JOC The Journal of Organic Chemistry

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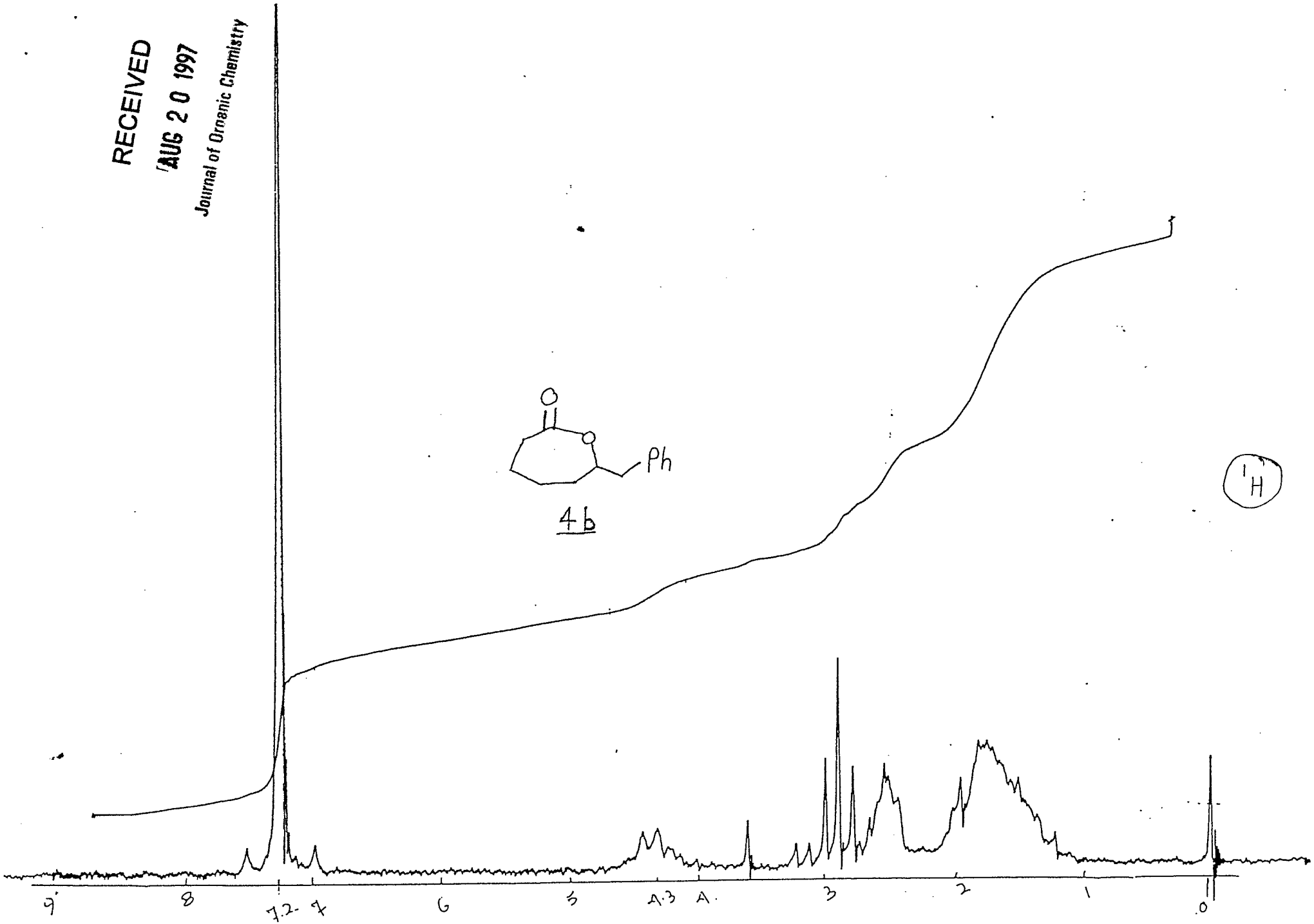
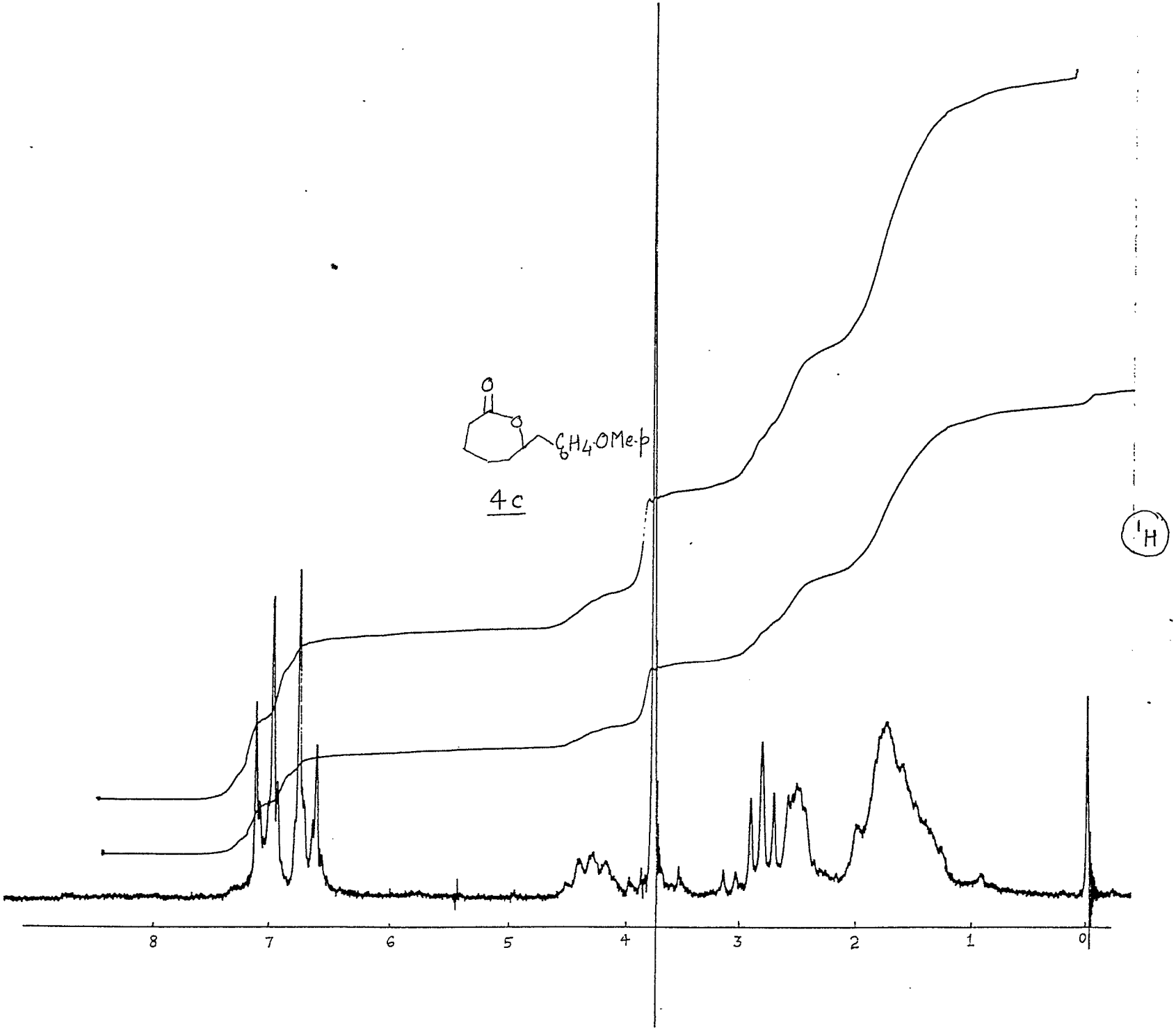
Journal of Organic Chemistry

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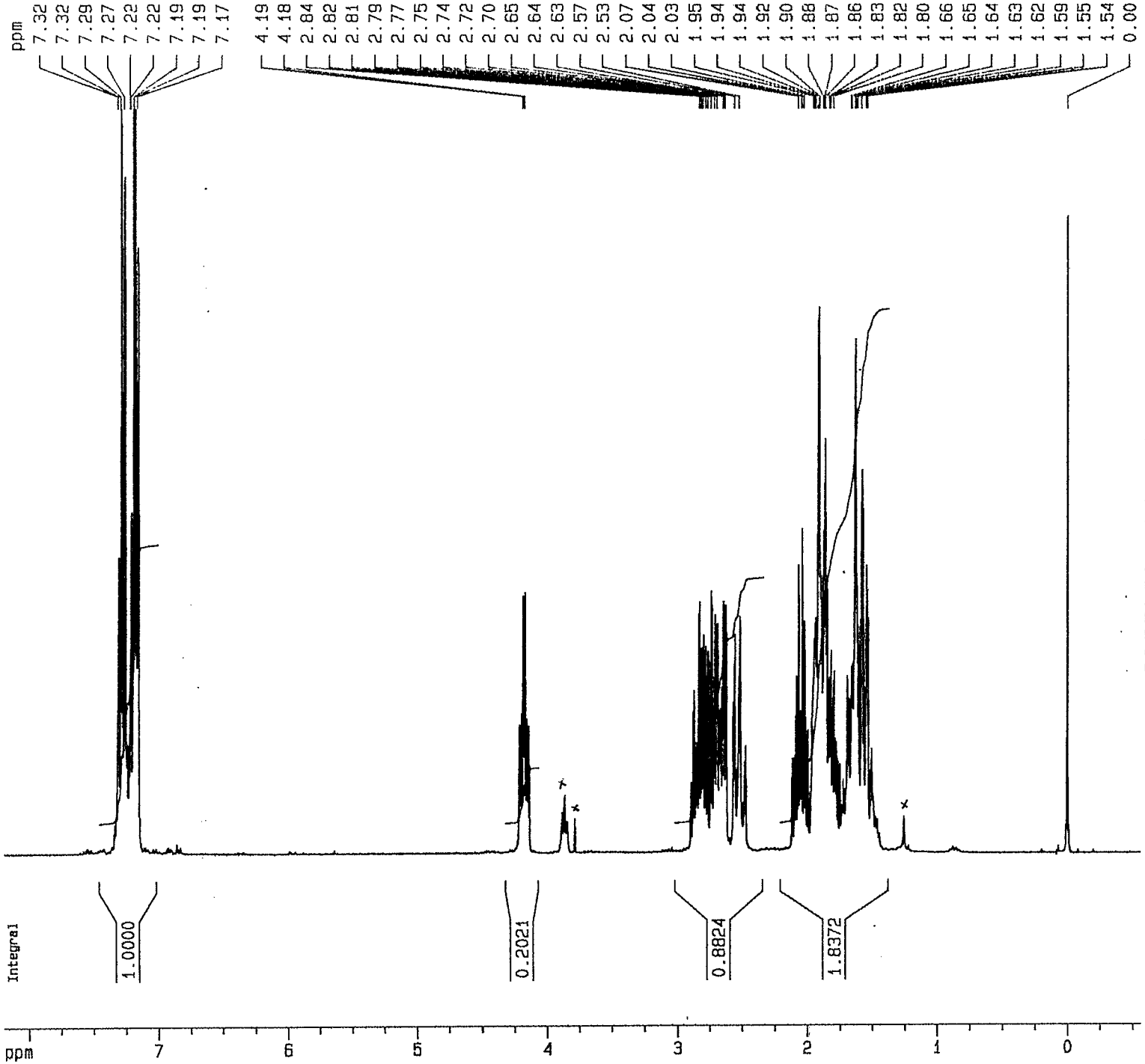

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PROCNO 1

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FIDRES 0.147002 Hz
DW 103.8 us
RG 181
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 us
DE 4.5 us
SFO1 300.1318081 MHz
SWH 4916.56 Hz
TD 32768
NS 16
DS 0

F2 - Processing parameters
SI 16384
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
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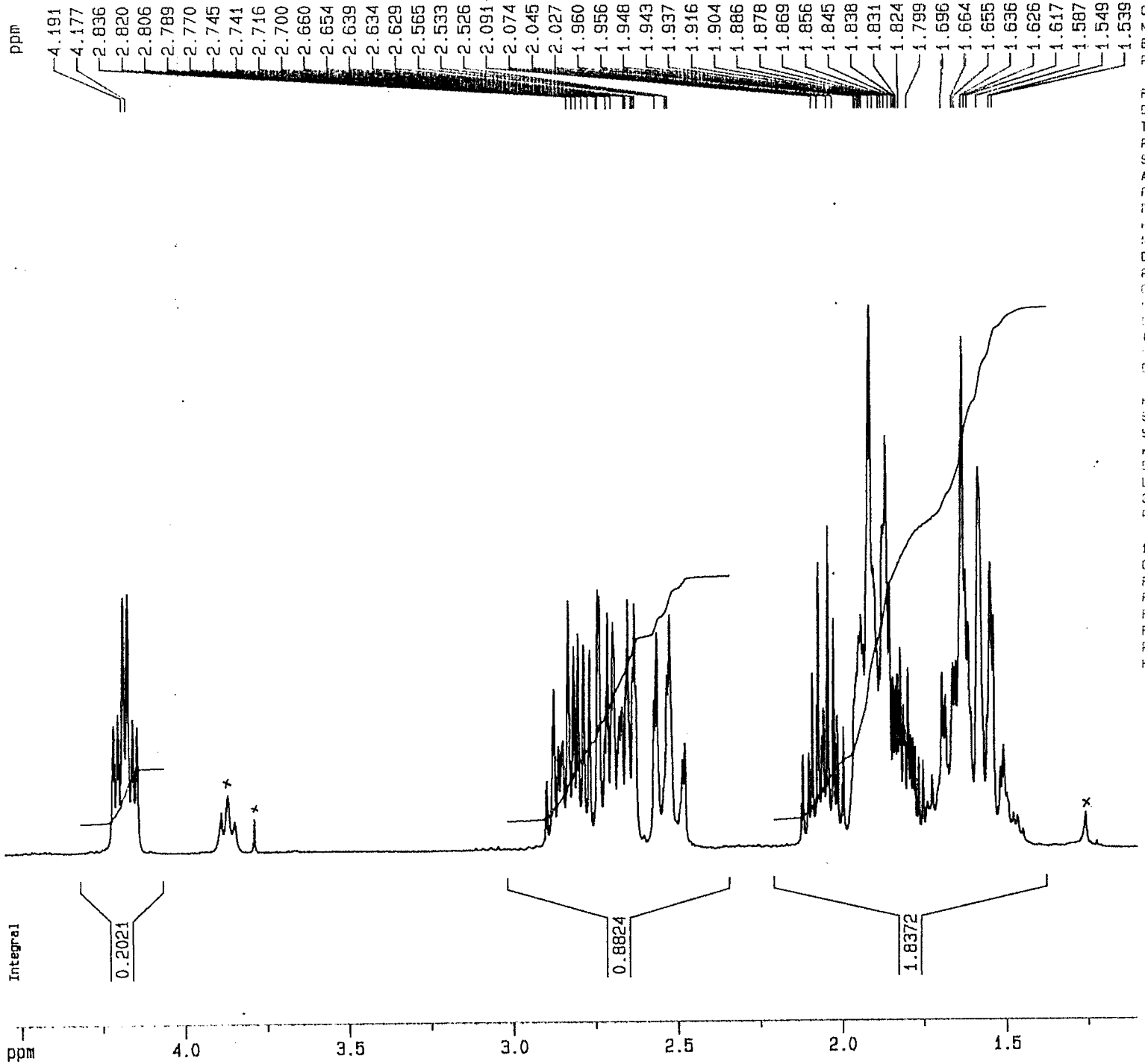
4d

^{1}H



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PROCNO 1

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FIDRES 0.147002 Hz
DW 103.8 us
NS 181
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 us
DE 4.5 us
SW 300.1318061 ppm
SWH 4816.98 Hz
TD 32768
RG 16
DS 0

F2 - Processing parameters
SI 16384
SF 300.1300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
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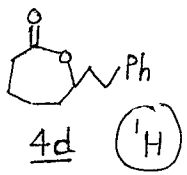

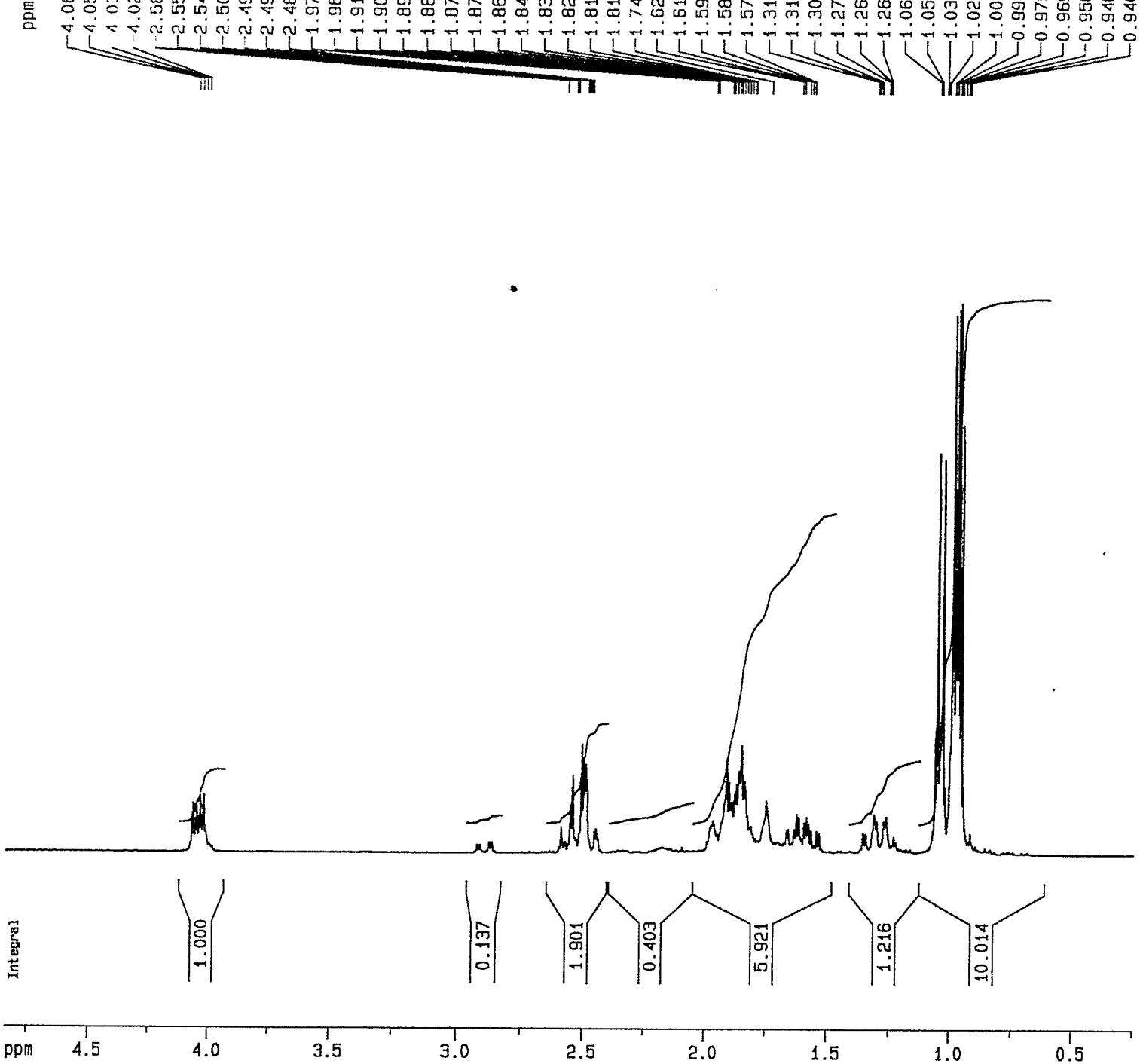
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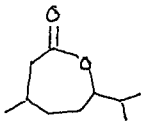

