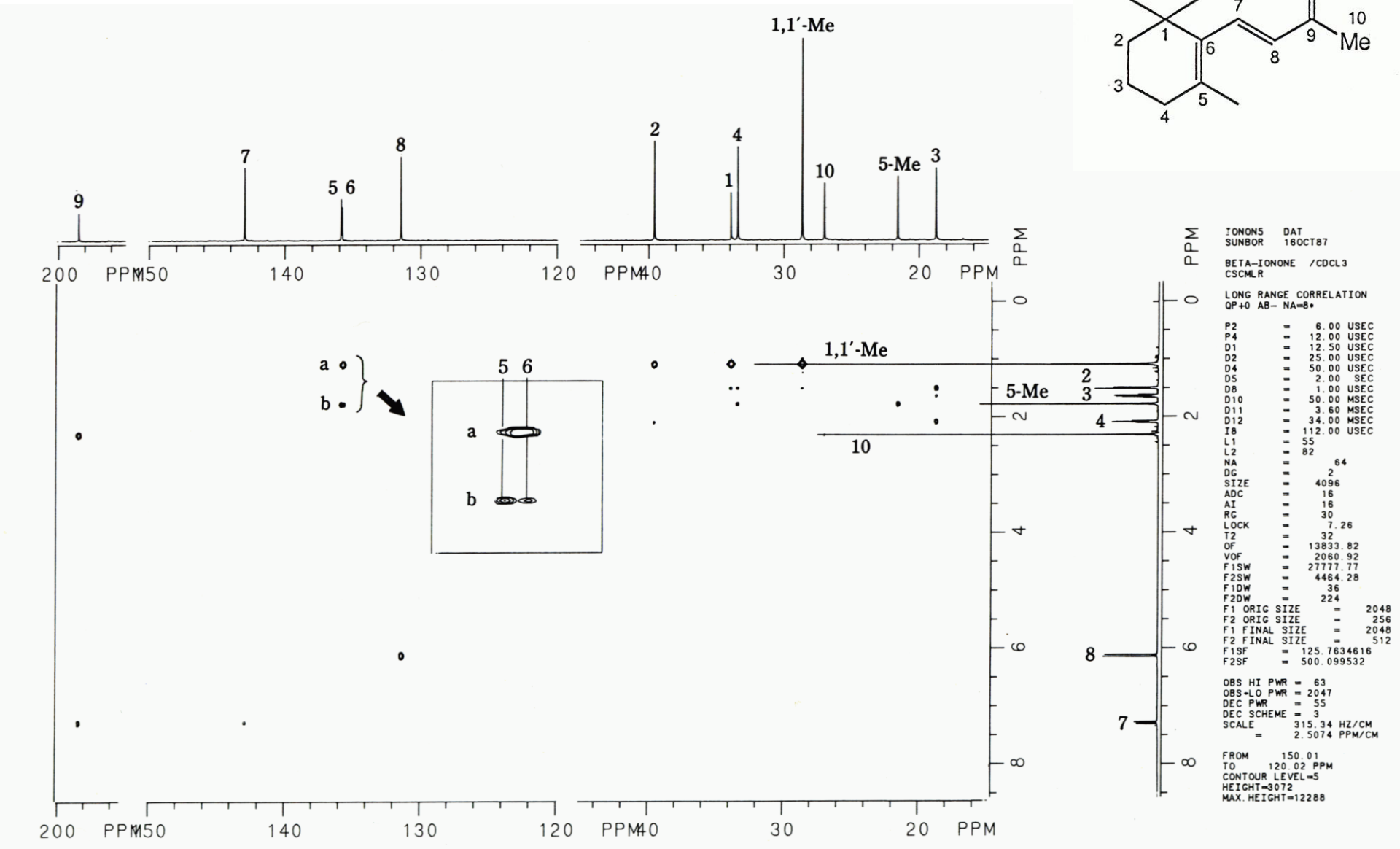
90MHz ^{13}C-NMR ($CDCl_3$) [TI]

HETCOR with F1 decoupling



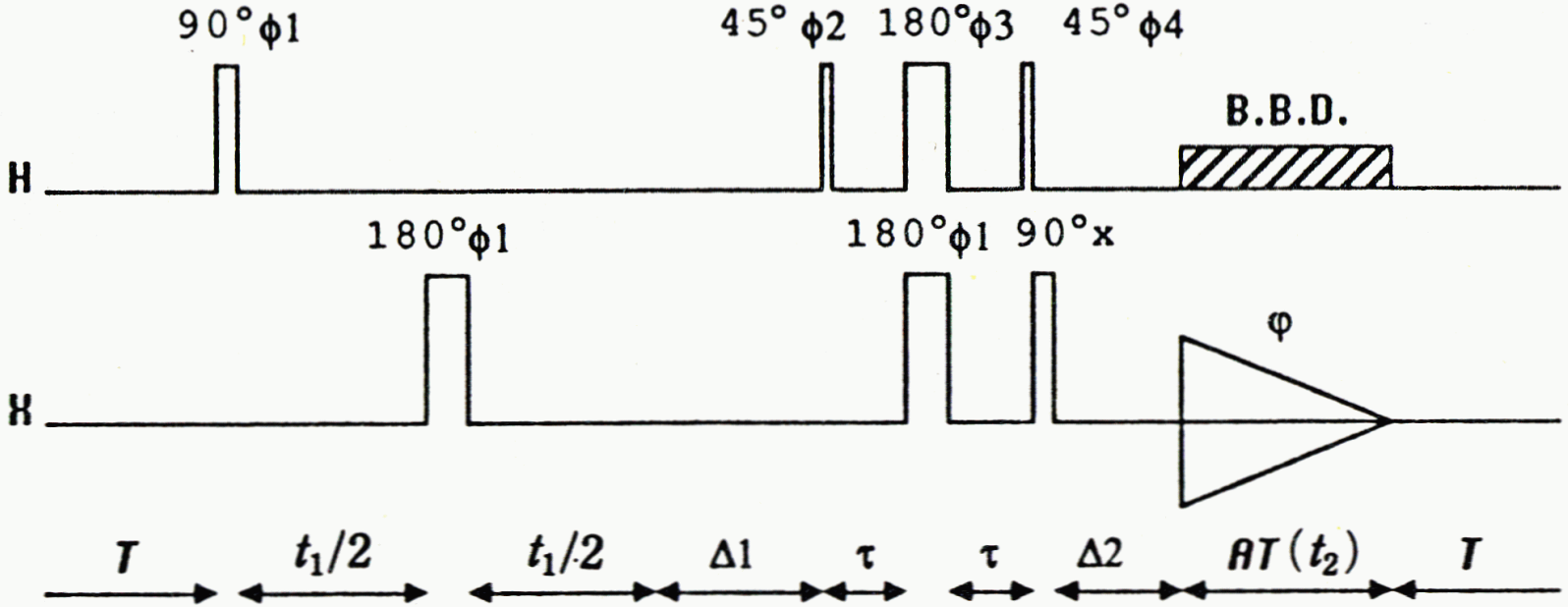
$\Delta_1 = 1/2\ {}^1J_{XH}$, $\Delta_2 = 1/3\ {}^1J_{XH}$, $\tau = 1/2\ {}^1J_{XH}$ $({}^1J_{XH} \gg J_{HH})$