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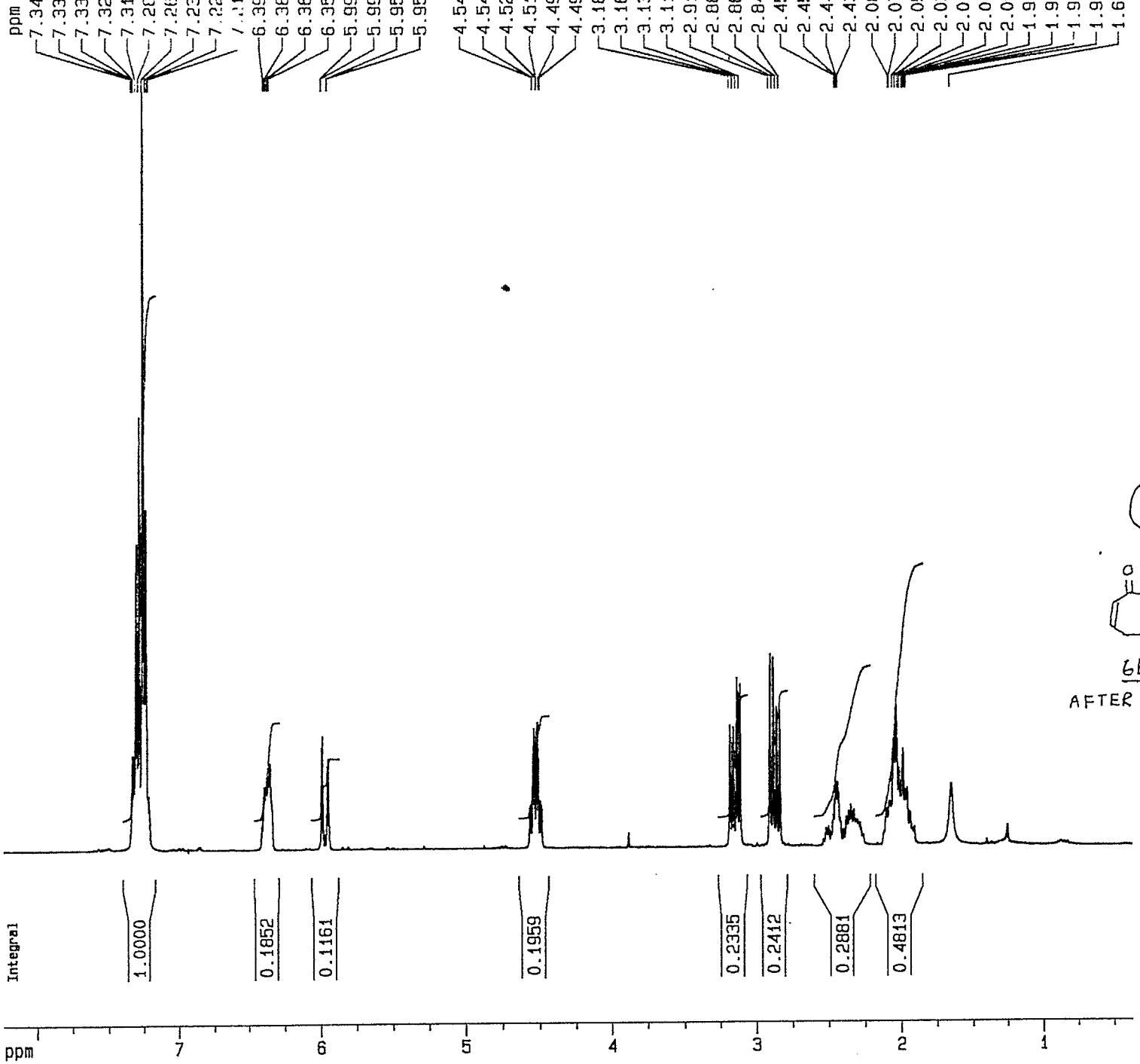
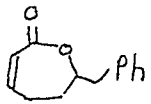


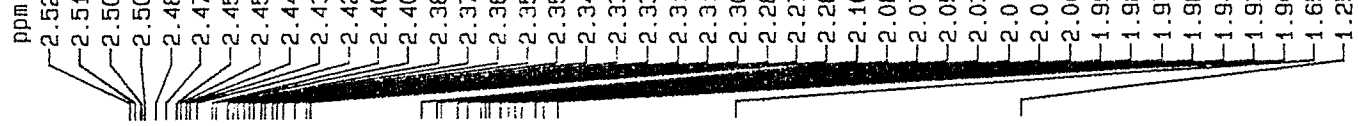
5-7

^{1}H



6b ē D

AFTER EQUILIBRATION IN $CDCl_3$



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EXPNO	1
PROCNO	1

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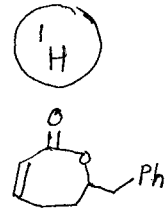
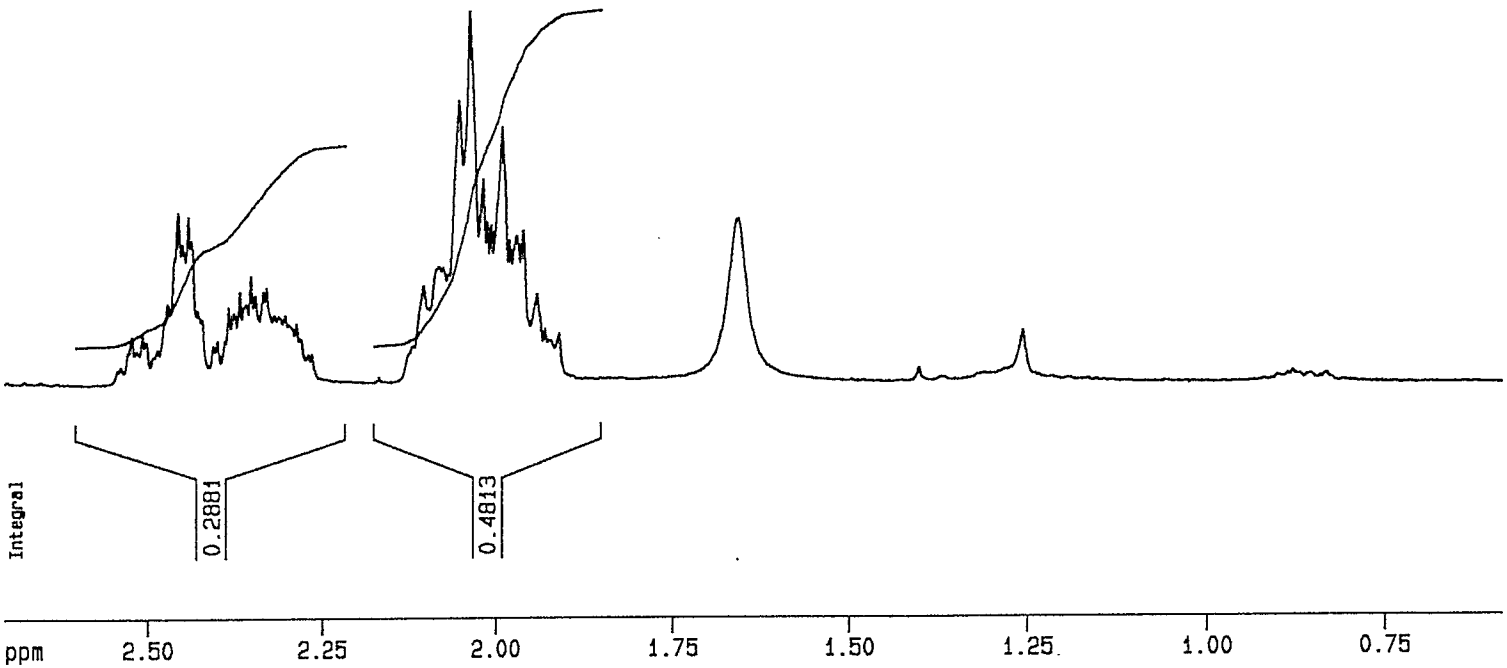
AQ	3.4015260 se
FIDRES	0.147002 H:
DW	103.8 us
RG	90.5
NUCLEUS	1H
D1	3.0000000 se
P1	6.9 us
DE	4.5 us
SFO1	300.1318383 MH
SWH	4816.96 H:
TD	32768
NS	16
DS	0



6b c̄ D

After equilibration in $CDCl_3$

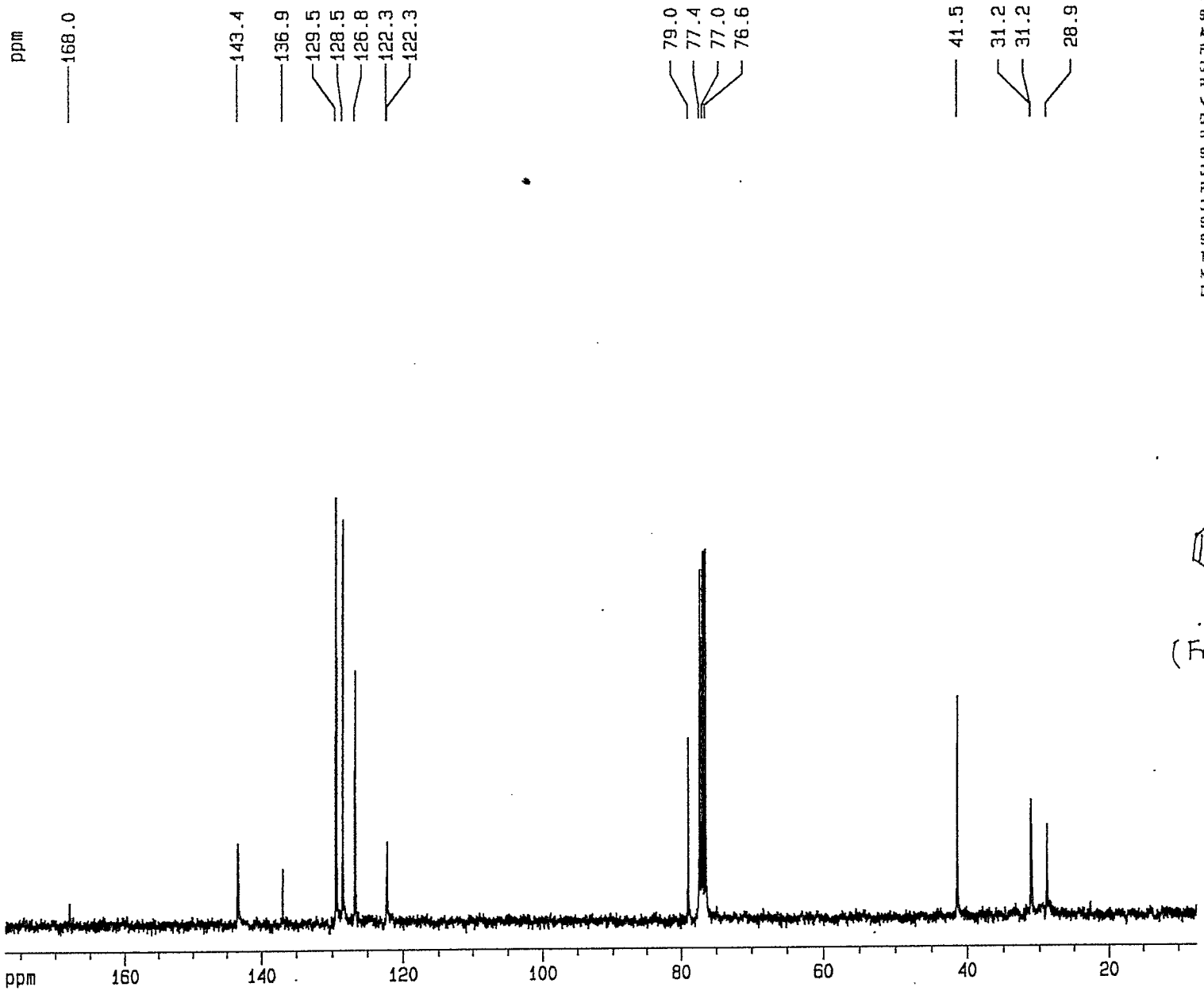
J-7



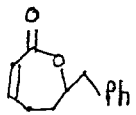
NAME	07.HM
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date	960708
Time	14.19
PULPROG	zgdc
SOLVENT	CDCl3
AQ	0.8356350 s
FIDRES	0.598384 H
DW	25.5 u
RG	20642.5
NUCLEUS	off
D11	0.0300000 s
P31	108.0 u
SFO2	300.1310073 M
D1	1.0000000 s
P1	5.0 u
DE	4.5 u
SFO1	75.4760000 M
SWH	19607.84 H
TD	32768
NS	3871
DS	0



^{13}C



6b with D.
(From equilibrium expt.)

DR V K YADAV
J-7

GAUNIYAL H.M.

NAME	J7.HM
EXPNO	3
PROCNO	1

F2 - Acquisition Parameter

Date	960708
Time	14.19
PULPROG	zgdc
SOLVENT	CDCl3
AQ	0.8356350 s
FIDRES	0.598384 H
DW	25.5 u
RG	20642.5
NUCLEUS	off
D11	0.0300000 s
P31	108.0 u
SFO2	300.1310073 M
D1	1.0000000 s
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DE	4.5 u
SFO1	75.4760000 M
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TD	32768
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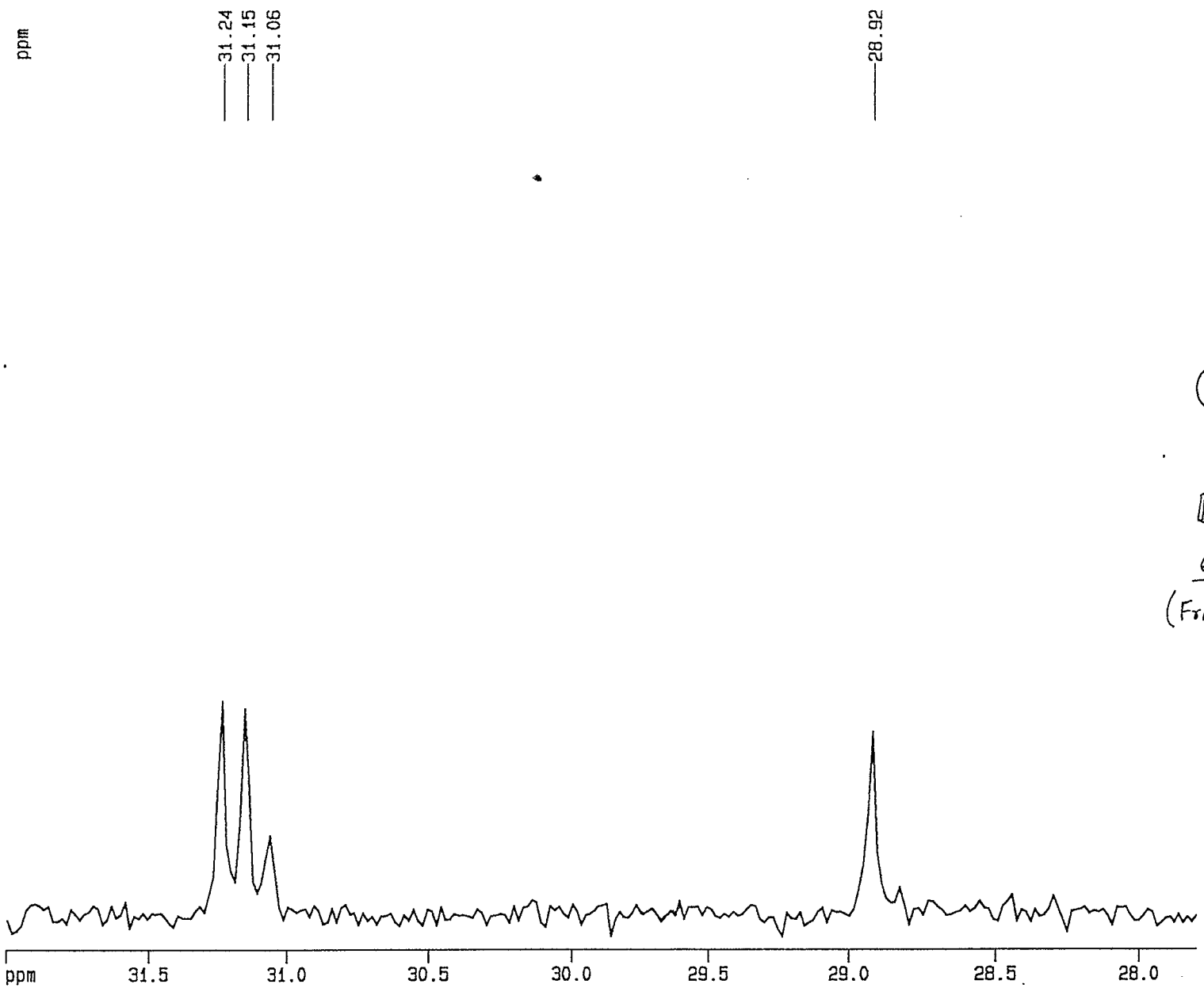
DS	0



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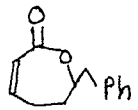

6b with D

(From equilibrium expt.)

DR V K YADAV
J-7

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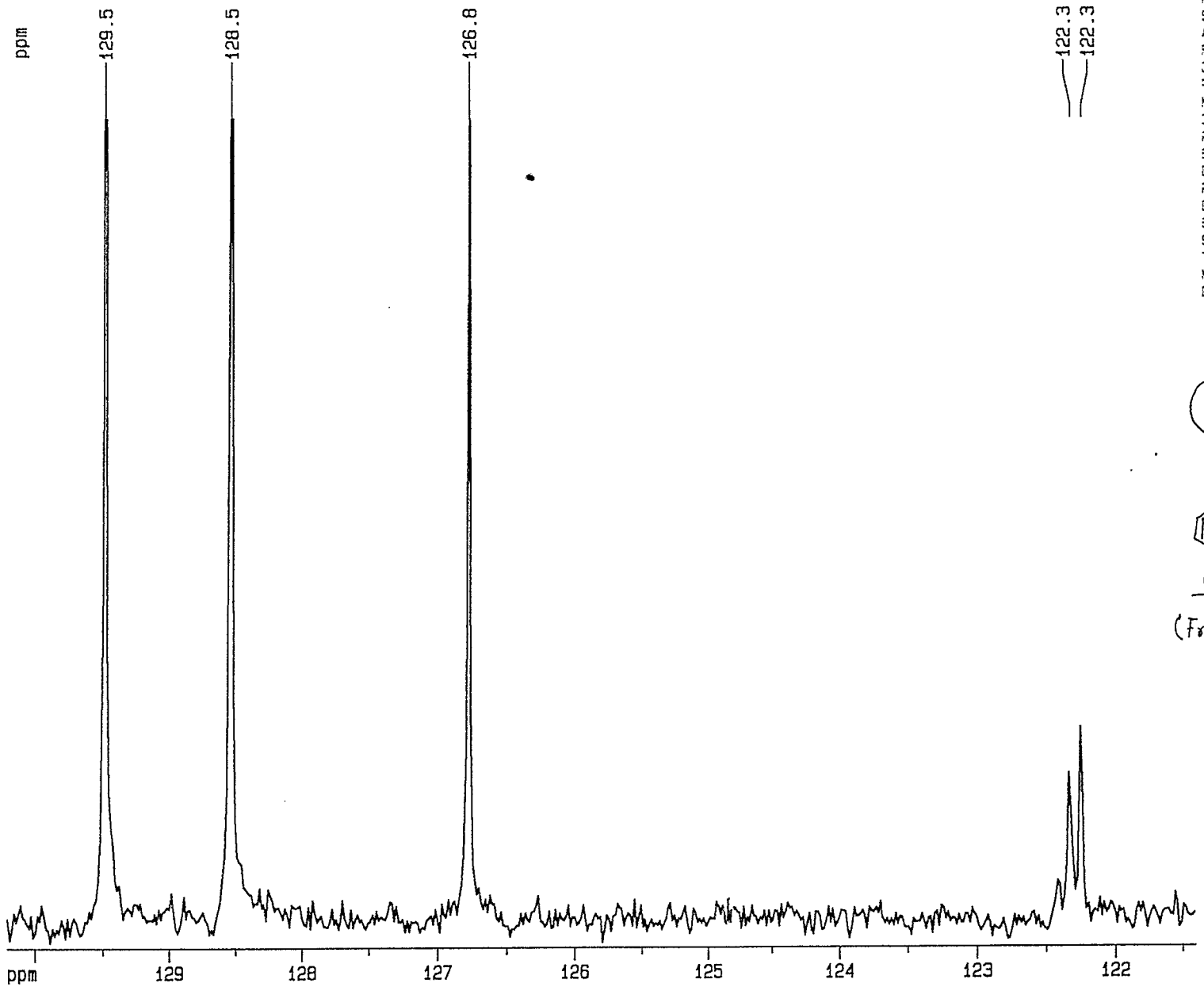