φ1	x	-x	x	-x	-x	x	-x	x
φ2	x	y	-x	-y	-x	-y	x	y
φ	x	-y	-x	y	x	-y	-x	y



125 MHz ^{13}C-NMR ($CDCl_3$) [TI]

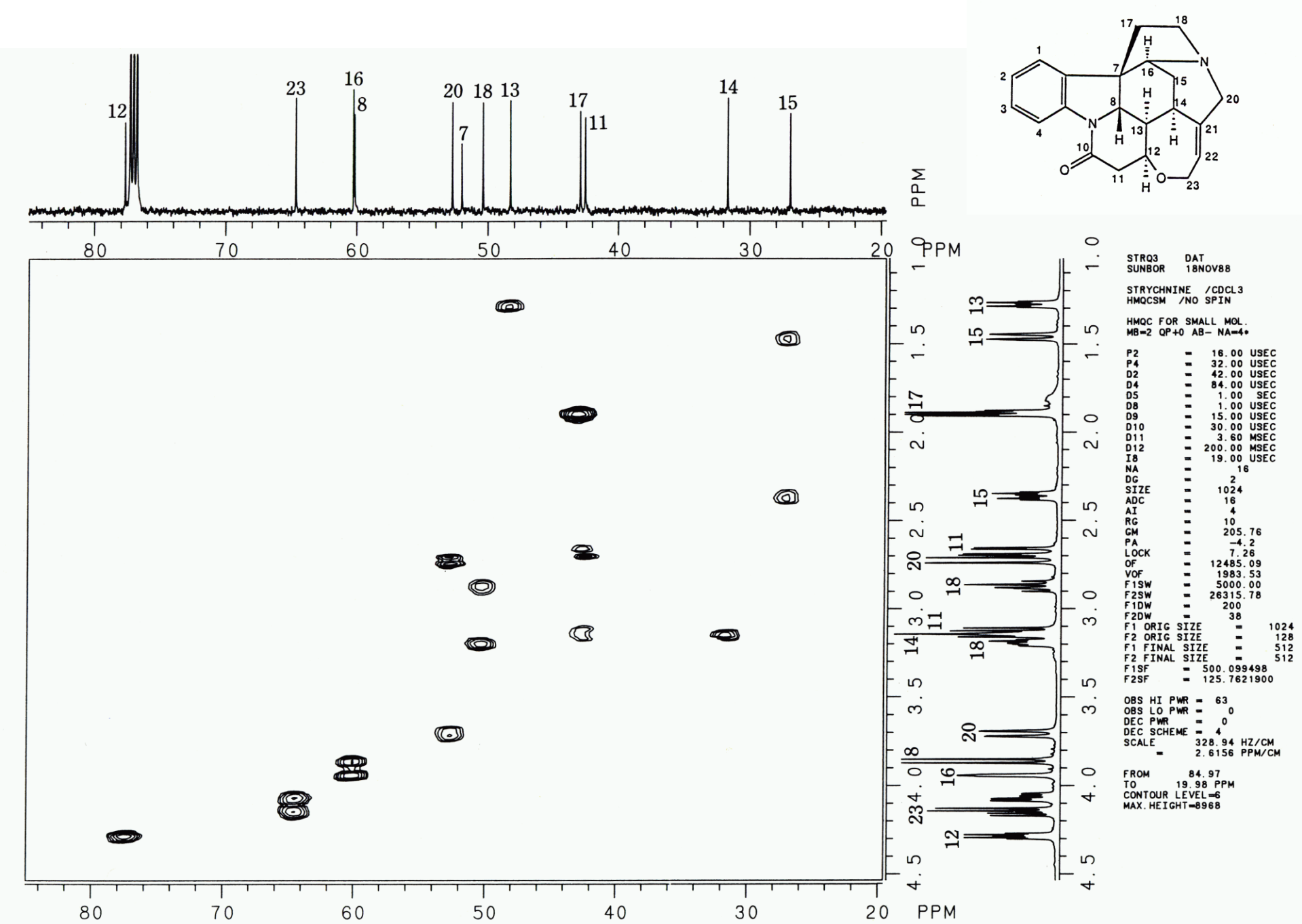
HETCOR-LR for long range spin-spin coupling



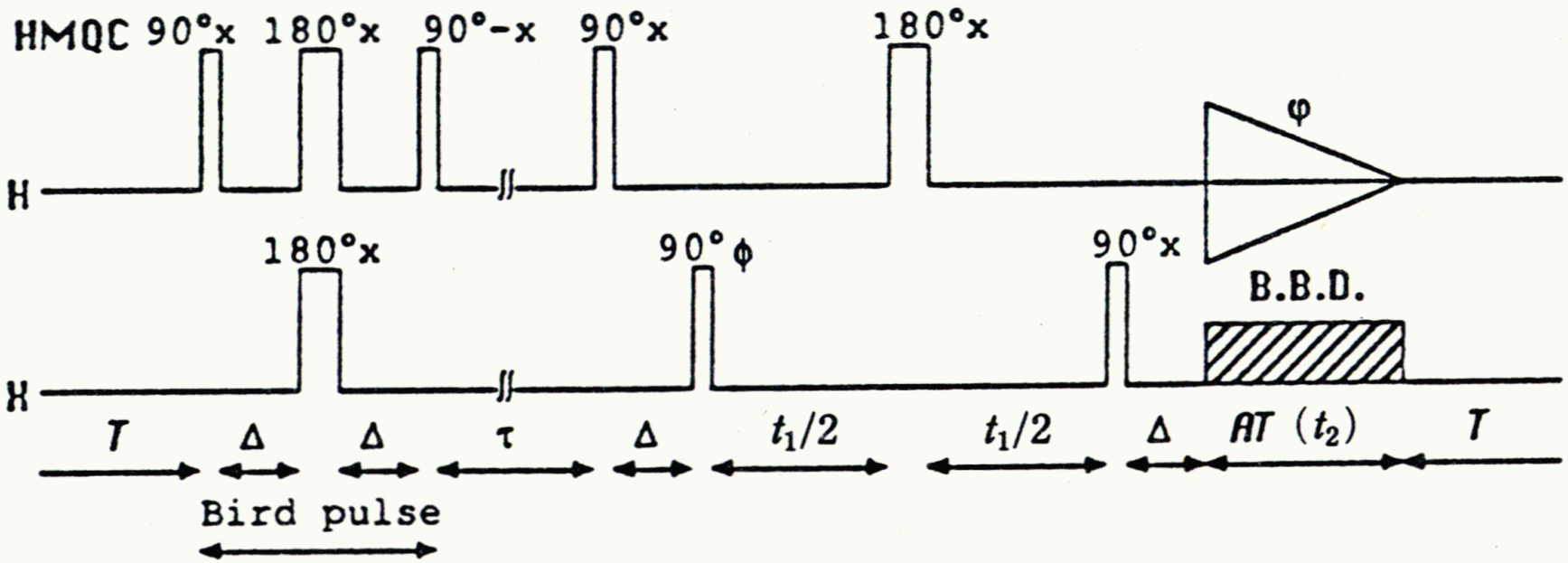
ϕ1	x	-x	x	-x	-x	x	-x	x
ϕ2	x	y	-x	-y	-x	-y	x	y
ϕ3	y	-x	-y	x	-y	x	y	-x
ϕ4	-x	-y	x	y	x	y	-x	-y
φ	x	-y	-x	y	x	-y	-x	y