NAME	J7.HM
EXPNO	3
PROCNO	1

F2 - Acquisition Paramete

Date	960708
Time	14.19
PULPROG	zgdc
SOLVENT	CDCl3
AQ	0.8356350 s
FIDRES	0.598384 H
DW	25.5 u
RG	20642.5
NUCLEUS	off
D11	0.0300000 s
P31	108.0 u
SFO2	300.1310073 M
D1	1.0000000 s
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SFO1	75.4760000 M
SWH	19607.84 H
TD	32768
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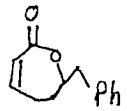
DS	0

^{13}C

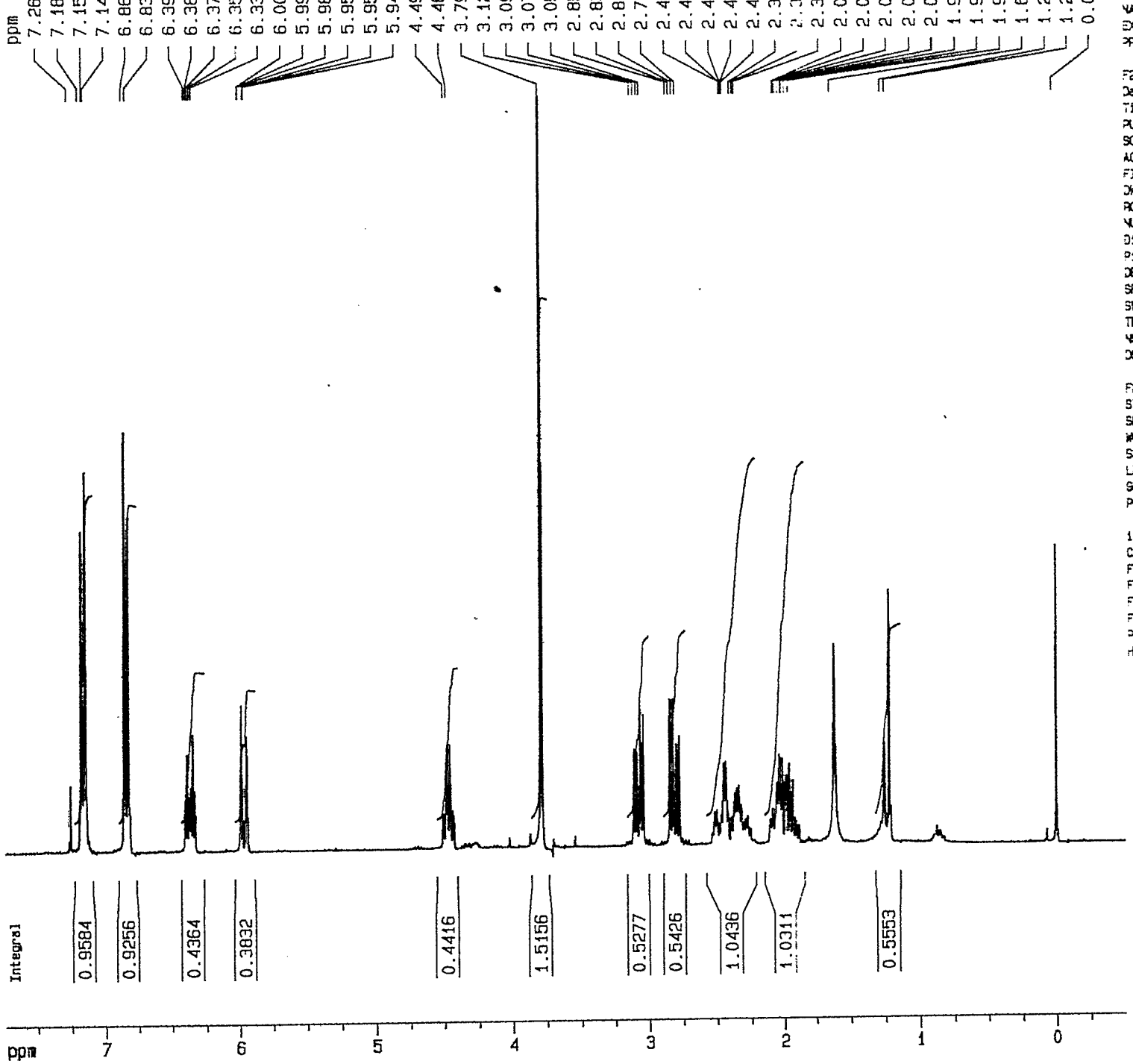


6b with D

(From equilibrium expt.)

DR V K YADAV
J-7

GAUNIYAL H.M.



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SOLVENT CDCl3
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FIDRES 0.147002 H
DW 103.8 u
RG 181
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4818.96 H
TD 32768
NS 16
DS 0

F2 - Processing parameter
SI 16384
SF 300.1300055 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 c
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F2P -0.492 p
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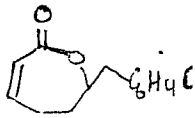
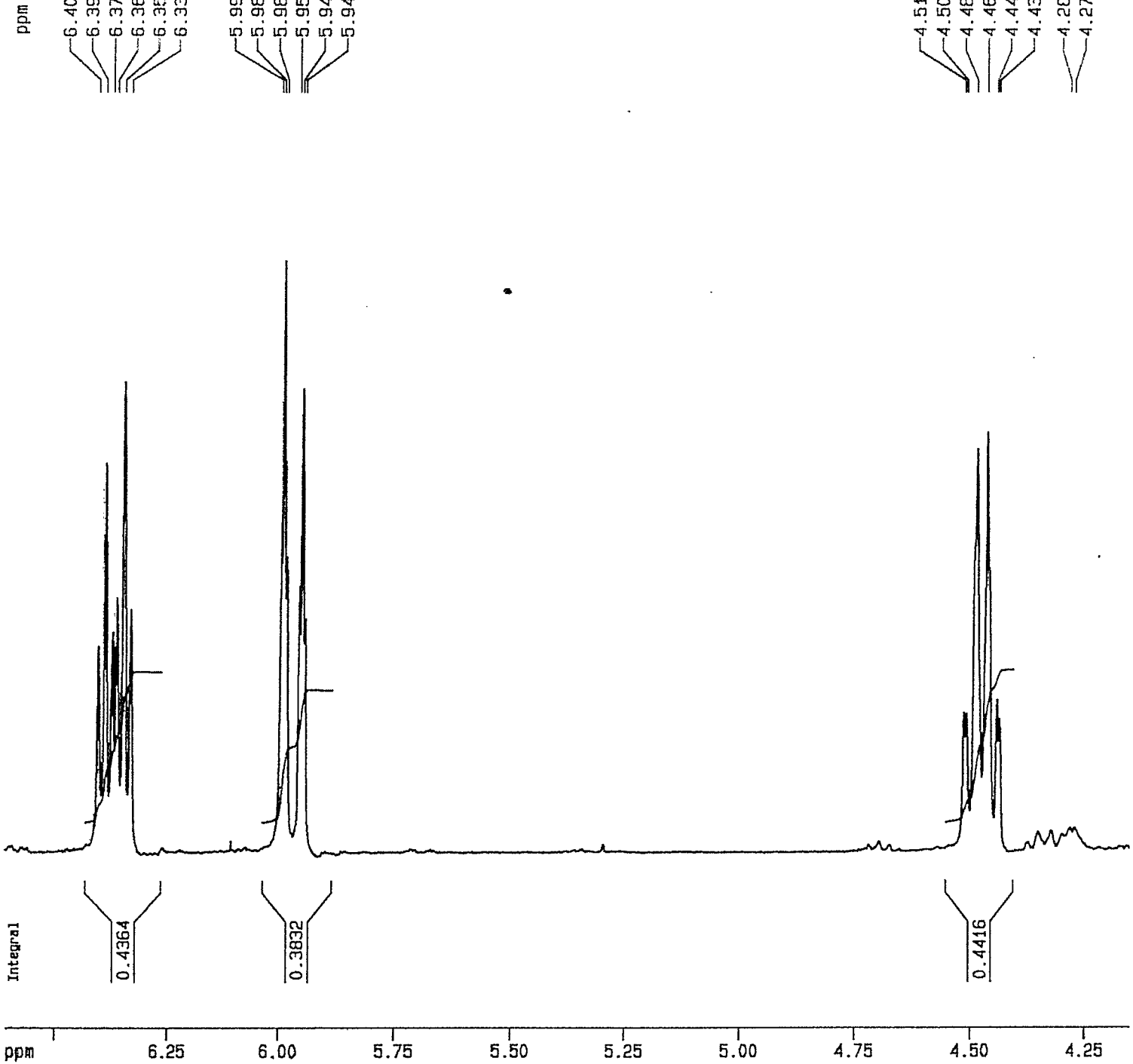


6c

^{1}H



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PROCNO 1

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FIDRES 0.147002 Hz
DW 103.8 us
RG 181
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P1 6.9 us
DE 4.5 us
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TD 32768
NS 16
DS 0

F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
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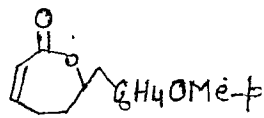
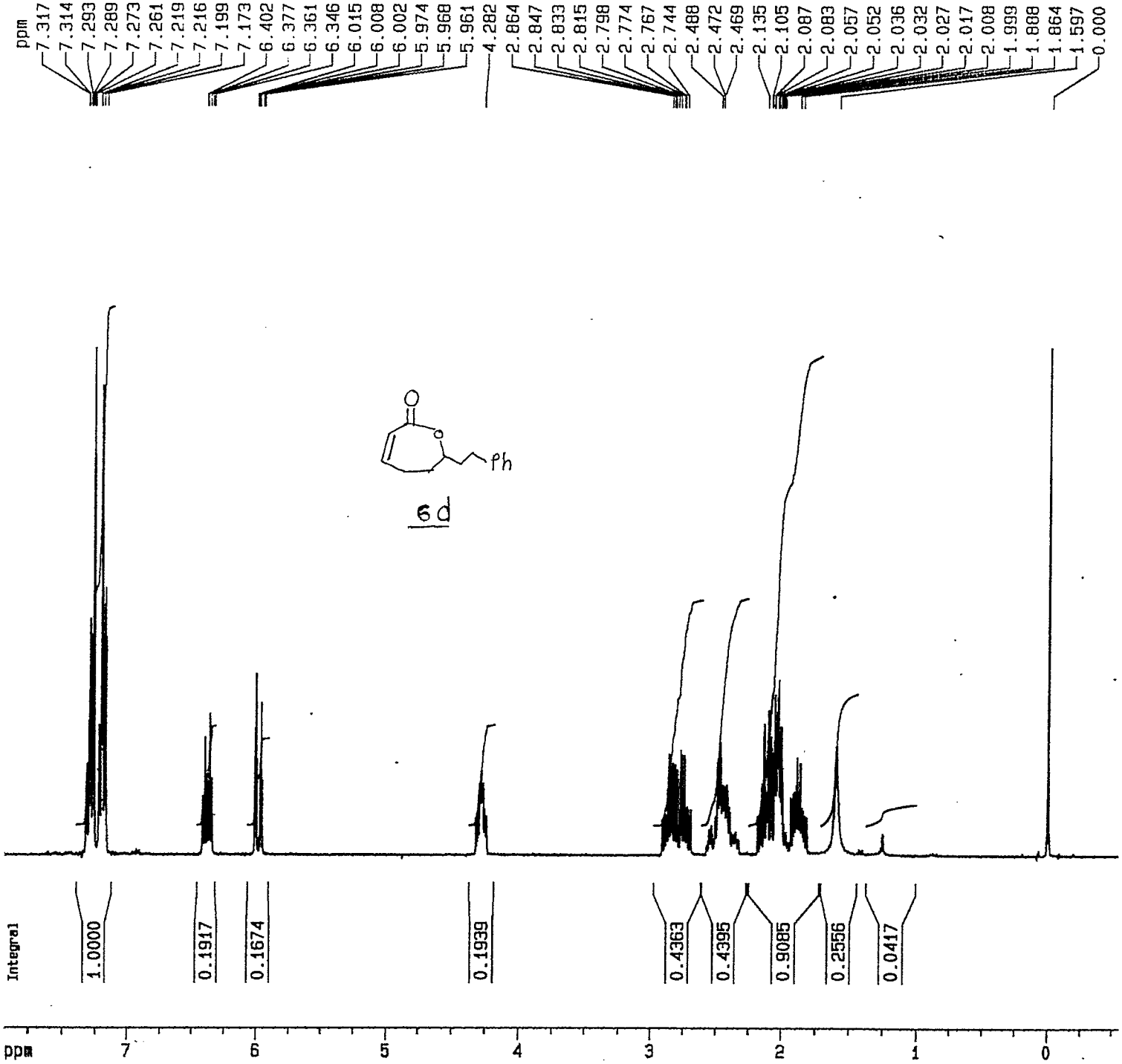


6c

^{1}H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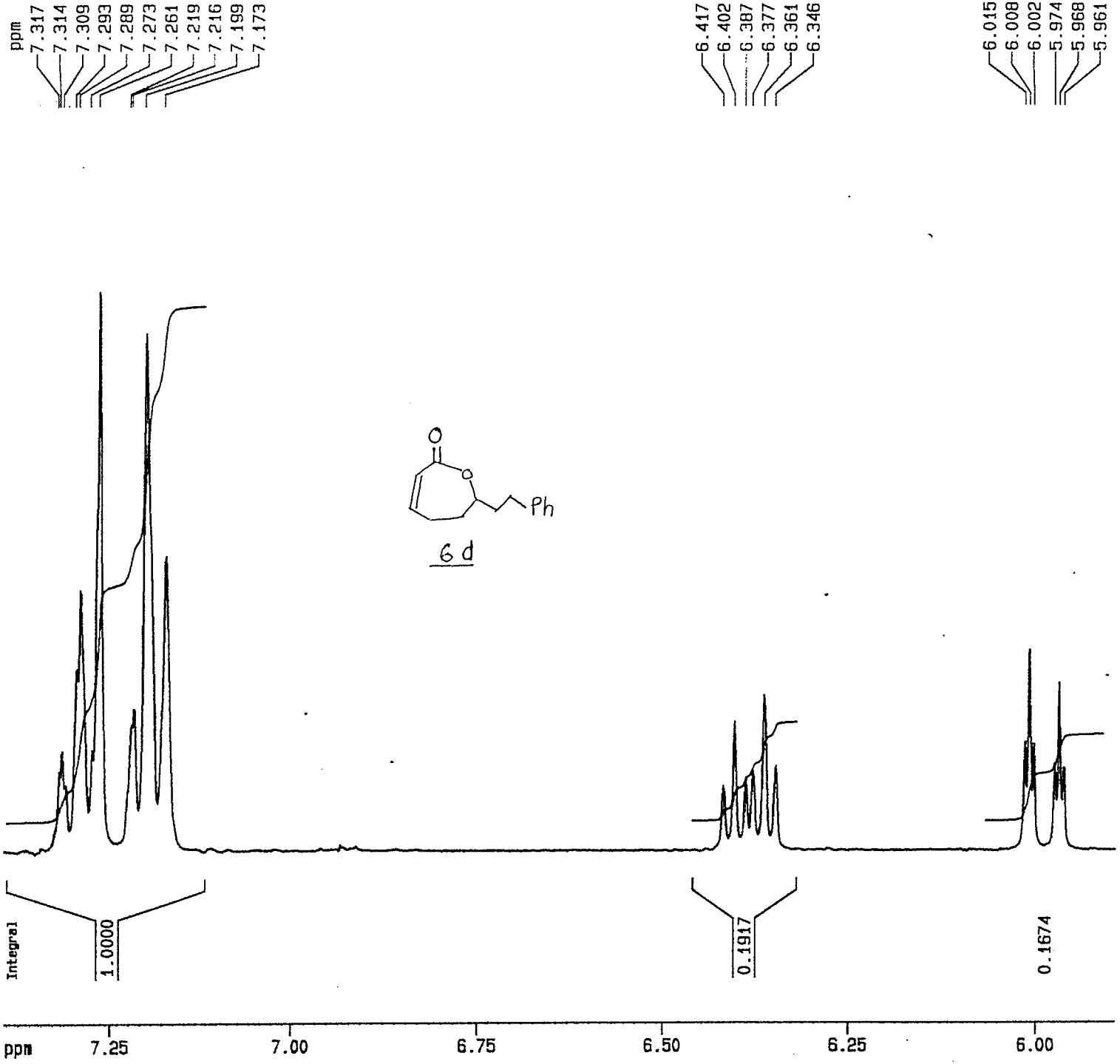
^1H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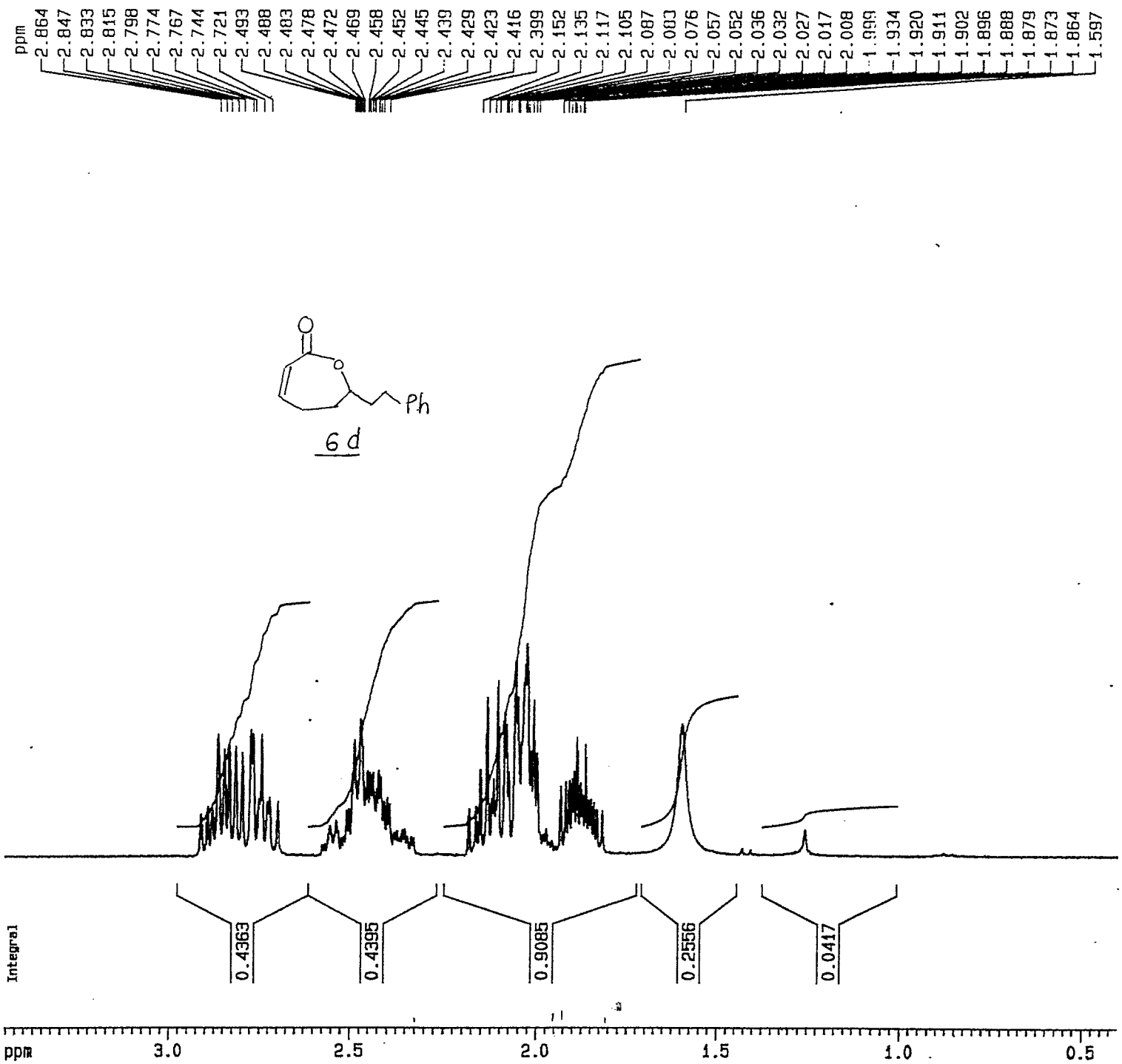
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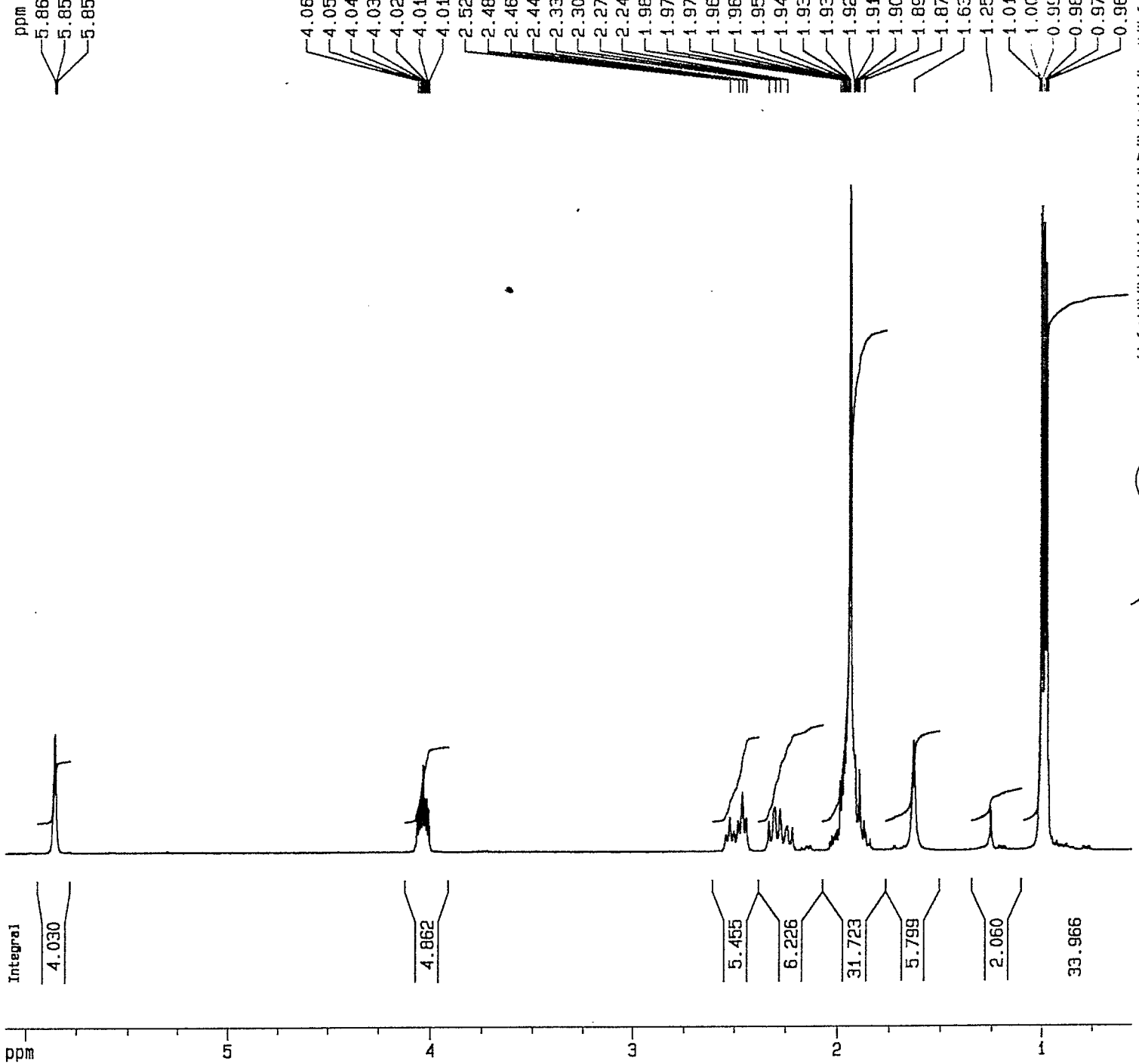

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EXPNO 1
PROCNO 1

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Date 950725
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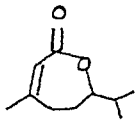
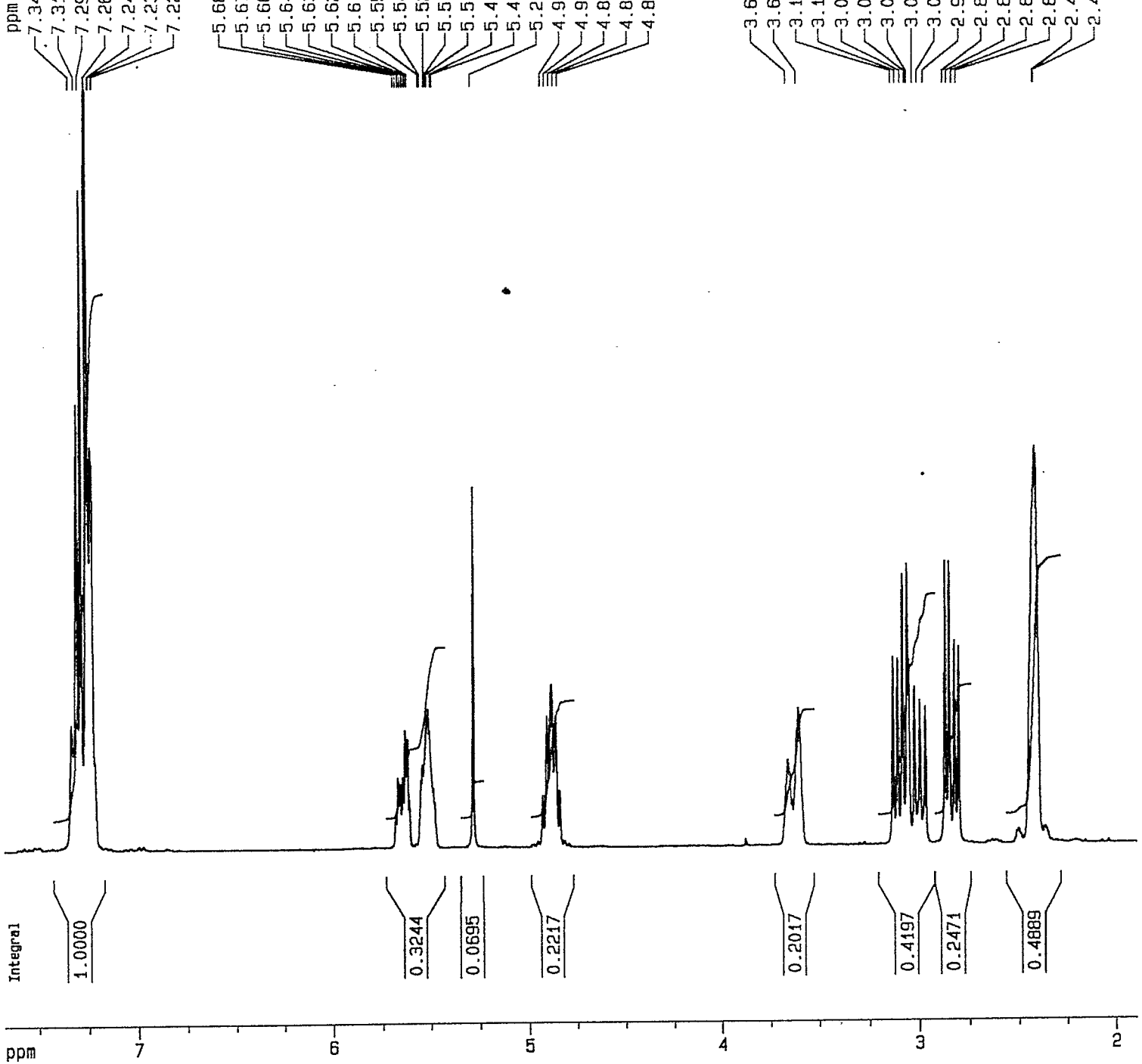
AQ 3.4015260 s
FIDRES 0.147002 H
SW 103.9 u
RG 181
NUCLEUS 1H
D1 3.0000000 s
P1 5.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4815.96 H
TD 32768
NS 16
DS 0

^{1}H



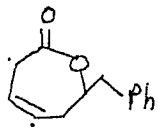
6e

J-12

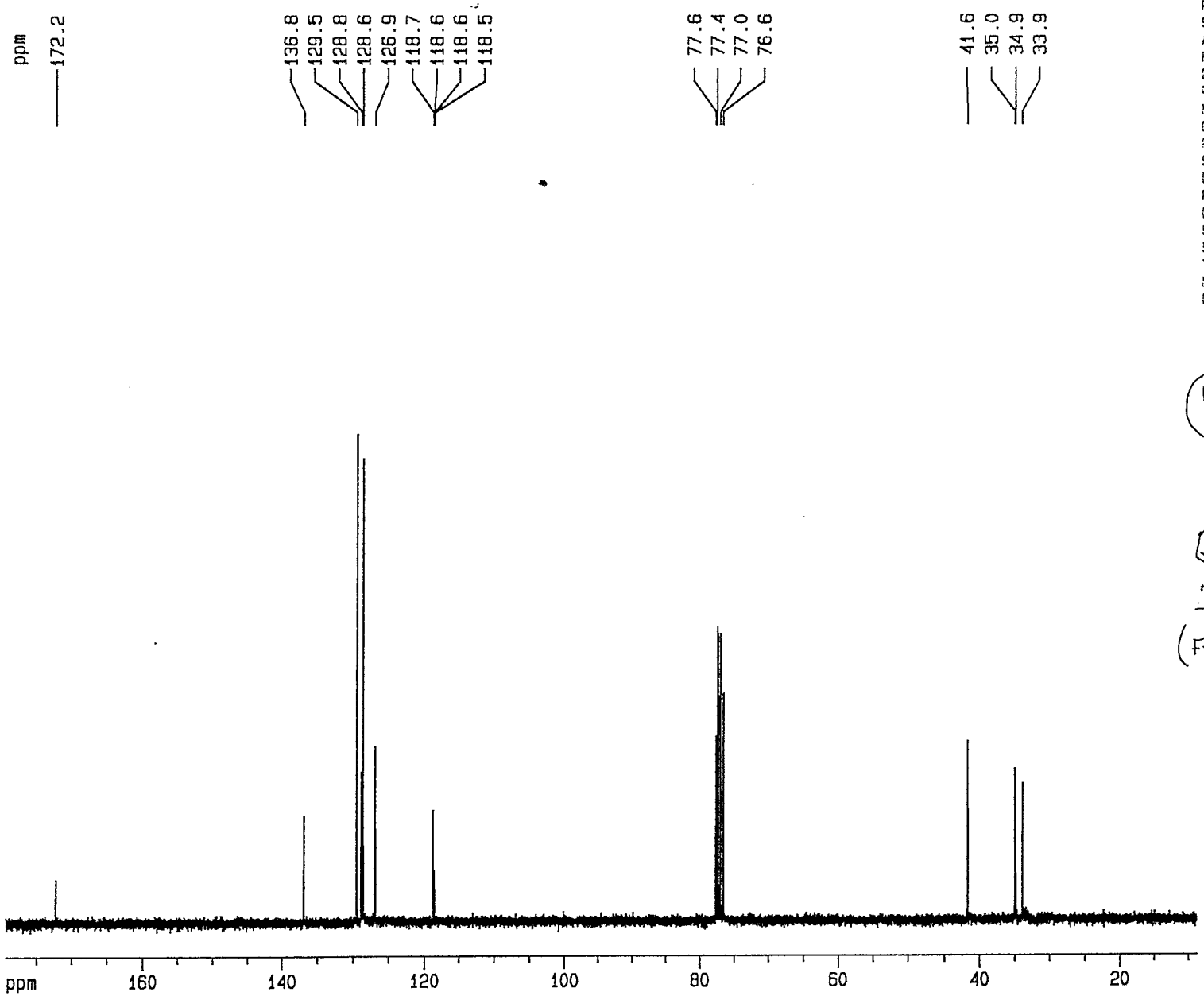


FROM EQUILIBRTTION EXPT.

^1H



7bCD

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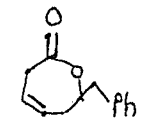
EXPNO             3
PROCNO            1

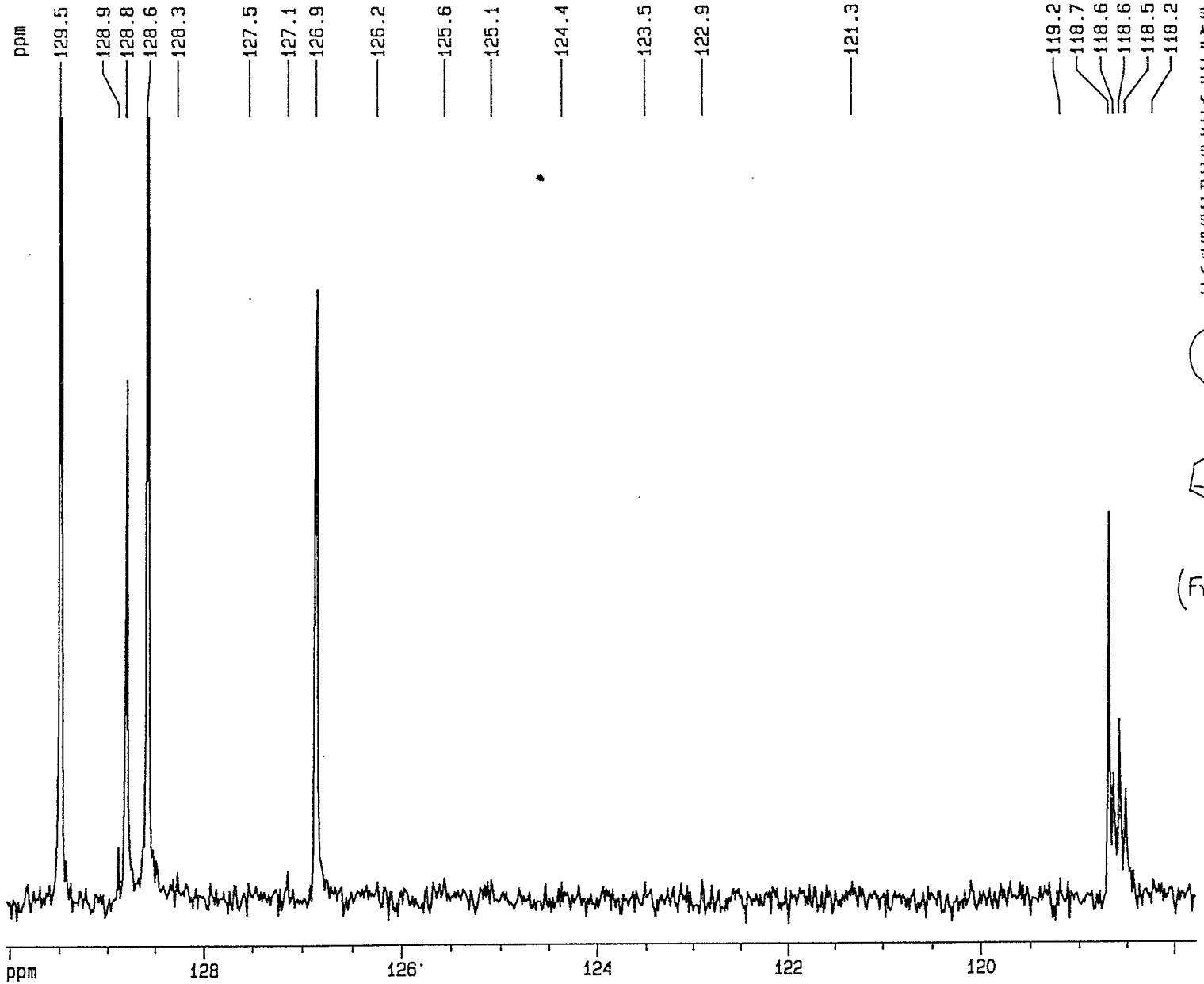
F2 - Acquisition Paramete
Date         S60708
Time          15.45
PULPROG        zgdc
SOLVENT       CDCl3
AQ        0.8356350 s
FIDRES    0.598384 H
DW           25.5 u
RG         8195.2
NUCLEUS        off
D11       0.0300000 s
P31         108.0 u
SFO2    300.1310073 M
D1        1.0000000 s
P1            5.0 u
DE            4.5 u
SFO1    75.4750000 M
SWH     19607.84 H
TD          32768
NS           5731
DS              0
```

^{13}C



7b with D

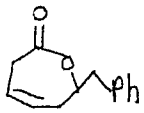
(From equilibrium expt)