$\Delta_1 = 1/2\ {}^{1r}J_{XH}$, $\Delta_2 = 1/2\ {}^{1r}J_{XH} \sim 1/3\ {}^{1r}J_{XH}$, $\tau = 1/2\ {}^{1}J_{XH}$

(${}^{1r}J_{XH}$: long range spin-spin coupling between X and H)



500 MHz ^{1}H-NMR ($CDCl_3$) [TI]



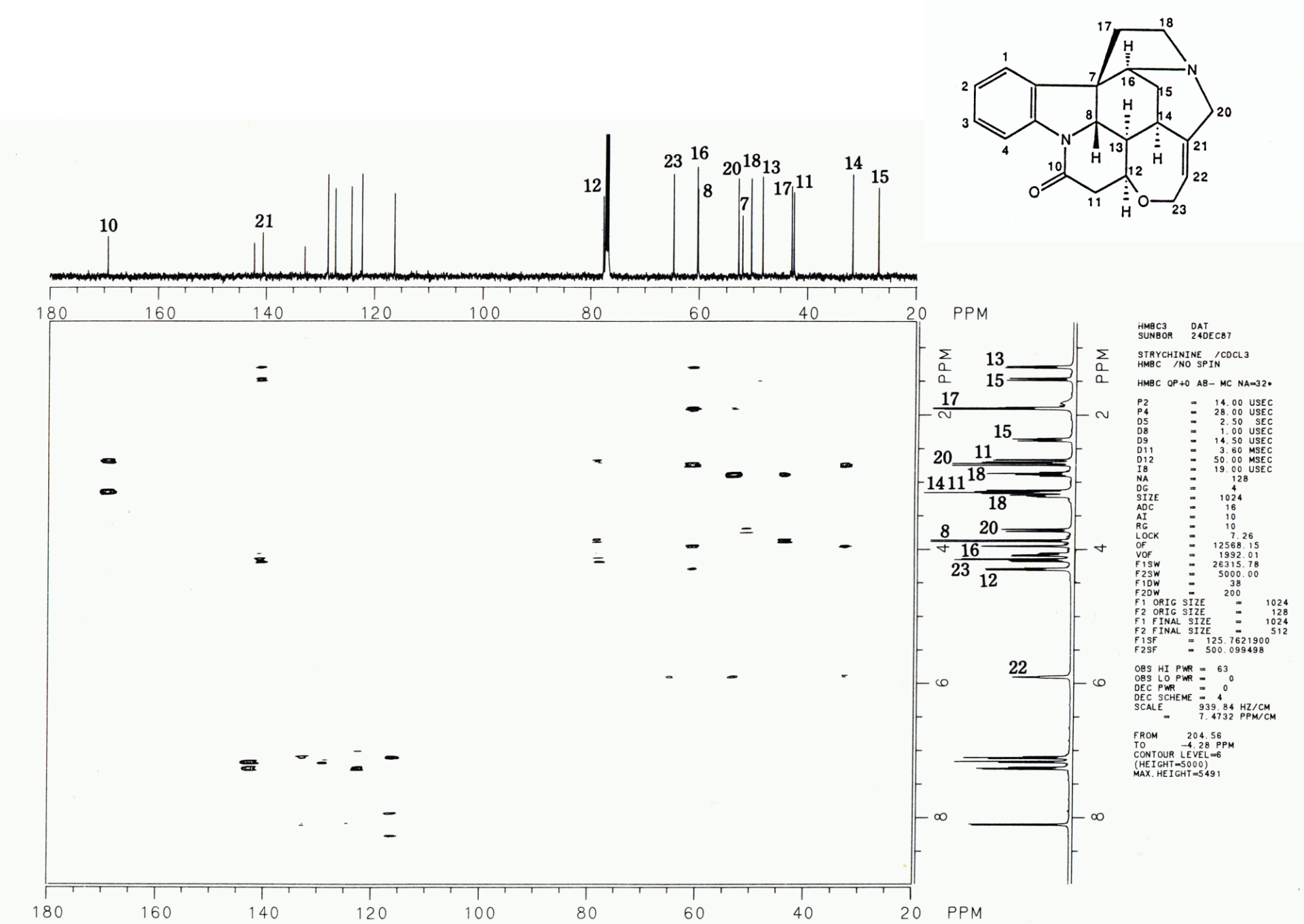
Real t_1 :