```
NAME          06.HM
EXPNO             3
PROCNO            1

F2 - Acquisition Parameter
Date         960708
Time          15.45
PULPROG        zgdc
SOLVENT        CDCl3
AQ        0.8388350 s
FIDRES    0.598384 H
DW           25.5 u
RG         9195.2
NUCLEUS       off
D11       0.0300000 s
P31         108.0 u
SFO2    300.1310073 M
D1        1.0000000 s
P1            5.0 u
DE            4.5 u
SFO1     75.4760000 M
SWH      19607.84 H
TD          32768
NS           5731
DS              0
```

^{13}C

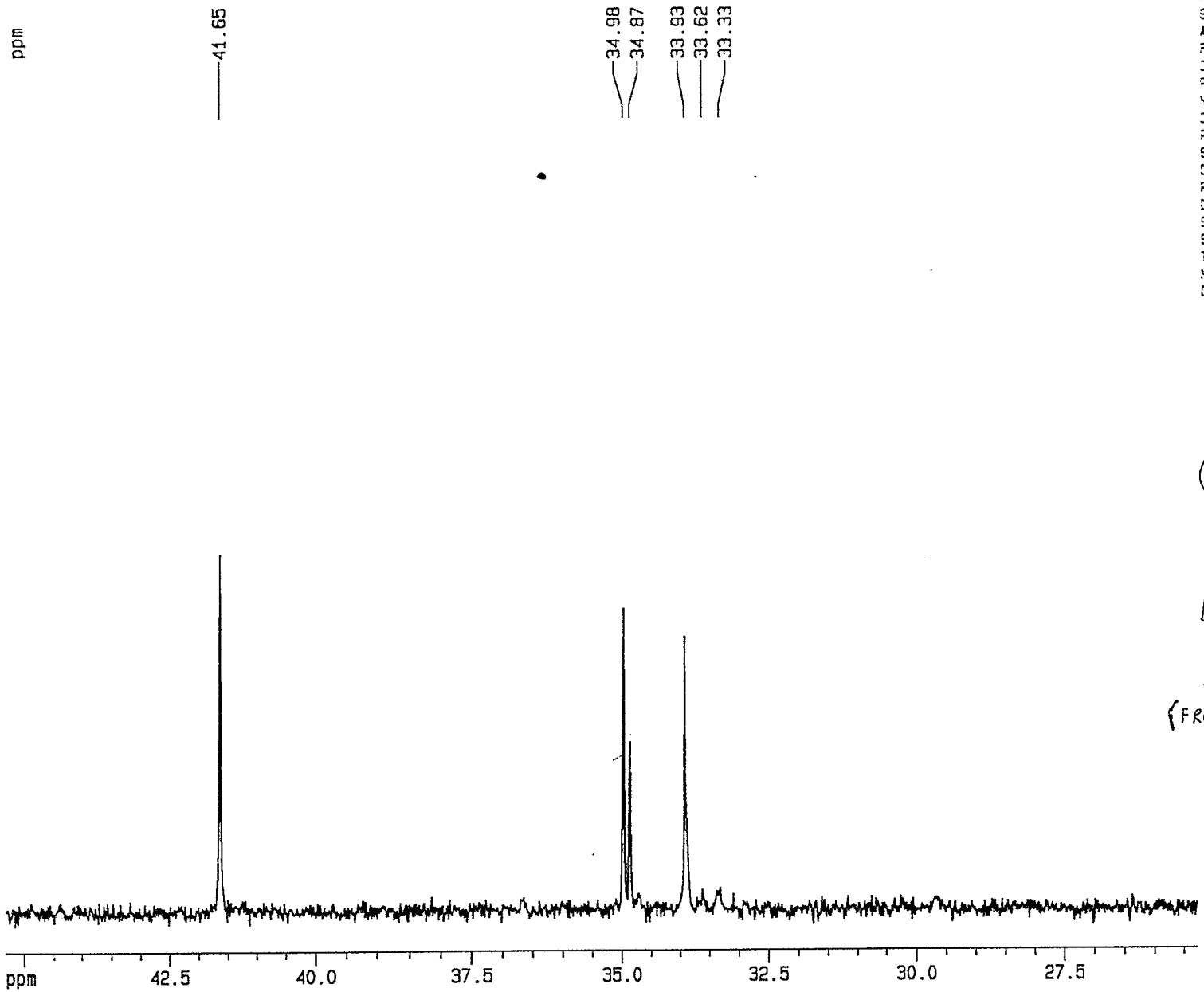


7b with D
(From equilibrium expt)

NAME	J6.HM
EXPNO	3
PROCNO	1

F2 - Acquisition Paramete

Date	960708
Time	15.45
PULPROG	zgdc
SOLVENT	CDCl3
AQ	0.8356350 s
FIDRES	0.598384 H
DW	25.5 u
RG	9195.2
NUCLEUS	off
D11	0.0300000 s
P31	108.0 u
SFO2	300.1310073 M
D1	1.0000000 s
P1	5.0 u
DE	4.5 u
SFO1	75.4760000 M
SWH	19607.84 H
TD	32768
NS	5731
DS	0



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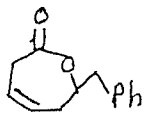
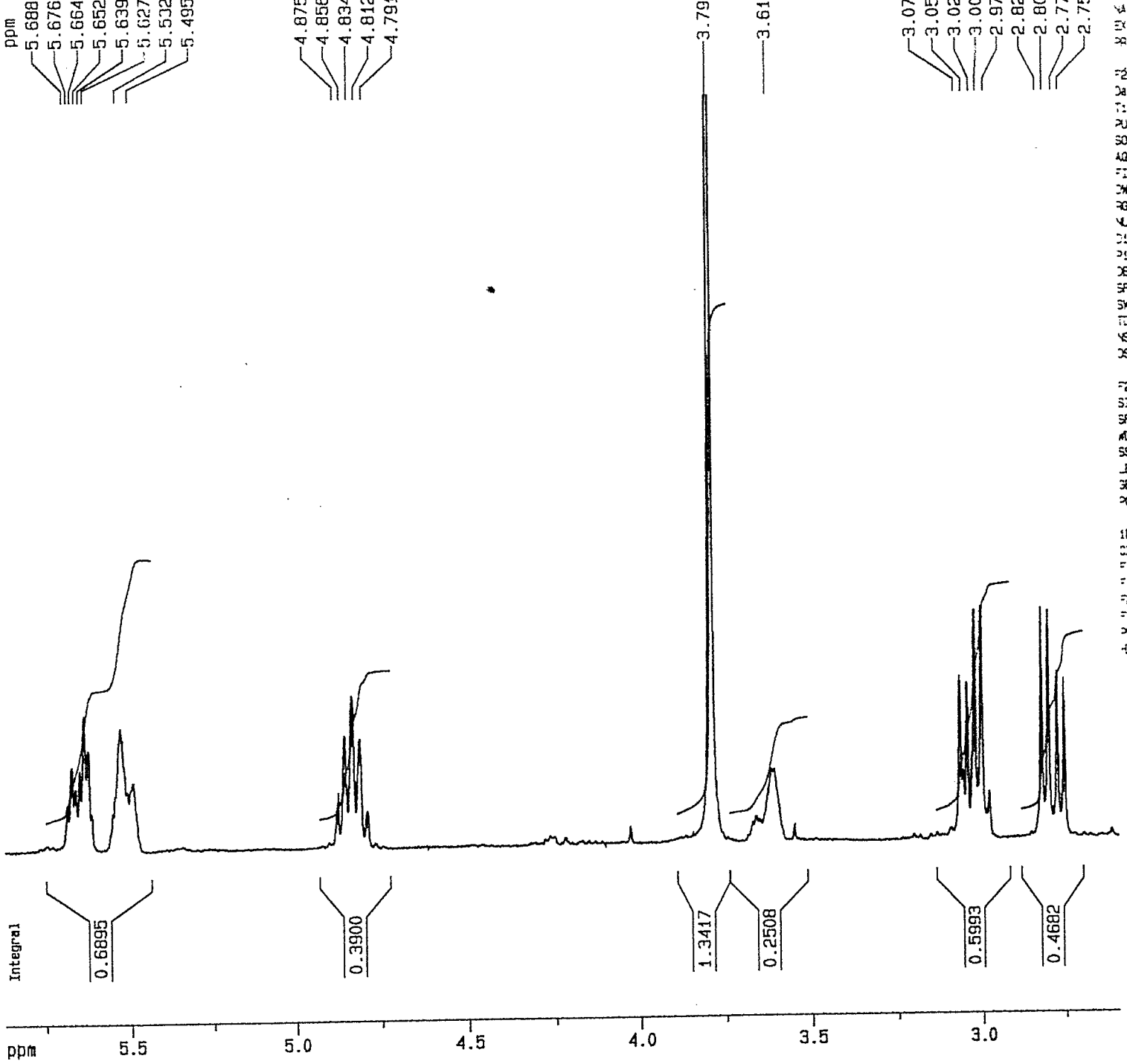


7b with D

(FROM EQUILIBRATION EXPT)

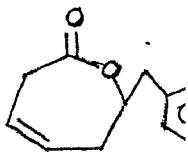



```
NAME          v11.nm
EXPNO              1
PROCNO             1

F2 - Acquisition Paramete
Date          960607
Time           14.38
PULPROG           zg
SOLVENT        CDCl3
AQ        3.4015260 s
FIDRES    0.147002 H
DW            103.8 u
RG              181
NUCLEUS          1H
D1        3.0000000 s
P1              6.9 u
DE              4.5 u
SFO1    300.1318383 M
SWH       4816.96 H
TD             32768
NS                16
DS                 0

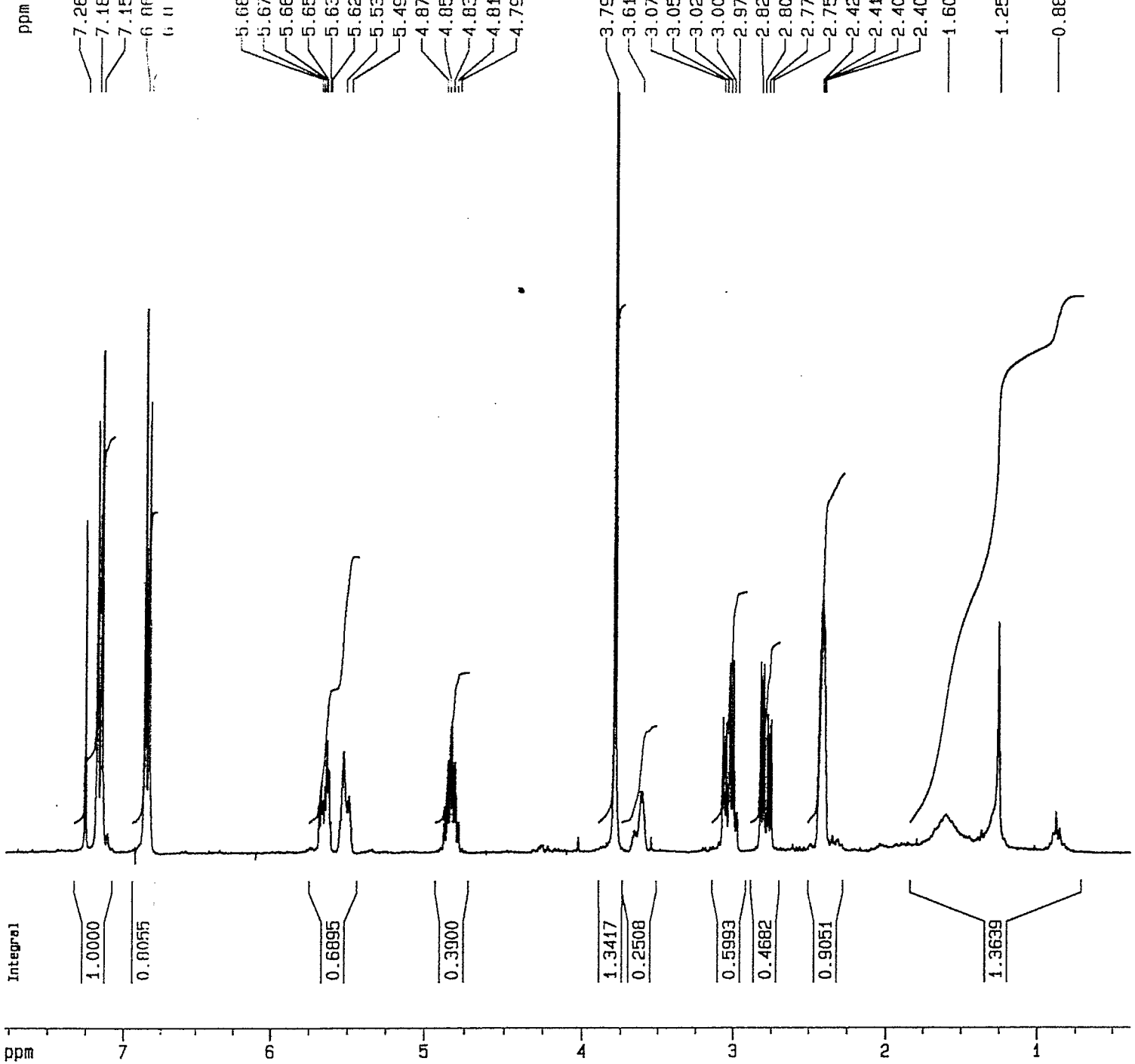
F2 - Processing parameter
SI             16384
SF     300.1300067 M
WDW               EM
SSB                0
LB              0.30 H
GB                 0
PC              1.00

1D NMR plot parameters
CX             20.00 c
F1P             5.869 p
F1           1761.39 H
F2P             2.592 p
F2           777.93 H
PPMCM        0.16384 p
HZCM        49.17309 H
```



7c

^1H

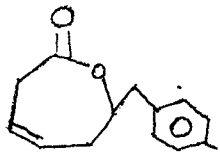



```
NAME          Vij.nm
EXPNO              1
PROCNO             1

F2 - Acquisition Parameter
Date          960607
Time           14.38
PULPROG           zg
SOLVENT        CDCl3
AQ        3.4015260 s
FIDRES     0.147002 Hz
DW            103.8 us
RG              181
NUCLEUS          1H
D1        3.0000000 s
P1              6.9 us
DE              4.5 us
SFO1    300.1318383 MHz
SWH        4816.96 Hz
TD            32768
NS               16
DS                0

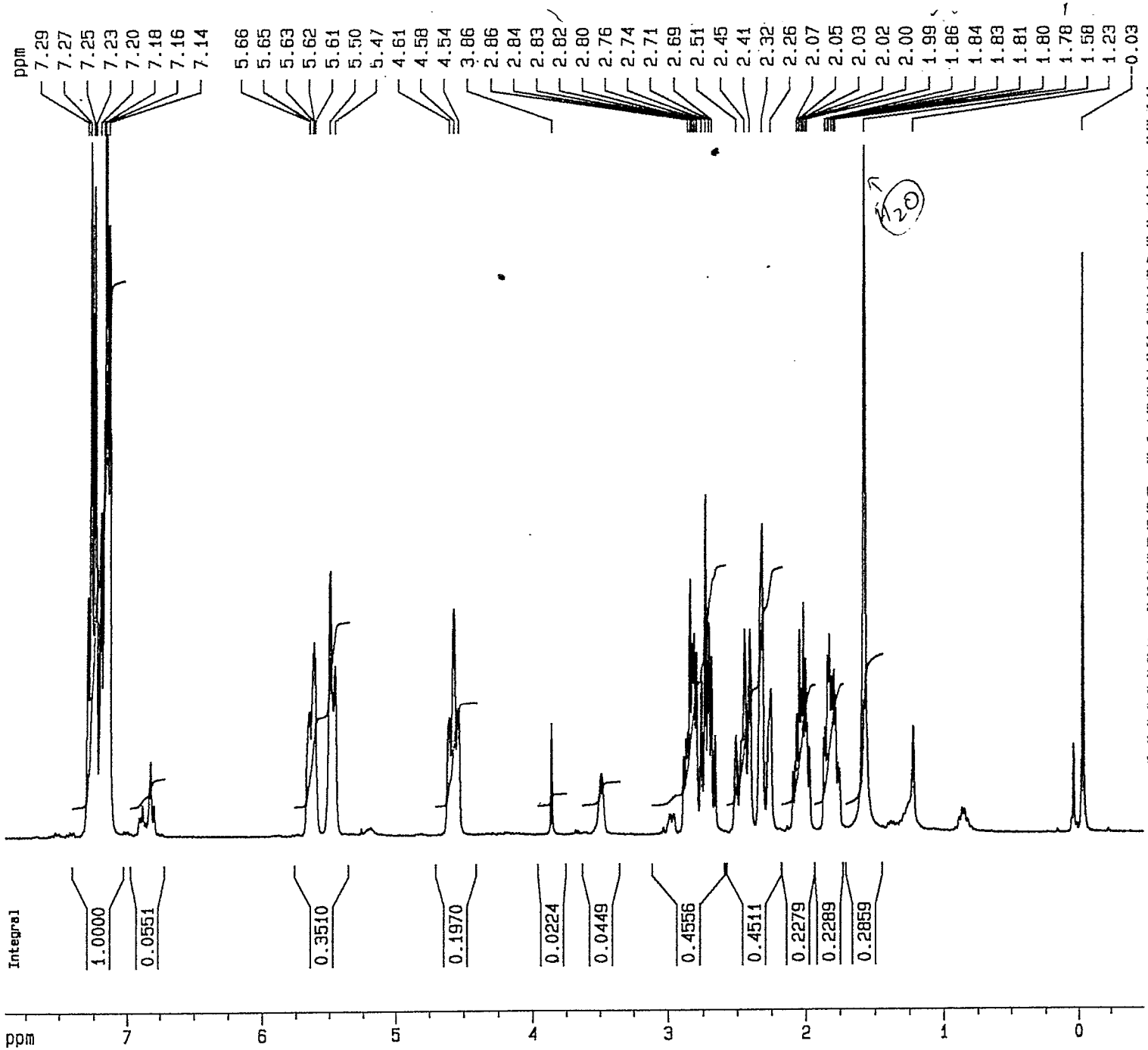
F2 - Processing parameter
SI            16384
SF     300.1300067 MHz
WDW              EM
SSB               0
LB             0.30 Hz
GB                0
PC             1.00

1D NMR plot parameters
CX            20.00 cm
F1P            7.775 ppm
F1          2333.41 Hz
F2P            0.385 ppm
F2           115.60 Hz
PPMCM        0.36947 ppm/cm
HZCM       110.89034 Hz
```



7C

^{1}H



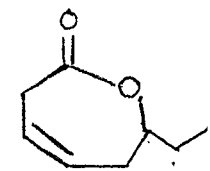
Current Data Parameters
NAME gaj2.rr
EXPNO 1
PROCNO 1

F2 - Acquisition Paramete
Date 960322
Time 11.57
PULPROG zg
SOLVENT CDCl3
AQ 3.4015260 s
FIDRES 0.147002 H
DW 103.8 u
RG 256
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4816.96 H
TD 32768
NS 24
DS 0

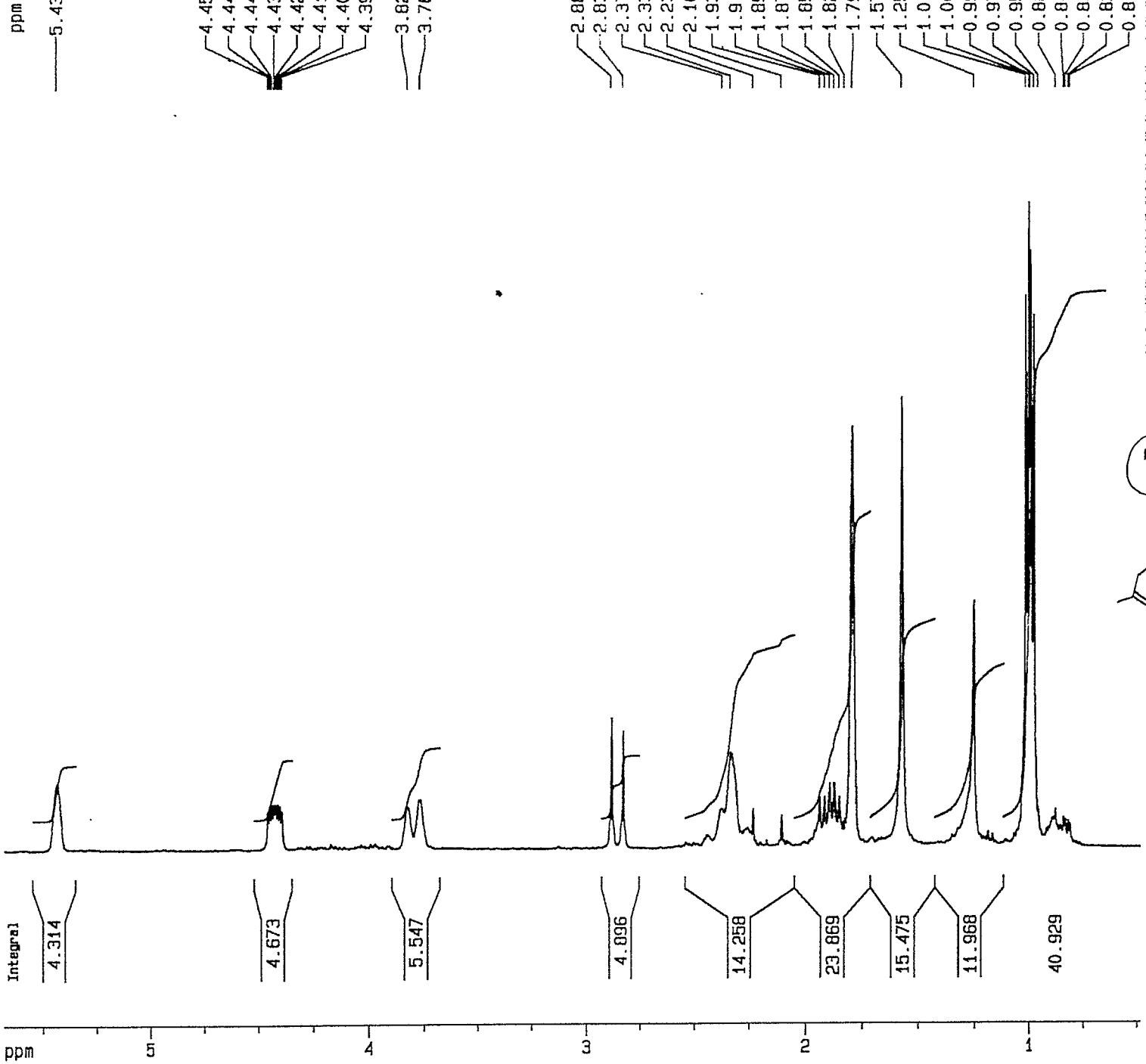
F2 - Processing parameter
SI 8192
SF 300.1300156 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 c
F1P 7.897 p
F1 2370.20 H
F2P -0.492 p
F2 -147.75 H
PPMCM 0.41948 p
HZCM 125.89771 H

^{1}H



7d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4.39
3.82
3.76
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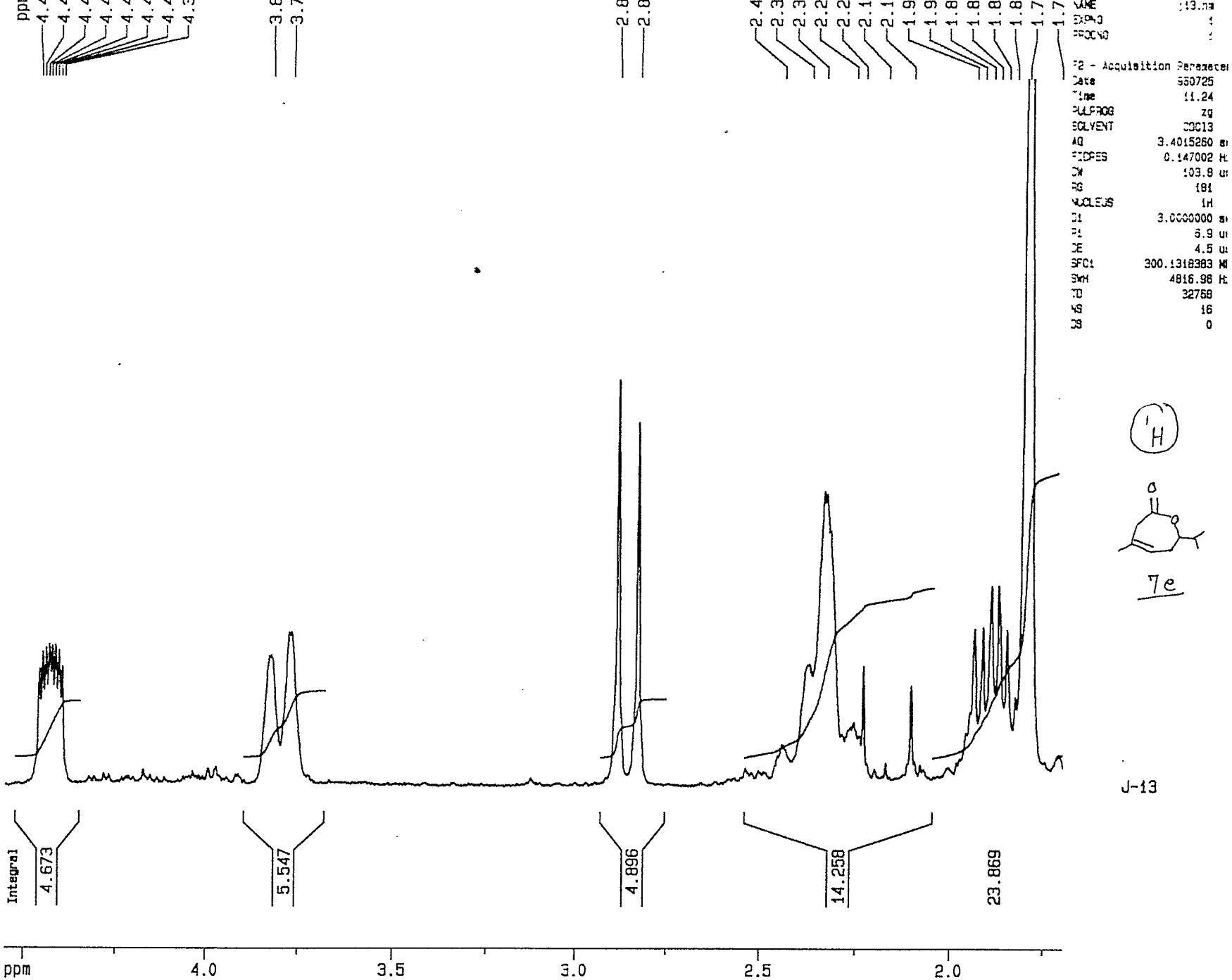
2.22
2.19
1.93
1.91
1.88
1.87
1.86
1.85
1.79
1.57
1.22
1.07
1.06
0.99
0.99
0.99
0.88
0.88
0.88
0.88
0.88
NAME 113.hm
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date 960725
Time 11.24
PULPROG zg
SOLVENT CDCl3
AQ 3.4015260 s
FIDRES 0.147002 H
DW 103.8 u
RG 181
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4816.96 H
TD 32768
NS 16
DS 0
1H
7e
J-13
Integral
4.314
4.673
5.547
4.096
14.258
23.869
15.475
11.968
40.929
ppm
5
4
3
2
1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4.4
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4.4
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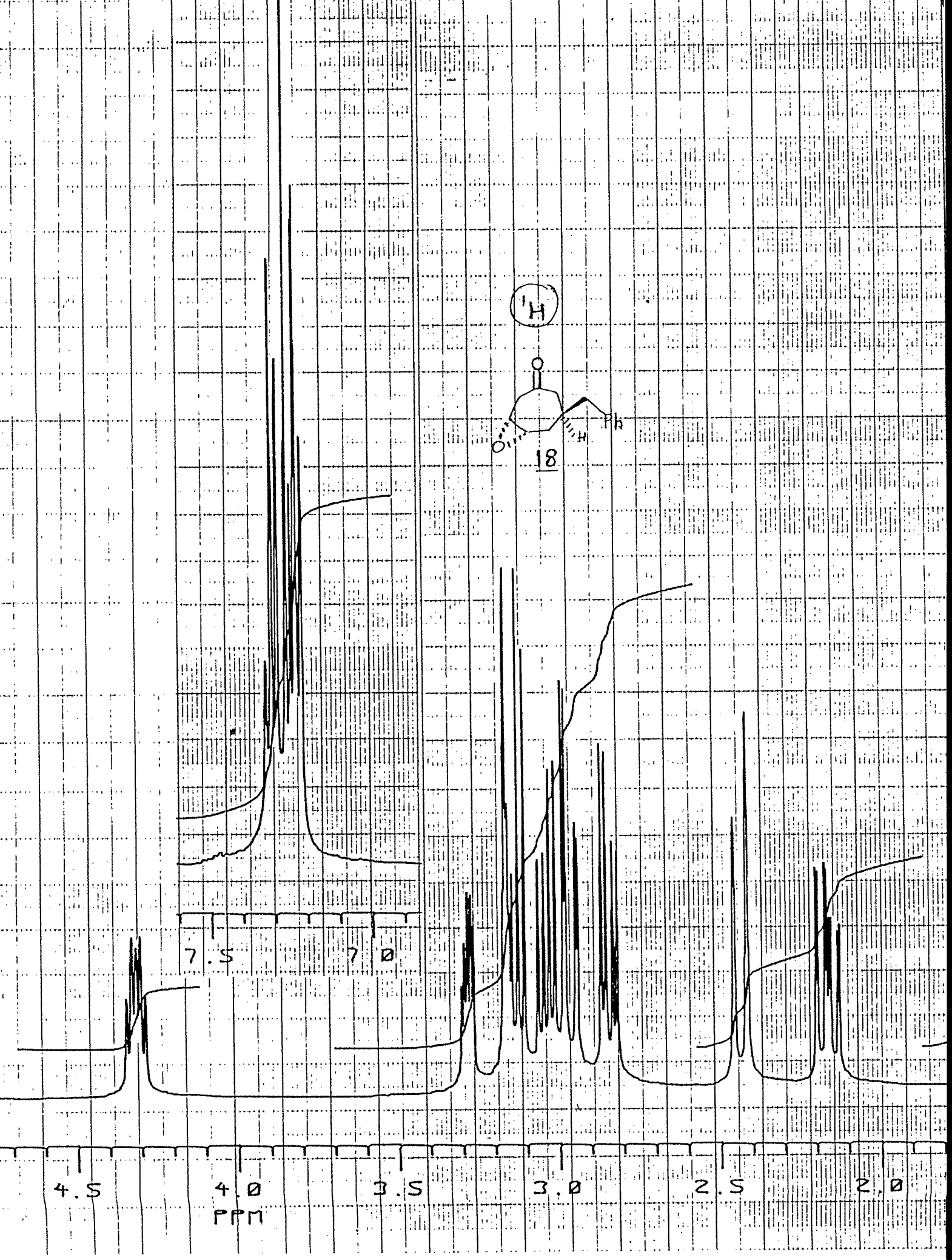
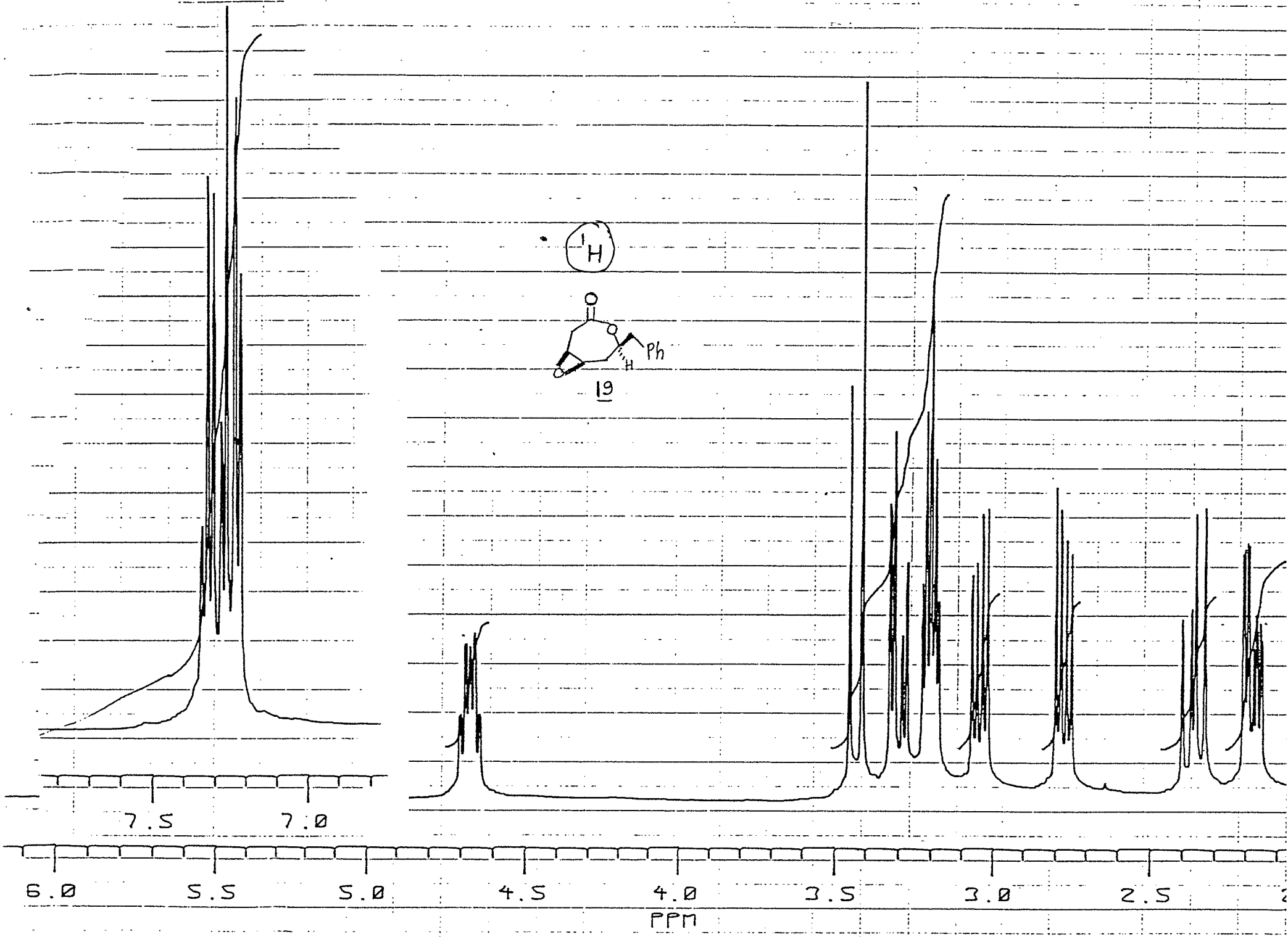
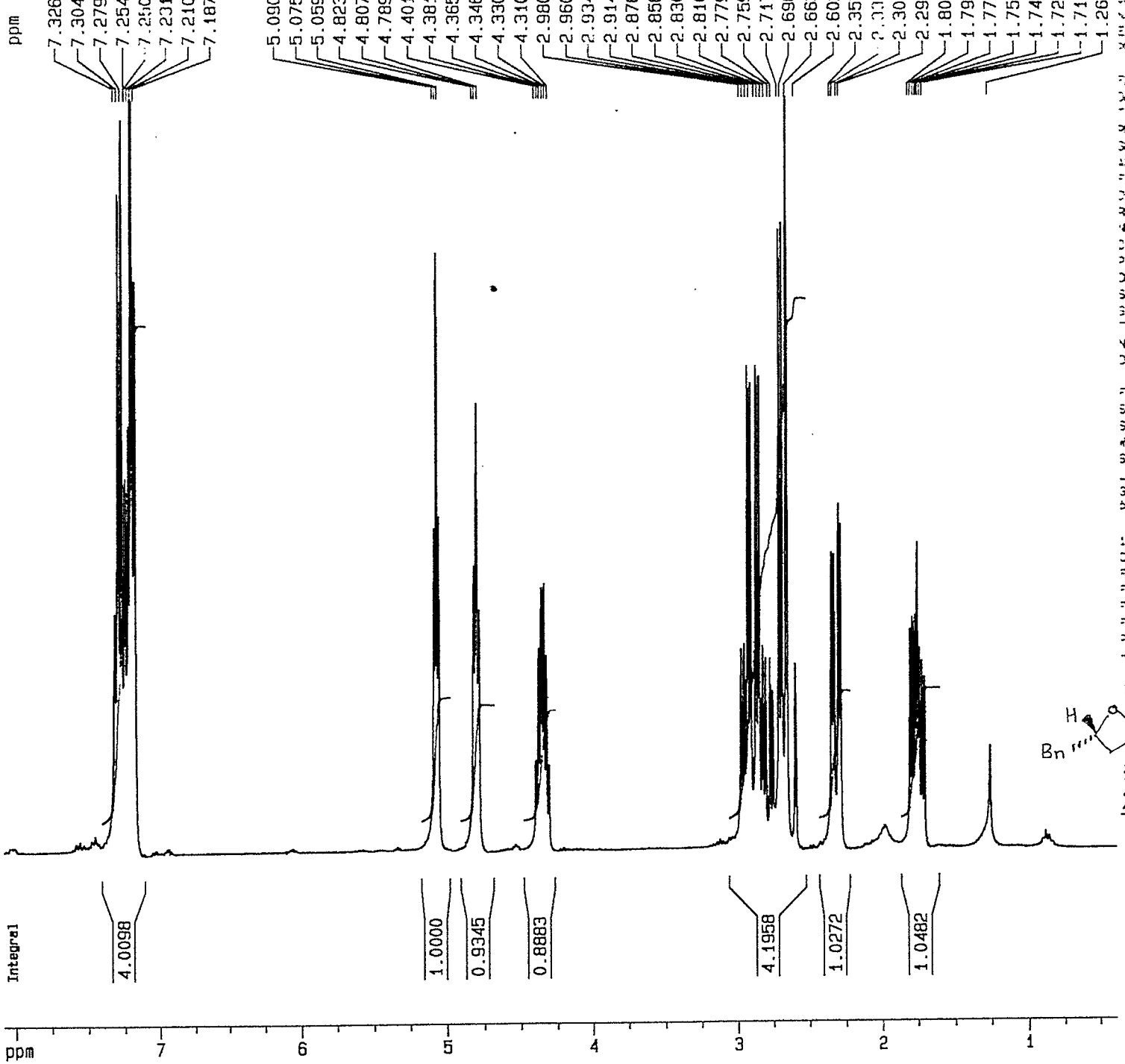
4.3
3.8
3.7
2.8
2.8
2.4
2.3
2.3
2.2
2.2
2.1
2.1
1.9
1.9
1.8
1.8
1.8
1.8
1.7
1.7
NAME 113.ma
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date 950725
Time 11.24
PULPROG zg
SOLVENT CDCl3
AQ 3.4015260 s
FIDRES 0.147002 H
DW 103.8 u
RG 181
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4816.96 H
TD 32768
NS 16
DS 0
1H
7e
J-13
Integral
4.673
5.547
4.896
14.258
23.869
ppm
4.0
3.5
3.0
2.5
2.0


^{1}H
O
Ph
H
O
18
7.5
7.0
4.5
4.0
3.5
3.0
2.5
2.0
PPM

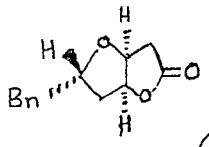

^{1}H
Ph
H
19
7.5
7.0
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
PPM



F2 - Acquisition Paramete
Date 980603
Time 19.04
PULPROG zg
SOLVENT CDCl3
NS 3.4015260 s
FIDRES 0.147002 -
DW 103.8 u
RG 64
NUCLEUS 1H
D1 3.0000000 s
P1 6.9 u
DE 4.5 u
SFO1 300.1318383 M
SWH 4816.96 -
TD 32768
NS 16
DS 0

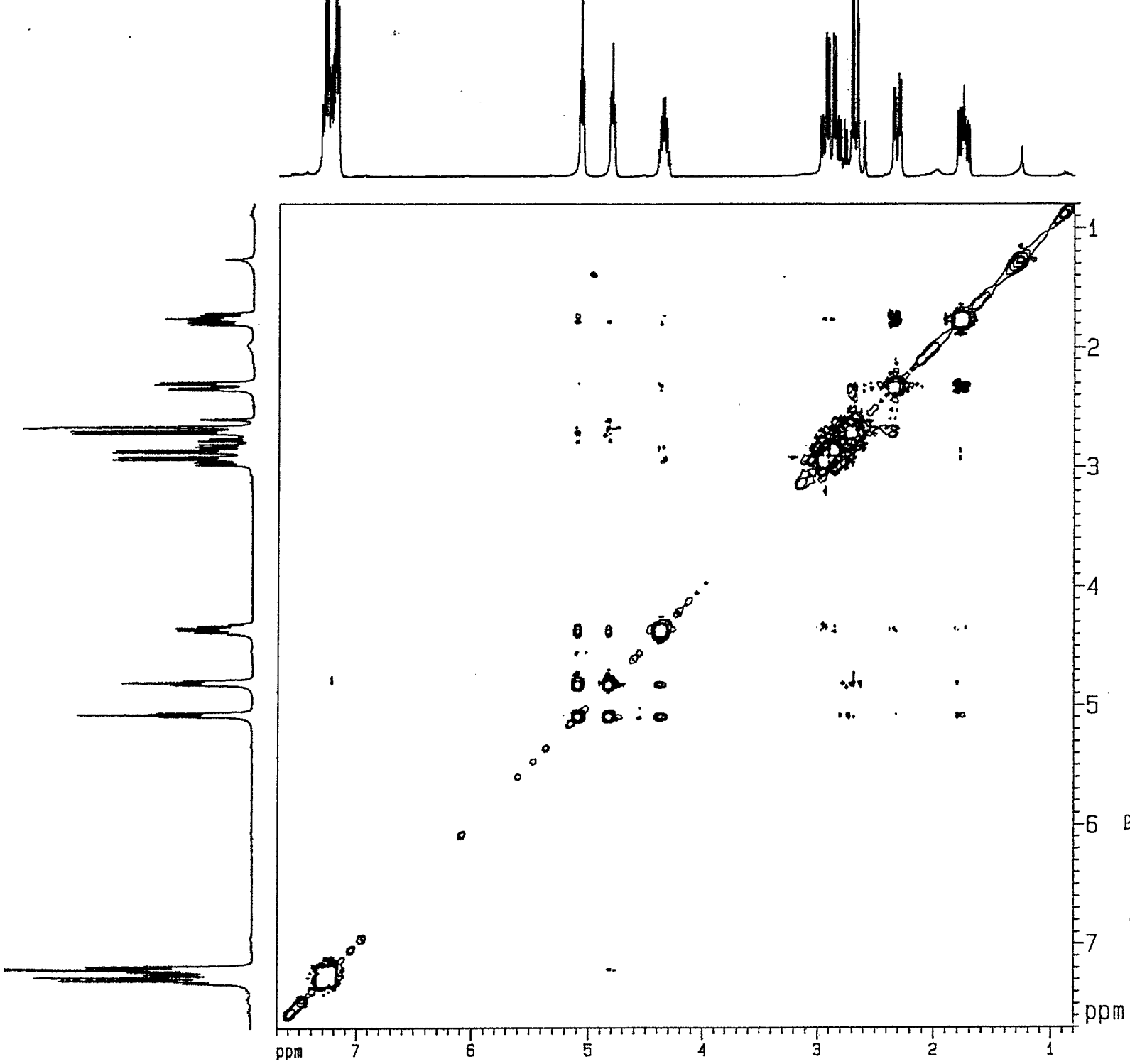