ϕ	x	-x	x	-x
φ	x	-x	x	-x

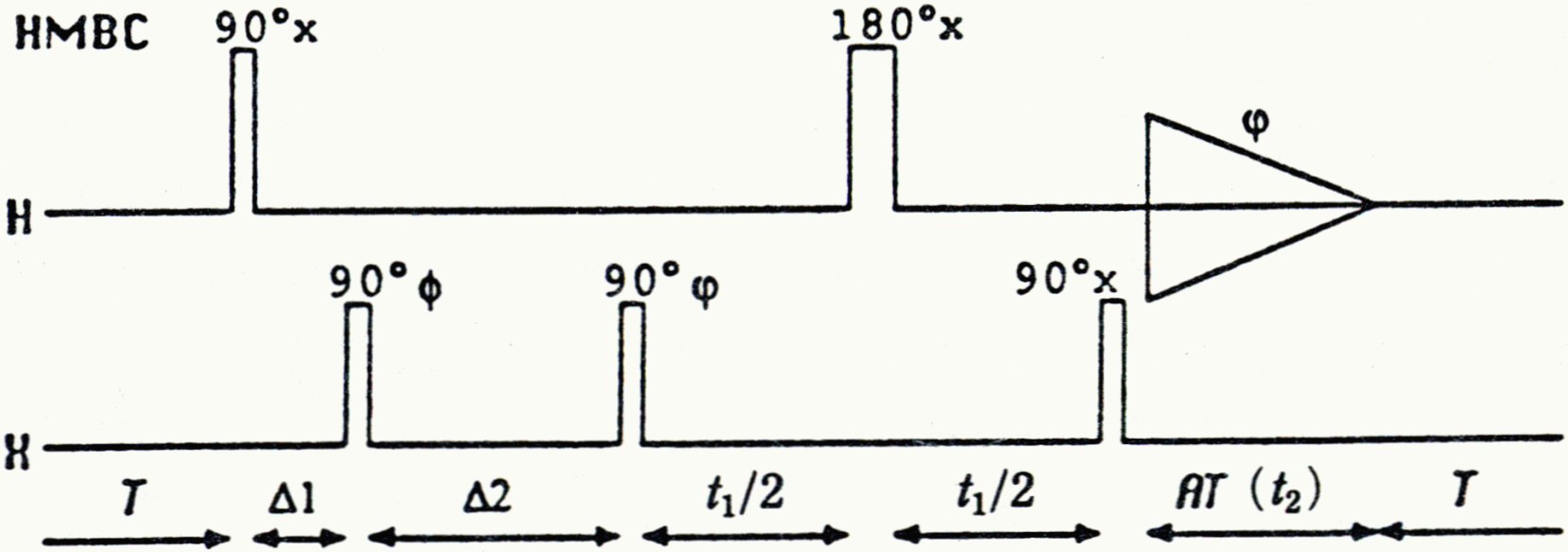
Imaginary t_1 :

ϕ	y	-y	y	-y
φ	x	-x	x	-x

$\Delta_1 = 1/2\ {}^1J_{XH}$, τ depends on T_1 of proton.



500 MHz ^{1}H-NMR ($CDCl_3$) [TI]



ϕ	x	x	-x	-x	x	x	-x	-x
φ	x	-x	x	-x	y	-y	y	-y

$\Delta_1 = 1/2\ {}^1J_{XH}$, Δ_2 is about 60 ms.