F2 - Processing parameter
SI 16384
SF 300.1300055 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 c
F1P 8.080 p
F1 2424.90 -
F2P 0.389 p
F2 116.78 -
PPMCM 0.38452 p
HZCM 115.40623 -



21

^{1}H



Current Data Parameter

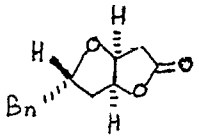
NAME	vkyji.rr
EXPNO	2
PROCNO	1

F2 - Acquisition Paramei

Date	960624
Time	23.31
PULPROG	roesytp
SOLVENT	CDCl3
AQ	0.1625184 sec
FIDRES	3.082584 Hz
DW	158.4 usec
RG	181
NUCLEUS	1H
D1	2.0000000 sec
D12	0.0000200 sec
P1	6.9 usec
D0	0.0000030 sec
P15	300000.0 usec
DE	4.5 usec
SFO1	300.1314352 MHz
SWH	3156.57 Hz
TD	1024
NS	0
DS	8162
DW0	0.0001584 sec

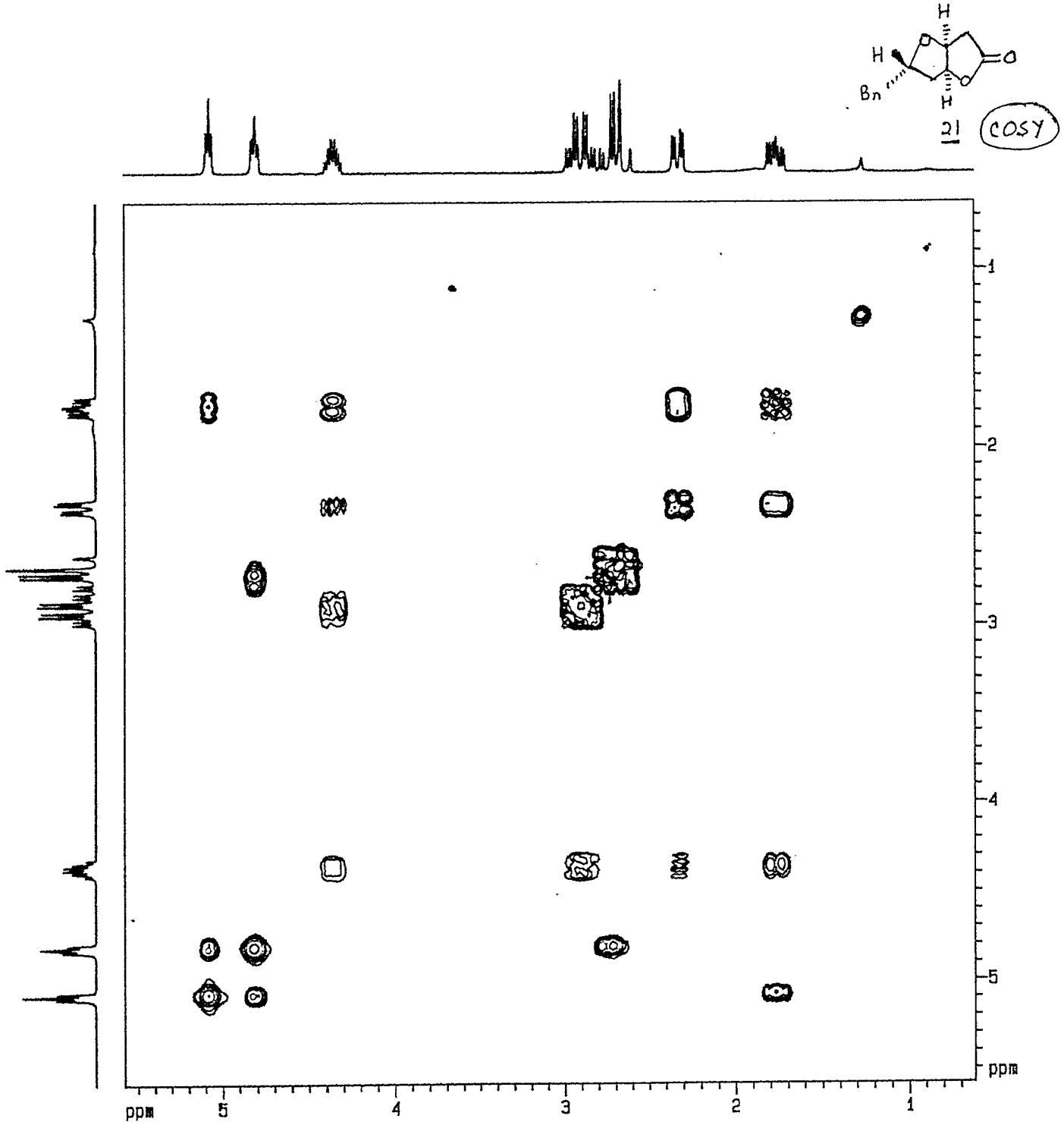
F1 - Acquisition paramei

NO0	2
TD	256
SFO1	300.1314 MHz
FIDRES	12.330336 Hz
SW	10.517 ppm



21

ROESY



Current Data Parameters

NAME	j1.hm
EXPNO	2
PROCNO	1

F2 - Acquisition Parameter

Date	950614
Time	12.56
PULPROG	cosy90
SOLVENT	CDCl3
AQ	0.1727040 sec
FIDRES	2.906436 Hz
DW	336.0 usec
RG	64
NUCLEUS	1H
D1	1.5000000 sec
P1	6.9 usec
D0	0.0000030 sec
DE	4.5 usec
SFO1	300.1309351 MHz
SWH	1458.10 Hz
TD	512
NS	0
DS	16
IN0	0.0006720 sec

F1 - Acquisition parameter

ND0	1
TD	128
SFO1	300.1309 MHz
FIDRES	11.625742 Hz
SW	4.958 ppm

F2 - Processing parameters

SI	256
SF	300.1300046 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

F1 - Processing parameters

SI	256
MC2	QF
SF	300.1300046 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

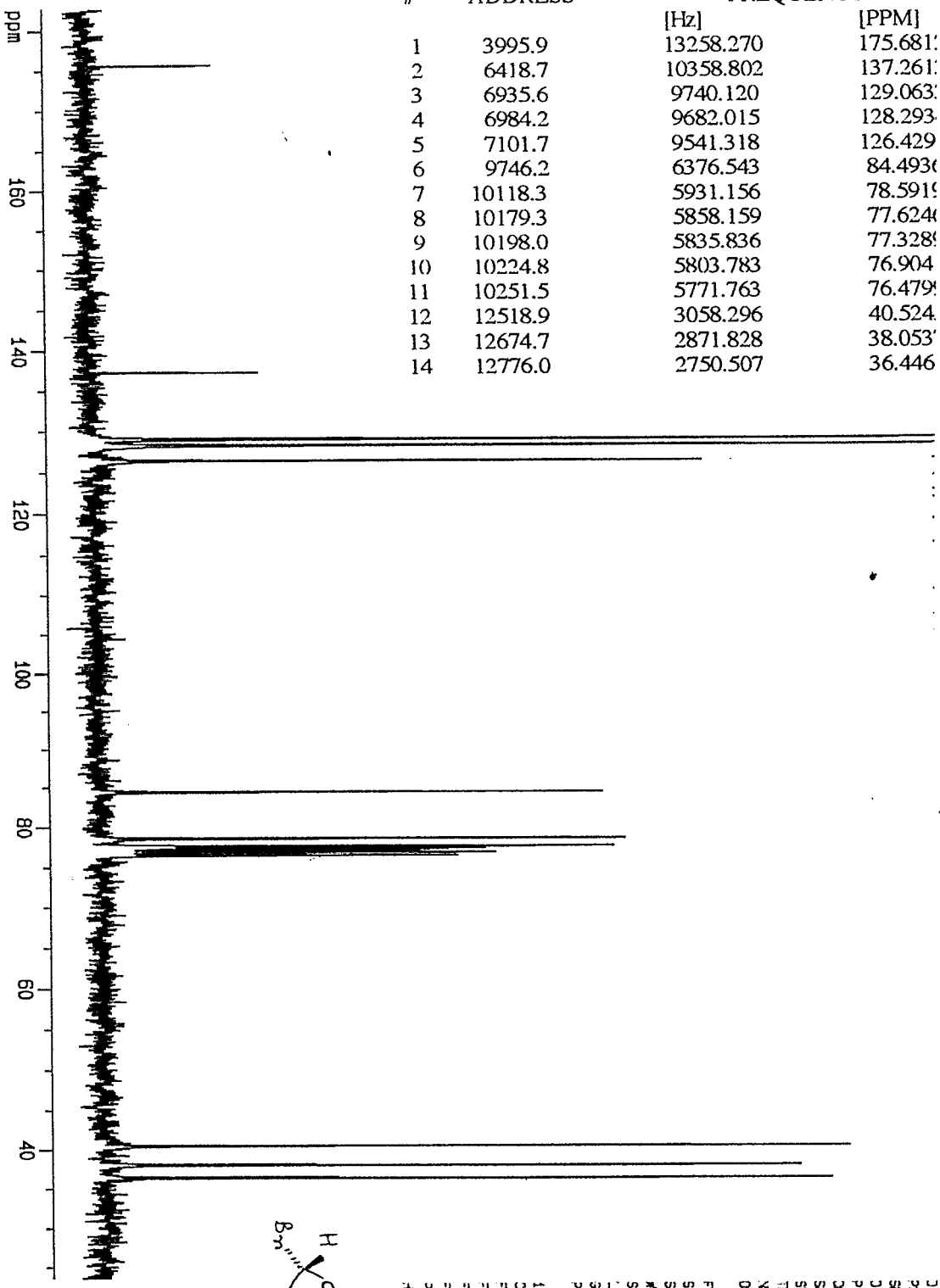
2D NMR plot parameters

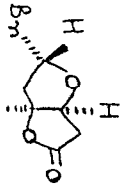
CX2	15.00 cm
CX1	15.00 cm
F2PLO	5.579 ppm
F2LO	1674.57 Hz
F2PHI	0.821 ppm
F2HI	186.47 Hz
F1PLO	5.579 ppm
F1LO	1674.55 Hz
F1PHI	0.821 ppm
F1HI	186.45 Hz
F2PPMCM	0.33054 ppm/cm
F2HZCM	99.20635 Hz/cm
F1PPMCM	0.33054 ppm/cm
F1HZCM	99.20632 Hz/cm

/u/data/ep/nmr/vkyj1.rr/3/pdata/1/screen Thu Jun

DU=u, USER=ep, NAME=vkyj1.rr, EXPNO=3, PROCNO=1
F1=182.755ppm, F2=23.617ppm, MI=0.60cm, MAXI=10000.0

#	ADDRESS	FREQUENCY [Hz]	[PPM]
1	3995.9	13258.270	175.681
2	6418.7	10358.802	137.261
3	6935.6	9740.120	129.063
4	6984.2	9682.015	128.293
5	7101.7	9541.318	126.429
6	9746.2	6376.543	84.493
7	10118.3	5931.156	78.591
8	10179.3	5858.159	77.624
9	10198.0	5835.836	77.328
10	10224.8	5803.783	76.904
11	10251.5	5771.763	76.479
12	12518.9	3058.296	40.524
13	12674.7	2871.828	38.053
14	12776.0	2750.507	36.446





21

13C

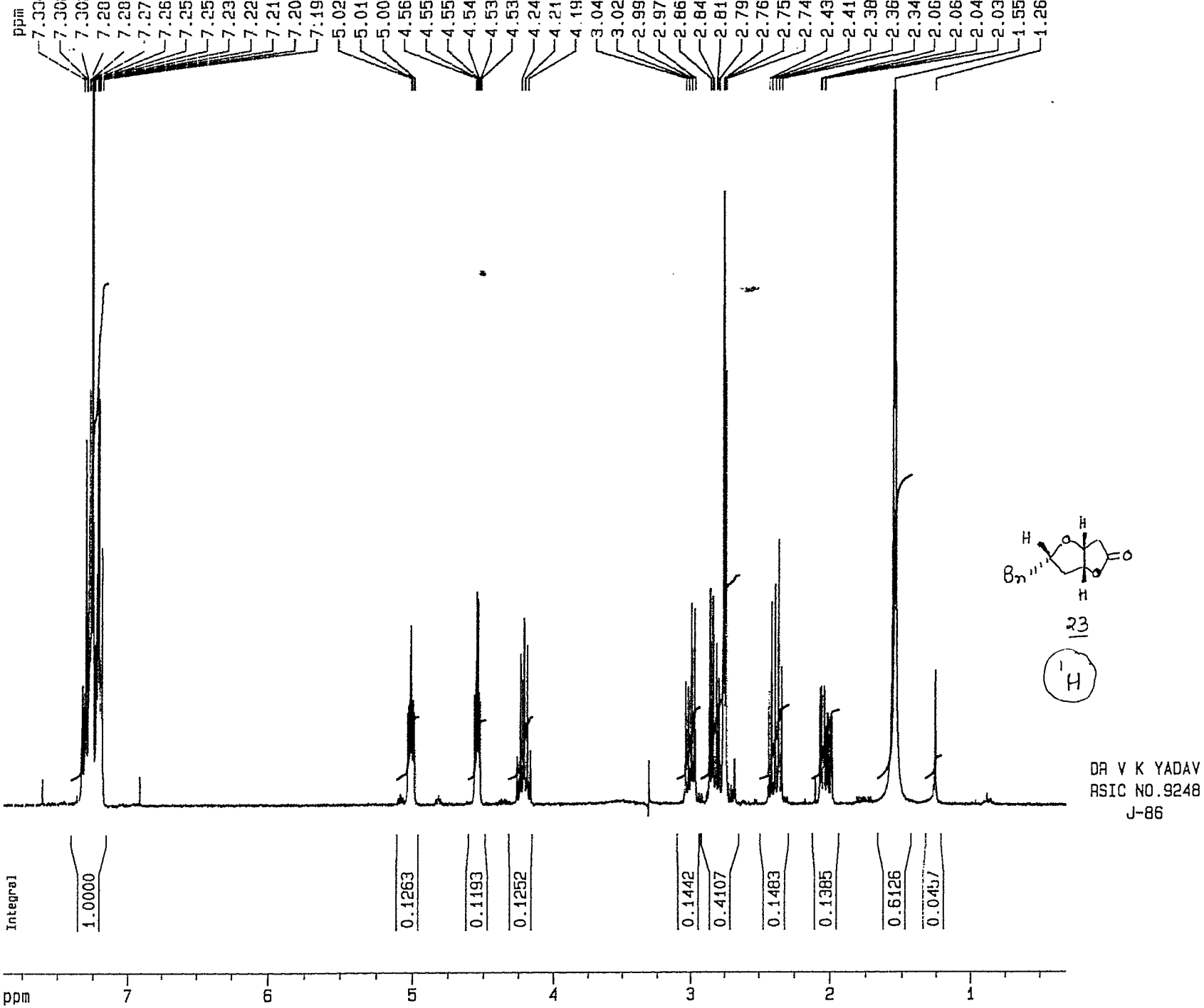
```
NAME                                   vkyj1.rr
EXPNO                                  3
PROCNO                                 1

F2 - Acquisition Parameters
Date_                                  960506
Time                                   15.50
INSTRUM                                spec
PROBHD                                 CDCl3
SOLVENT                                0.8356356 s
NS                                     0.596394 -
DS                                     25.5
SWH                                    14596.5
FIDRES                                 0.4
AQ                                     1.1010073 s
RG                                     300
DW                                     1.0000000 s
DE                                     4.5
TE                                     108.0
D1                                     5.0
D11                                    0.0300000 s
TD                                     32768
SFO1                                   75.4769000 M
SFO2                                   300.1310073 M
NUCLEUS                                off
P1                                     0
PL1                                    0
PL1W                                   EM
SWNT                                   75.4677635 M
SI                                     16384
SF                                     75.4677635 M
SSB                                    0
LB                                     3.00 Hz
GB                                     0
PC                                     1.00

F2 - Processing parameters
SI                                     16384
MC                                     2
SSB                                    0
GB                                     0
PC                                     1.00

1D NMR plot parameters
CX                                     20.00 cm
CY                                     20.00 cm
F1P                                    182.755 ppm
F1                                     13792.10 Hz
F2P                                    23.617 ppm
F2                                     1782.29 Hz
PPMCM                                  7.95681 ppm/cm
HZCM                                   600.49523 Hz/cm
```

13C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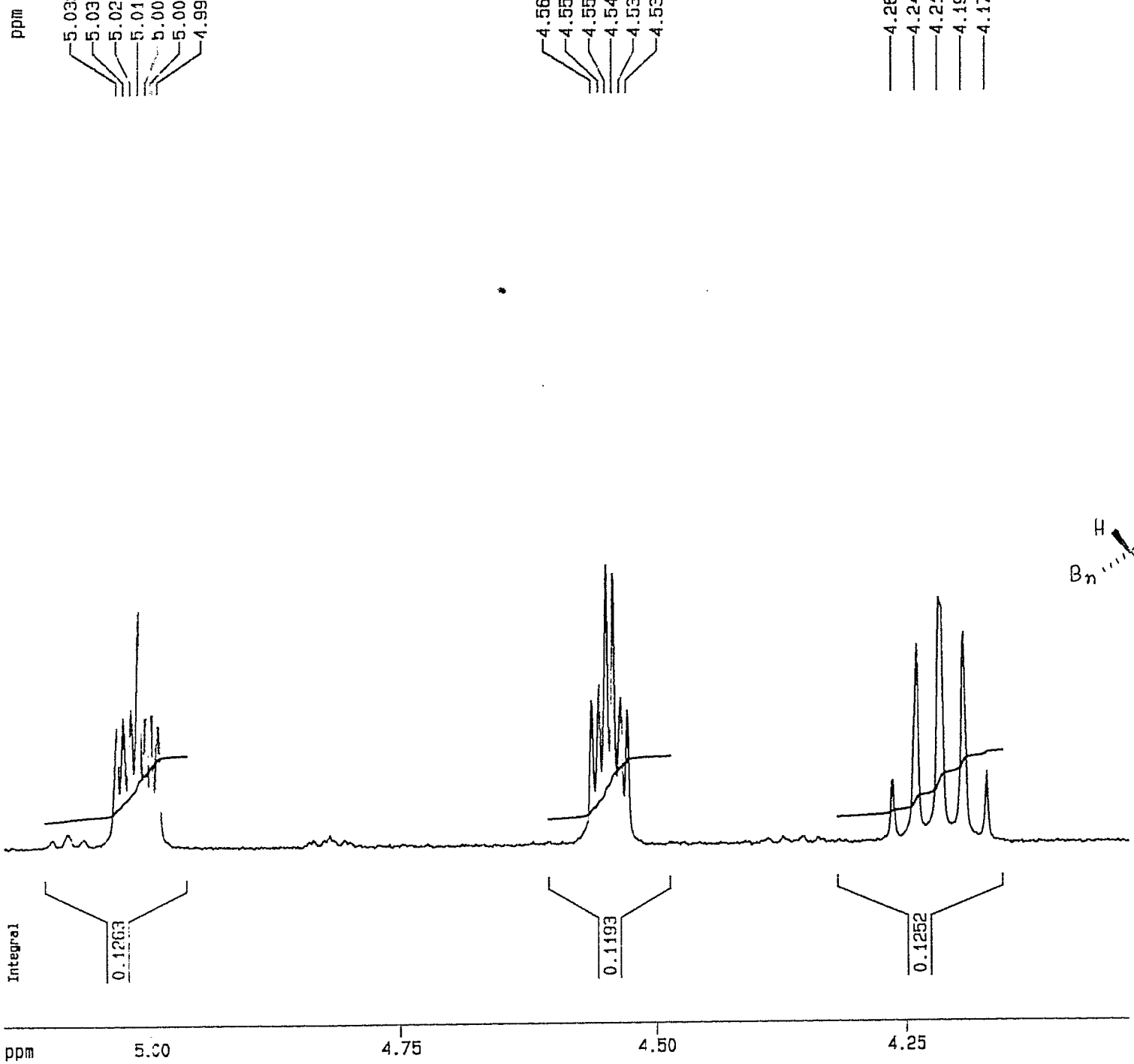
5.02 5.01 5.00 4.56 4.55 4.55 4.54 4.53 4.53 4.24 4.21 4.19
3.04 3.02 2.99 2.97 2.86 2.84 2.81 2.79 2.76 2.75 2.74 2.43 2.41 2.38 2.36 2.34 2.06 2.06 2.04 2.03 1.55 1.26
Integral
1.0000
0.1263
0.1193
0.1252
0.1442
0.4107
0.1483
0.1385
0.6126
0.0457
ppm
7
6
5
4
3
2
1
H
Bn
O
23
^{1}H
DR V K YADAV
RSIC NO.9248
J-86


ppm
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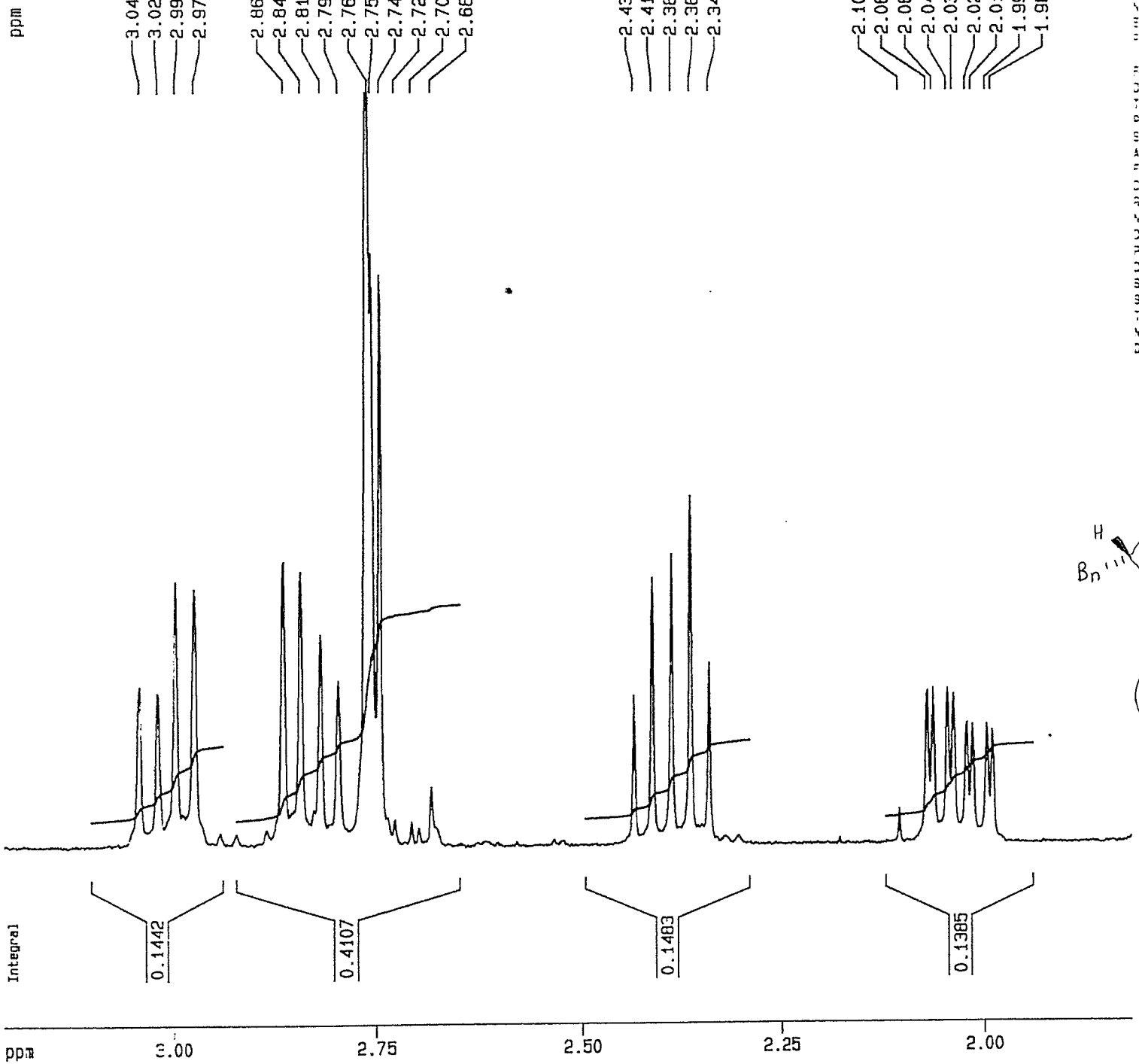
5.03
5.02
5.01
5.00
5.00
4.99
4.56
4.55
4.55
4.54
4.53
4.53
4.26
4.24
4.22
4.19
4.17
Integral
0.1263
0.1193
0.1252
ppm
5.00
4.75
4.50
4.25
H
H
Bn
O
O
H
23
1H
DR V K YADAV
RSIC NO.9248
J-86




```
NAME          186.hm
EXPNO              1
PROCNO             1

F2 - Acquisition Parameter
Date          970509
Time           13.36
PULPROG           zg
SOLVENT        CDCl3
AQ         3.4015260 s
FIDRES     0.147002 H
DW            103.8 u
RG              362
NUCLEUS          1H
D1         3.0000000 s
P1             6.9 u
DE             4.5 u
SFO1       300.1321838 M
SWH        4816.96 H
TD            32768
NS               16
DS                0
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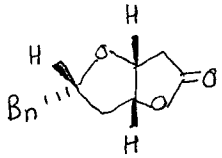
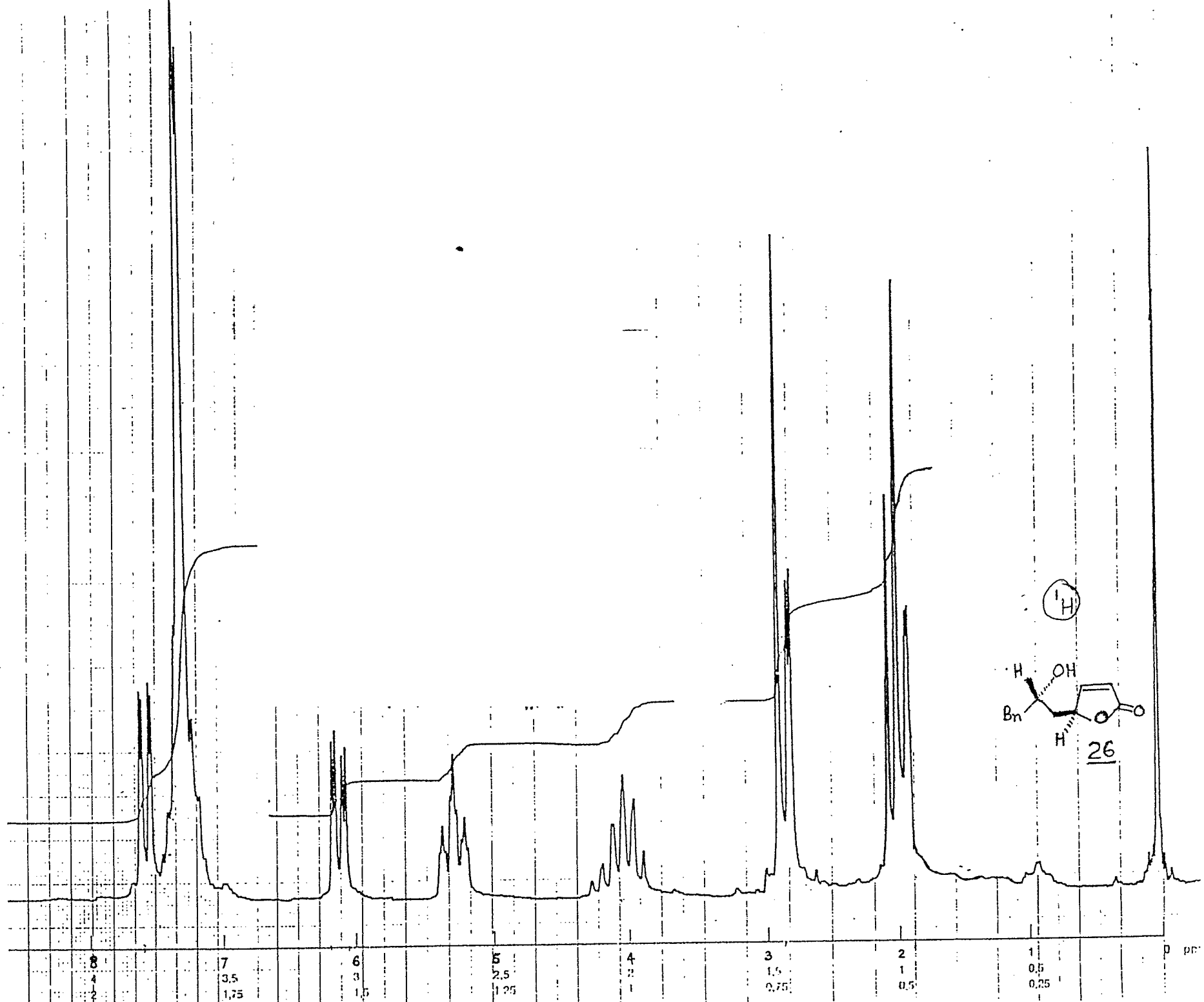


23

^{1}H

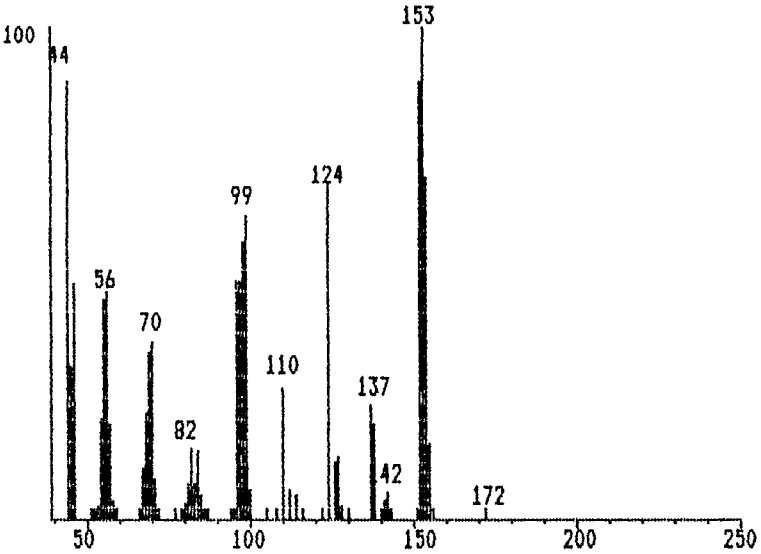
DR V K YADAV
RSIC NO.9248
J-86


^{1}H
H
OH
Bn
H
O
O
26
8
4
2
7
3,5
1,75
6
3
1,5
5
2,5
1,25
4
2
1
3
1,5
0,75
2
1
0,5
1
0,5
0,25
0
ppm

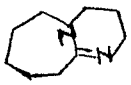
CENTRAL DRUG RESEARCH INSTITUTE
02-09-1996

FB0908X.LRP #2/ DR V K YADAV / IIT KANPUR [6075]
Date run : 02-09-1996 Operator : A.SONI /PRAKASH /SUNIL

Scan : 7 RT= 0:44 No.ions= 68 Base= 4.8%F TIC= 21609



Mass spectrum of



DBU with deuterium

CENTRAL DRUG RESEARCH INSTITUTE
02-09-1996

FB0908X.LRP #2/ DR V K YADAV / IIT KANPUR [6075]
Date run : 02-09-1996 Operator : A.SONI /PRAKASH /SUNIL

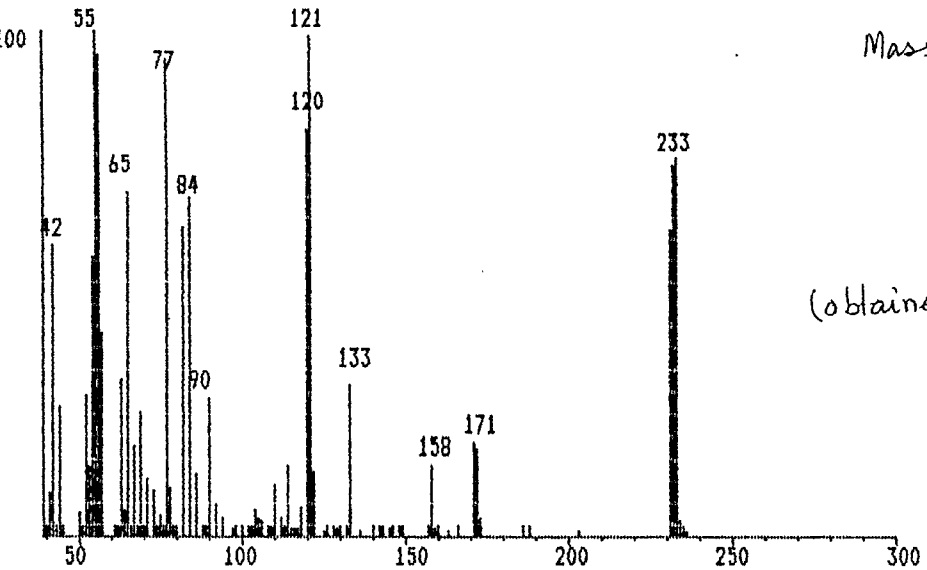
Scan : 7 RT= 0:44 No.ions= 28 Base= 4.8%F TIC= 8929

MASS	%BASE
172	2.3
156	2.8
155	18.3
154	69.1
153	100.0
152	88.7
151	2.3
143	3.2
142	8.7
141	7.1
140	3.2
138	21.6
137	25.4
130	3.4
129	6.4
127	15.6
127	8.5
126	14.3
125	26.8
124	67.3
123	4.8
122	4.1
116	4.6
115	8.1
114	5.8
113	7.4
112	9.3
111	28.1

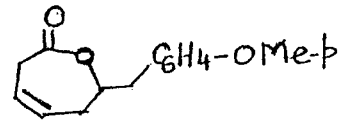
CENTRAL DRUG RESEARCH INSTITUTE
02-09-1996

FB0906.LRP 1/ DR VK YADAV/IIT KANPUR
Date run : 02-09-1996 Operator : A.SONI /PRAKASH /SUNIL

Scan : 18 RT= 2: 3 No.ions= 104 Base= 10.9%F TIC= 61373

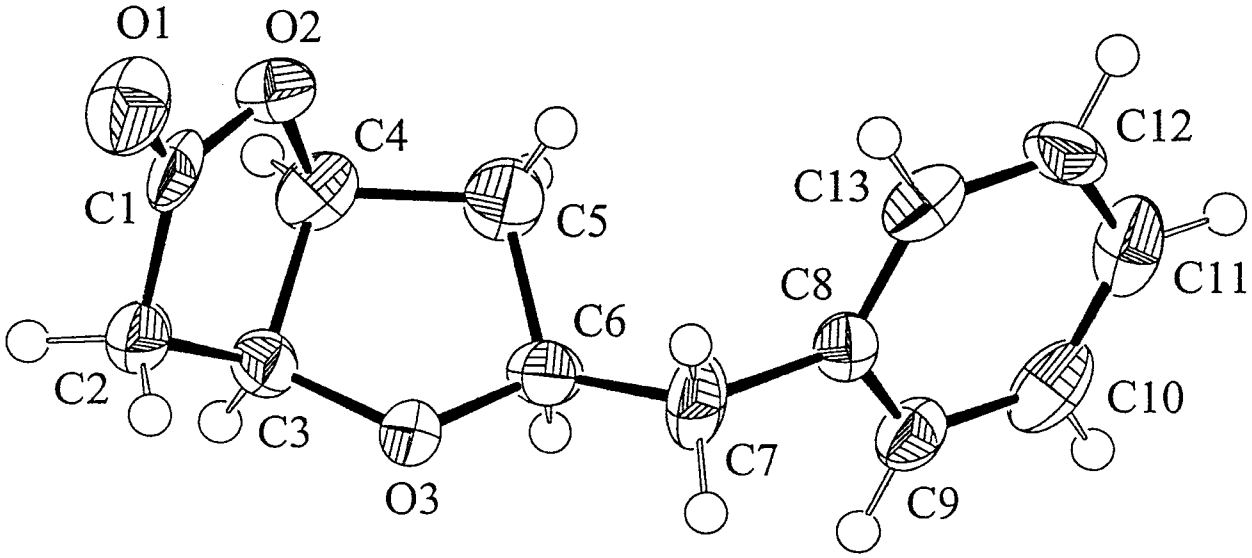


Mass spectrum of



7c with D
(obtained from equilibration expt.)

MASS	%BASE	MASS	%BASE
236	1.0	128	4.1
235	3.1	128	2.0
234	6.7	126	2.0
233	74.6	125	1.0
232	73.2	122	16.2
231	60.8	121	99.0
203	1.0	120	80.2
188	1.8	120	4.1
186	3.6	118	9.4
173	7.3	117	1.3
172	19.9	116	1.5
171	21.1	115	1.3
166	1.7	114	17.3
163	1.0	113	2.7
160	2.4	112	7.2
159	1.3	112	4.2
158	17.2	110	13.6
157	5.2		
149	4.0		
148	5.6		
146	5.6		
145	1.3		
143	1.7		
142	2.2		
140	1.8		
136	1.0		
133	31.9		
132	2.9		
130	4.8		
129	1.3		


O1
O2
C4
C1
C5
C13
C12
C11
C8
C6
C2
C3
O3
C7
C9
C10

Experimental

Data Collection

A colourless plate crystal of $C_{13}H_{14}O_3$ having approximate dimensions of 0.47 x 0.40 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC6S diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $18.58 < 2\theta < 23.08°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 15.237(2) Å

b = 20.217(3) Å

c = 7.329(1) Å

V = 2257.7(5) Å^3

For Z = 8 and F.W. = 218.25, the calculated density is 1.28 g/cm^3. The systematic absences of:

0kl: $k \neq 2n$

h0l: $l \neq 2n$

hk0: $h \neq 2n$

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of -103 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 55.1°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.39° with a take-off angle of 6.0°. Scans of $(1.52 + 0.34 \tan \theta)°$ were made at a speed of 4.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 4 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 400 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

A total of 2992 reflections was collected. The intensities of three representative reflection were measured after every 200 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.9 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included at geometrically idealized positions with C-H 0.95 Å and were not refined. The final cycle of full-matrix least-squares refinement[3] was based on 779 observed reflections (I > 3.00σ(I)) and 145 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.051$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)]} = 0.027$$

The standard deviation of an observation of unit weight[4] was 2.62. The weighting scheme was based on counting statistics and included a factor (p = 0.001) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.20 and -0.23 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SAPI91: Fan Hai-Fu (1991). Structure Analysis Programs with Intelligent Control, Rigaku Corporation, Tokyo. Japan.

(2) DIRDIF94: Beurskens, P.T., Admiraal. G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel. R. and Smits. J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables