

SUPPORTING INFORMATION

Synthesis of *o*-Nitrophenylphenyliodonium fluoride (NPIF, 1):

(a) *o*-Nitrophenylphenyliodonium iodide—To a stirred solution of *o*-iodonitrobenzene (5.0 g, 20 mmol) in H₂SO₄ (100 mL) was added K₂S₂O₈ (6.0 g, 22 mmol) in small portions followed by benzene (25 mL) at room temperature, and the mixture was stirred vigorously for 1.5 h. The reaction mixture was poured into ice (total volume 300 mL), and the insoluble material was removed by filtration. The filtrate was treated with aqueous KI (5 g/20 mL of H₂O), giving an orange precipitate that was filtered and washed thoroughly with H₂O (250 mL) followed by a small amount of acetone. The collected solid was dried over P₂O₅ under reduced pressure to give 7.95 g (87%) of *o*-nitrophenylphenyl-iodonium iodide. This procedure was developed from the method of Beringer et al. (ref 13a in the text of the paper. Note the typographical error in their original procedure: *o*-iodosonitrobenzene should be replaced with *o*-iodonitrobenzene). (b) *o*-Nitrophenylphenyliodonium fluoride—To a vigorously stirred solution of AgF (1.0 g, 7.9 mmol) in H₂O (30 mL) was added the iodonium iodide (3.6 g, 7.9 mmol) and the mixture was stirred for several hours. After removal of the insoluble material by filtration, the solution was concentrated below 30 °C under reduced pressure (< 5 torr). The residue was diluted with 8 mL of MeCN and crystallized at 0 °C to give NPIF (1, 1.80 g first crop, 0.33 g second crop; total 2.13 g, 78%).

1: ¹H NMR (DMSO-*d*₆) δ: 7.54 (2H, t, *J* = 7.5 Hz), 7.66-7.78 (4H, m), 7.13 (2H, d, *J* = 7.5 Hz), 8.29 (1H, d, *J* = 7.5 Hz). ¹³C NMR (DMSO-*d*₆) δ: 116.2, 120.0, 126.2, 131.6, 131.7, 131.8, 135.0, 136.2, 136.3, 148.0.

General Procedure for *o*-Nitrophenylation Reaction:

General procedure for arylation reaction: To a stirred solution of NPIF 1 (345 mg, 1 mmol) in dry DMSO-CH₂Cl₂ (1.4 mL-2.1 mL) was added a TMS ether 2 (1 mmol) dropwise at *ca.* -40 °C (CO₂-MeCN) under nitrogen. The mixture was stirred for 2 h at this temperature, and allowed to warm up to room temperature gradually over 2-3 h. The reaction mixture was poured into H₂O (10

mL), and the whole was extracted with ether (10 mL x 3). The extracts were washed with brine, dried over MgSO_4 , and concentrated. The residue was purified by flash column chromatography on silica gel (EtOAc:hexane = 1:30 – 1:5) to give the aryl ketone (**3**). Aryl ketones **3** were characterized from ^1H , ^{13}C NMR, and IR spectra. See supporting information for the spectral data as well as copies of actual spectra.

3a: ^1H NMR (CDCl_3) δ : 1.75-1.89 (2H, m), 2.01-2.12 (2H, m), 2.15-2.18 (1H, m), 2.33-2.38 (1H, m), 2.50-2.53 (2H, m), 4.28 (1H, dd, $J = 5.5, 12.5$ Hz), 7.34 (1H, d, $J = 8$ Hz), 7.39 (1H, t, $J = 8$ Hz), 7.57 (1H, t, $J = 8$ Hz), 7.95 (1H, d, $J = 8$ Hz). ^{13}C NMR (CDCl_3) δ : 25.2, 26.9, 33.4, 41.9, 53.2, 124.6, 127.5, 130.1, 133.0, 133.8, 149.0, 207.9.

3b: Major isomer; ^1H NMR (CDCl_3) δ : 1.36 (3H, d, $J = 7$), 1.86-1.90 (2H, m), 2.01-2.12 (3H, m), 2.30-2.39 (1H, m), 2.67-2.75 (1H, m), 4.52 (1H, dd, $J = 5, 13$ Hz), 7.33 (1H, d, $J = 8$ Hz), 7.40 (1H, dt, $J = 1, 8$ Hz), 7.58 (1H, dt, $J = 1, 8$ Hz), 7.97 (1H, dd, $J = 1, 8$ Hz). ^{13}C NMR (CDCl_3) δ : 17.0, 20.3, 32.7, 33.0, 44.8, 49.2, 124.9, 127.7, 130.1, 133.1, 134.2, 149.3, 211.8. Assignable peaks of minor isomer; ^1H NMR (CDCl_3) δ : 1.07 (3H, d, $J = 6.5$), 4.35 (1H, dd, $J = 5, 13$ Hz). ^{13}C NMR (CDCl_3) δ : 14.6, 25.5, 34.2, 36.7, 45.8, 53.1, 124.8, 129.9, 133.0, 210.0.

3c: ^1H NMR (CDCl_3) δ : 0.87 (3H, d, $J = 6$), 1.60 (1H, dq, $J = 3, 13$ Hz), 1.87 (1H, tq, $J = 4, 13.5$ Hz), 2.03-2.06 (1H, m), 2.11-2.15 (1H, m), 2.26-2.31 (1H, m), 2.47 (1H, dt, $J = 6, 14$ Hz), 2.55-2.58 (1H, m), 3.82 (1H, br s), 7.28 (1H, d, $J = 8$ Hz), 7.40 (1H, t, $J = 18$ Hz), 7.58 (1H, t, $J = 8$ Hz), 7.92 (1H, d, $J = 8$ Hz). ^{13}C NMR (CDCl_3) δ : 20.9, 25.2, 34.1, 39.4, 41.3, 60.7, 124.7, 127.7, 131.8, 132.6, 132.8, 150.1, 207.8.

3d: Major isomer; ^1H NMR (CDCl_3) δ : 1.10 (3H, d, $J = 6.5$ Hz), 1.54-1.63 (1H, m), 1.84 (1H, br q, $J = 12.5$ Hz), 2.11-2.19 (2H, m), 2.28-2.33 (1H, m), 2.50-2.55 (1H, m), 2.56 (1H, br dt, $J = 6, 14$ Hz), 4.36 (1H, dd, $J = 5, 13$ Hz), 7.35 (1H, d, $J = 8$ Hz), 7.42 (1H, t, $J = 8$ Hz), 7.60 (1H, t, $J = 8$ Hz), 7.99 (1H, dd, $J = 1, 8$ Hz). ^{13}C NMR (CDCl_3) δ : 21.3, 32.2, 35.1, 41.3, 41.7, 52.4, 124.8, 127.8, 130.3, 133.1, 133.9, 149.2, 208.3. Assignable peaks of minor isomer; ^1H NMR (CDCl_3) δ : 1.32 (3H, d, $J = 7$), 1.92-1.95 (1H, m), 2.40-2.45 (1H, m), 2.26-

2.72 (1H, m), 4.45 (1H, dd, $J = 5.5, 12.5$ Hz). ^{13}C NMR (CDCl_3) δ : 17.6, 27.2, 32.1, 37.5, 39.2, 48.2, 130.6.

3e: Less polar isomer; ^1H NMR (CDCl_3) δ : 1.74-1.84 (2H, m), 2.03 (1H, dq, $J = 3, 12.5$ Hz), 2.13-2.22 (1H, m), 2.27-2.34 (2H, m), 3.43 (3H, s), 3.66 (1H, br t, $J = 3$ Hz), 4.85 (1H, dd, $J = 5, 12.5$ Hz), 7.39-7.45 (2H, m), 7.59 (1H, dt, $J = 1, 8$ Hz), 7.91 (1H, dd, $J = 1, 8$ Hz). ^{13}C NMR (CDCl_3) δ : 19.9, 33.8, 34.4, 48.4, 57.7, 84.1, 124.5, 127.8, 130.3, 132.8, 133.0, 149.6, 208.9. More polar isomer; ^1H NMR (CDCl_3) δ : 1.73 (1H, dq, $J = 3, 12.5$ Hz), 1.90-2.05 (2H, m), 2.08-2.12 (1H, m), 2.34-2.38 (1H, m), 2.46-2.51 (1H, m), 3.47 (3H, s), 4.11 (1H, dd, $J = 6, 11.5$ Hz), 4.40 (1H, dd, $J = 5, 13$ Hz), 7.39 (1H, d, $J = 7$ Hz), 7.43 (1H, t, $J = 7.5$ Hz), 7.62 (1H, t, $J = 7.5$ Hz), 8.03 (1H, dd, $J = 1, 8$ Hz). ^{13}C NMR (CDCl_3) δ : 23.4, 33.3, 35.1, 51.7, 58.0, 84.6, 125.0, 127.9, 129.6, 132.9, 133.3, 148.7, 206.5.

3f: ^1H NMR (CDCl_3) δ : 1.93-2.02 (1H, m), 2.13-2.22 (2H, m), 2.33-2.40 (1H, m), 2.44-2.56 (2H, m), 3.89 (1H, dd, $J = 8.5, 11.5$ Hz), 7.24 (1H, d, $J = 8$ Hz), 7.38 (1H, dt, $J = 1, 7.5$ Hz), 7.53 (1H, dt, $J = 1, 7.5$ Hz), 7.95 (1H, dd, $J = 1, 8$ Hz). ^{13}C NMR (CDCl_3) δ : 20.8, 31.2, 37.8, 53.8, 125.2, 127.9, 131.4, 133.3, 133.4, 149.1, 215.4.

3g: Major isomer; ^1H NMR (CDCl_3) δ : 1.18 (3H, d, $J = 6.5$ Hz), 1.54-1.62 (1H, m), 2.03-2.10 (1H, m), 2.33-2.41 (2H, m), 2.46-2.51 (1H, m), 3.84 (1H, dd, $J = 8.5, 12.5$ Hz), 7.22 (1H, d, $J = 8$ Hz), 7.38 (1H, t, $J = 8$ Hz), 7.54 (1H, t, $J = 7.5$ Hz), 7.94 (1H, d, $J = 8$ Hz). ^{13}C NMR (CDCl_3) δ : 14.7, 29.7, 30.1, 44.3, 53.3, 125.2, 127.9, 131.3, 133.3, 133.9, 149.2, 217.2. Assignable peaks of minor isomer; ^1H NMR (CDCl_3) δ : 1.56-1.67 (1H, m), 1.78-1.83 (1H, m), 2.10-2.17 (1H, m), 2.24-2.30 (1H, m), 2.49-2.56 (1H, m), 4.16 (1H, br t, $J = 10$ Hz), 7.17 (1H, d, $J = 8$ Hz). ^{13}C NMR (CDCl_3) δ : 15.3, 28.6, 41.6, 52.0, 125.0, 127.7, 130.4, 133.1, 217.6.

General Procedure for TiCl_3 Reduction:

Aqueous TiCl_3 can be purchased from Aldrich. Alternatively, it can be prepared inexpensively by dissolving $\text{TiCl}_3 \cdot 1/3 \text{AlCl}_3$ (available from Strem Chemicals, Inc.) in water (CAUTION! Strong

exotherm!), filtering off the precipitate. The concentration of the purple solution can be determined by titrating with 0.1 M KBrO_3 , wherein disappearance of the purple color indicates the endpoint. To a purple solution of 1.44 M TiCl_3 in H_2O (0.56 mL, 0.8 mmol of TiCl_3) was added 2.5 M NH_4OAc in H_2O (1 mL) followed by acetone (1 mL) at room temperature. The purple color turned dark brown, and the mixture formed a two-phase system. A solution of a nitro ketone **3** (0.1 mmol) in acetone (1 mL) was added dropwise at room temperature with vigorous stirring. After 15 min, the reaction mixture was diluted with H_2O (10 mL), and extracted with EtOAc (10 mL x 3). The extracts were washed with NaHCO_3 aqueous solution followed by NaCl aqueous solution, then dried over Na_2SO_4 and concentrated. The residue was purified by flash column chromatography on silica gel (EtOAc:hexane = 1:60 – 1:50, containing a small amount of Et_3N) to give the indole product (**4**). Indoles **4a**,² **4b**,³ **4d**,¹ **4e**,⁴ **4f**,² **4g**³ were identified with reported ^1H NMR spectra. The known indole **4c**¹ was characterized from ^1H spectrum.

4c: ^1H NMR (CDCl_3) δ : 1.37 (3H, d, $J = 7$ Hz), 1.55-1.59 (1H, m), 1.79-1.83 (1H, m), 1.95-2.00 (2H, m), 2.69-2.71 (2H, m), 3.11-3.13 (1H, m), 7.06-7.11 (2H, m), 7.28 (1H, d, $J = 7.5$ Hz), 7.57 (1H, d, $J = 7.5$ Hz), 7.64 (1H, br s).

4h: ^1H NMR (CDCl_3) δ : 0.69 (3H, s), 0.83 (3H, s), 0.87 (3H, d, $J = 6.5$ Hz), 0.87 (3H, d, $J = 7$ Hz), 0.92 (3H, d, $J = 6.5$ Hz), 0.69-1.65 (25H, m), 1.84-1.86 (1H, m), 2.00-2.04 (1H, m), 2.67-2.78 (2H, m), 6.99 (1H, t, $J = 8$ Hz), 7.04 (1H, t, $J = 8$ Hz), 7.25 (1H, d, $J = 8$ Hz), 7.67 (1H, br s), 7.70 (1H, d, $J = 8$ Hz). ^{13}C NMR (CDCl_3) δ : 12.1, 12.2, 18.7, 20.6, 21.8, 22.6, 22.8, 23.9, 24.2, 25.0, 28.0, 28.3, 31.9, 34.1, 35.2, 35.8, 36.2, 37.0, 39.5, 40.2, 42.6, 46.1, 52.7, 56.3, 56.6, 110.4, 112.3, 118.8, 119.9, 120.2, 128.0, 132.9, 136.2.

- (1) Stoermer, D.; Heathcock, C. H. *J. Org. Chem.* **1993**, *58*, 564-568.
- (2) Wender, P. A.; Cooper, C. B. *Tetrahedron* **1986**, *42*, 2985-2991.
- (3) Naruse, Y.; Ito, Y.; Inagaki, S. *J. Org. Chem.* **1991**, *56*, 2256-2258.
- (4) Owellen, R. J. *J. Org. Chem.* **1974**, *39*, 69-72.

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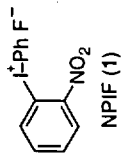
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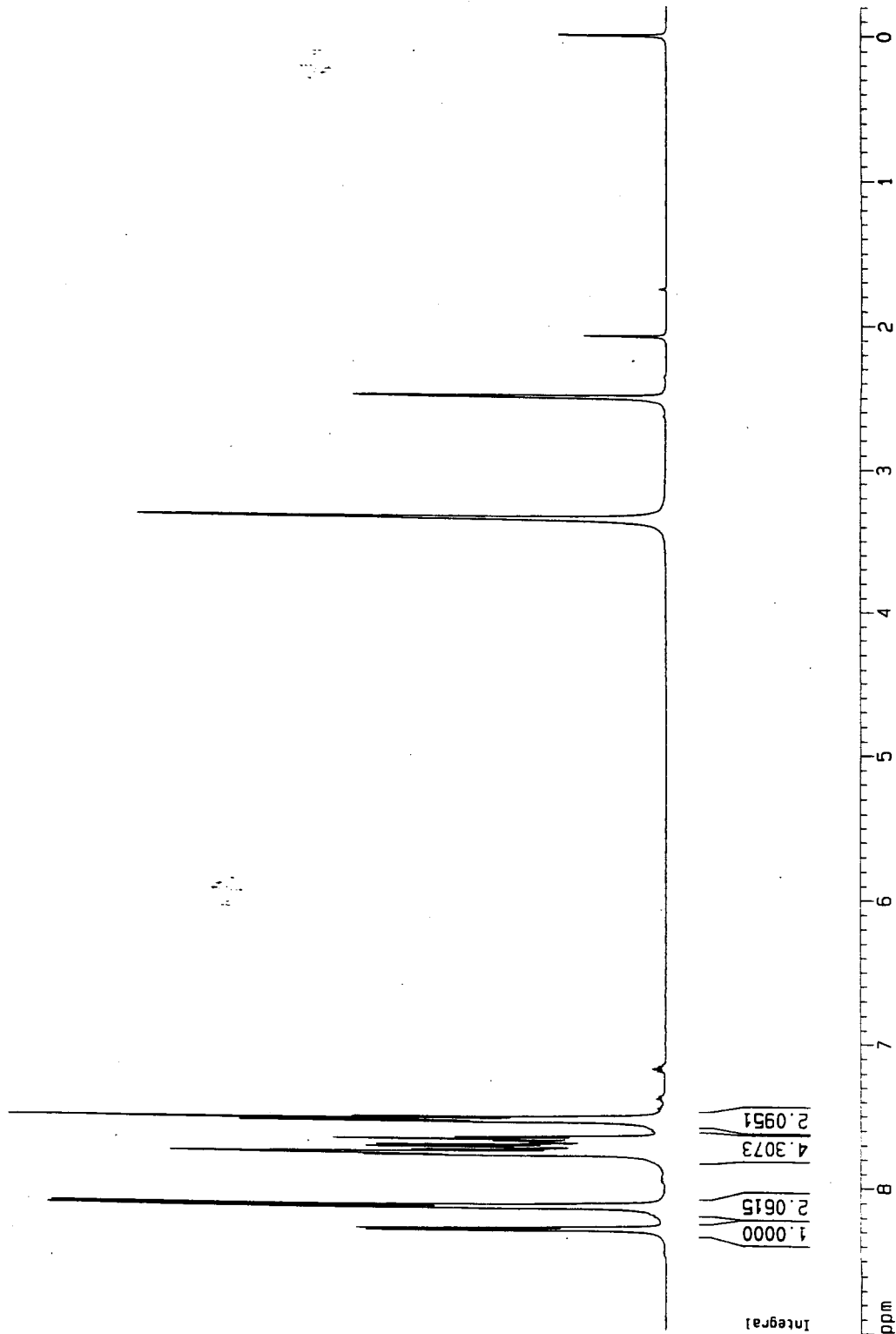
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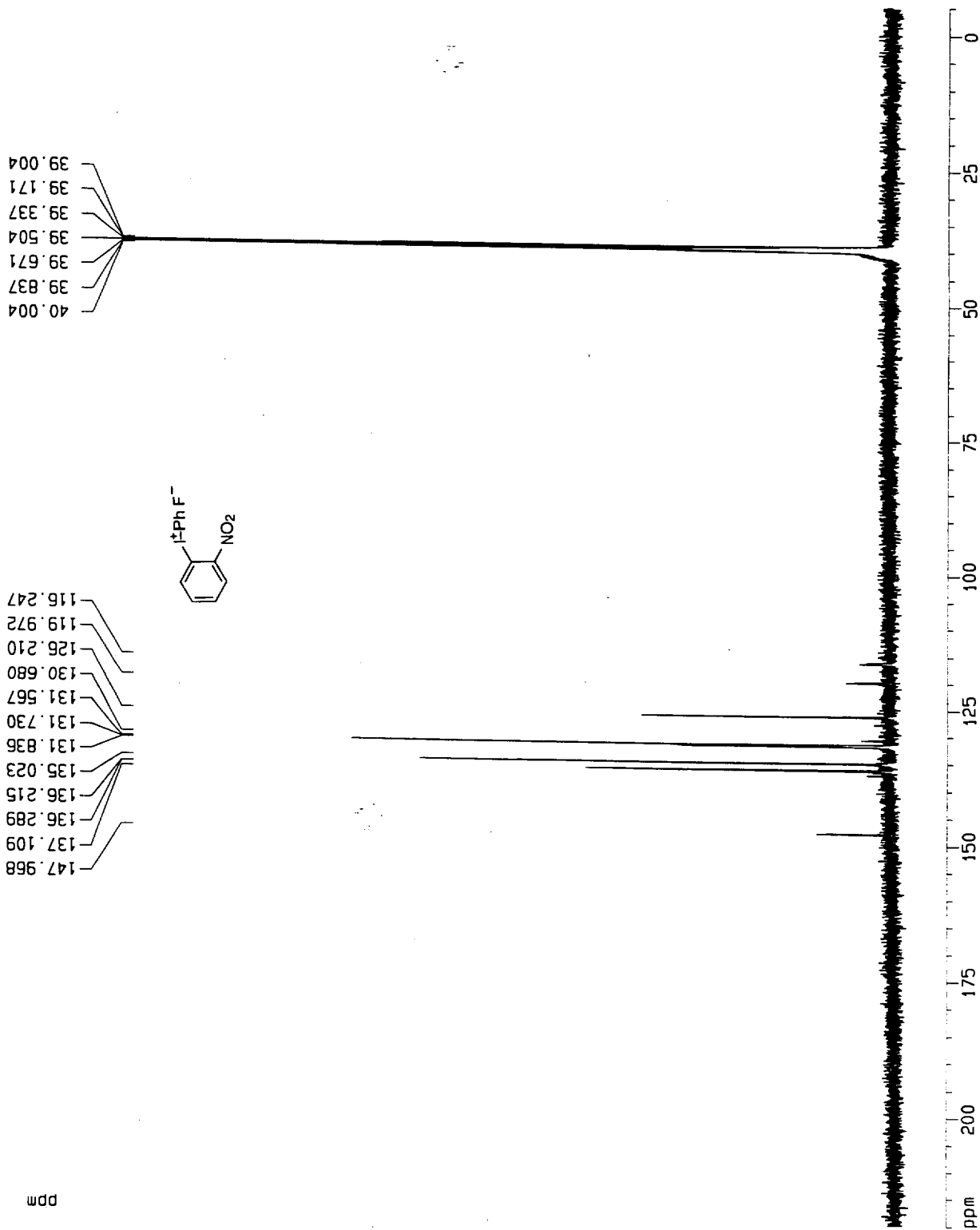
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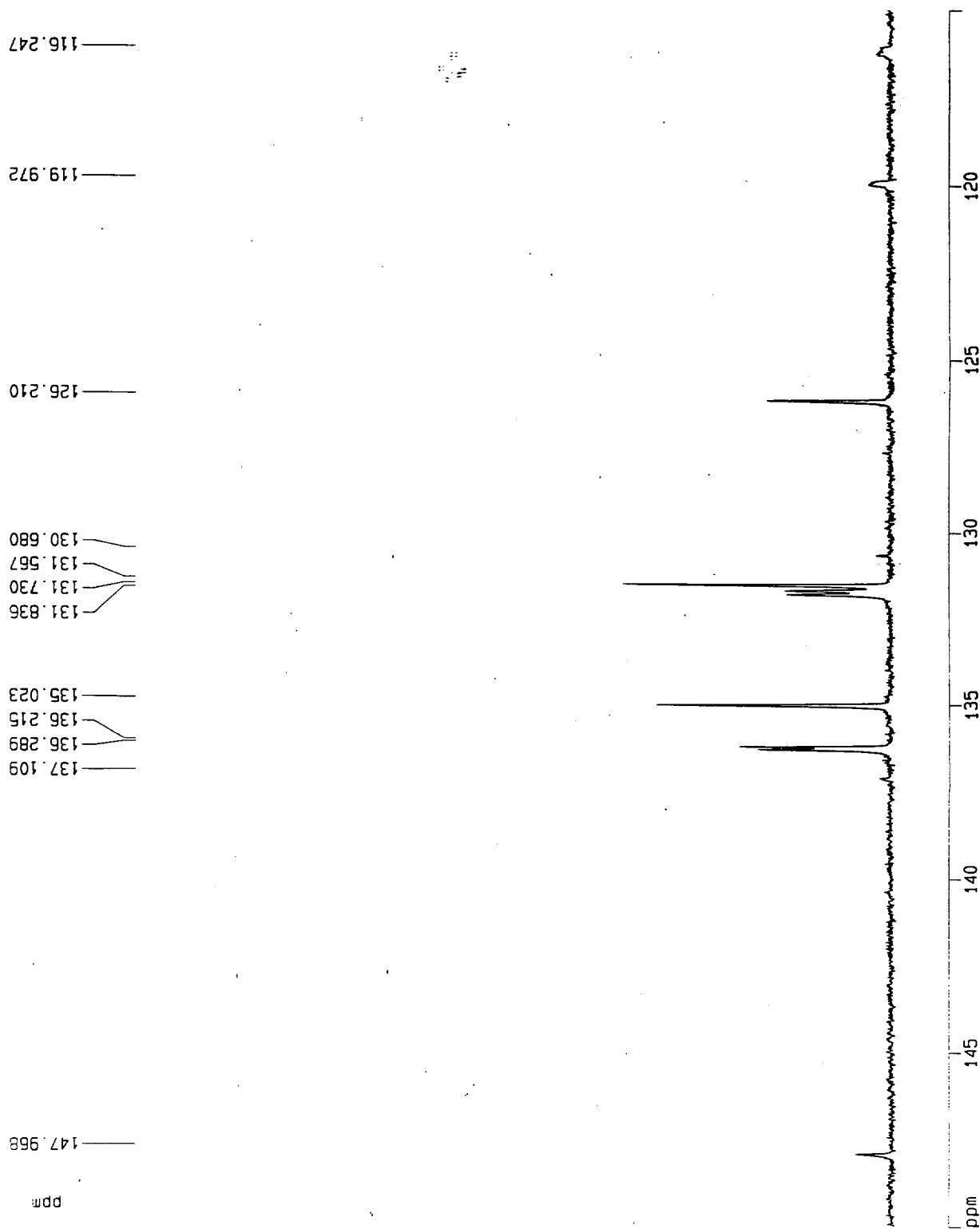


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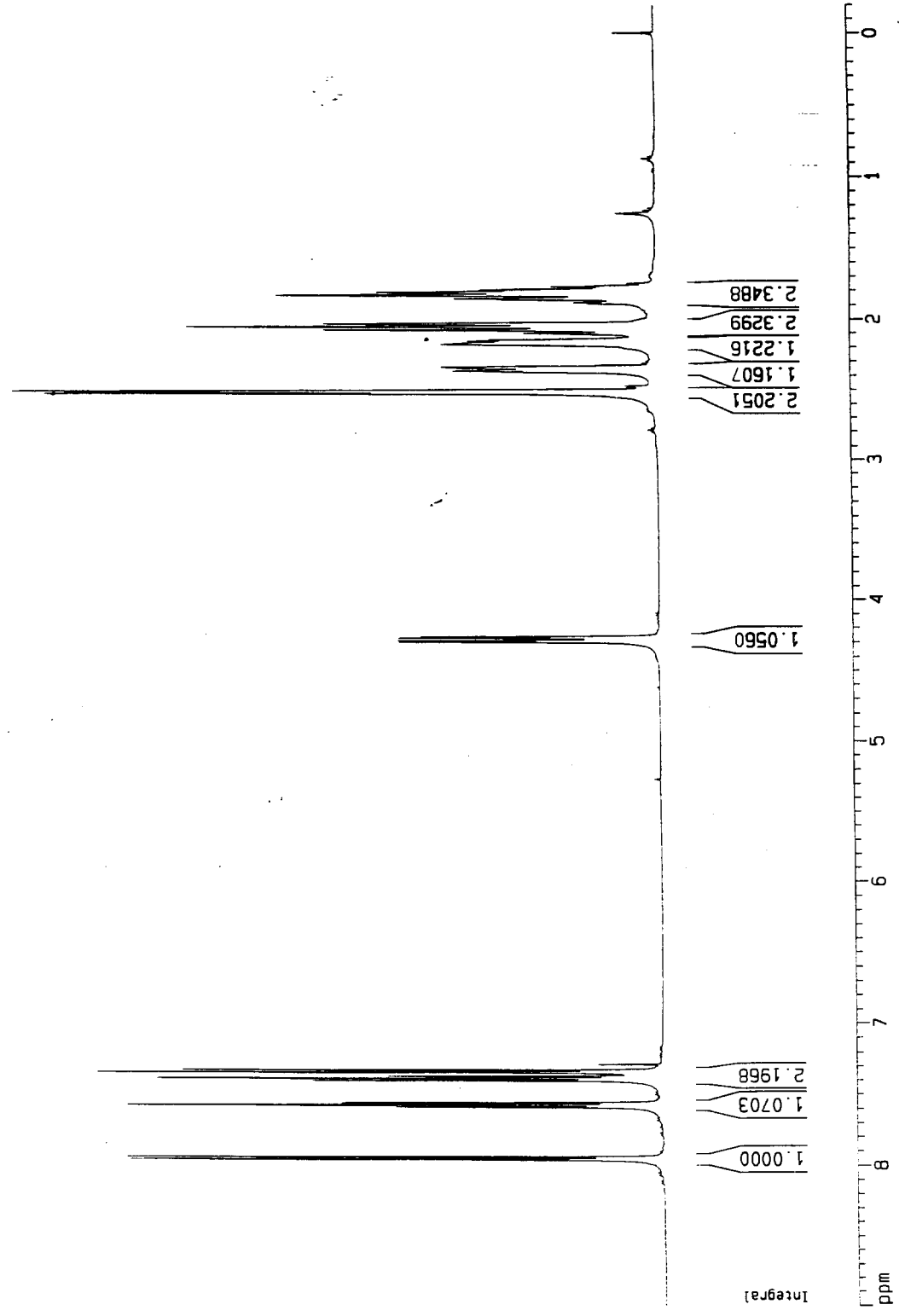
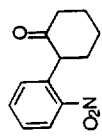


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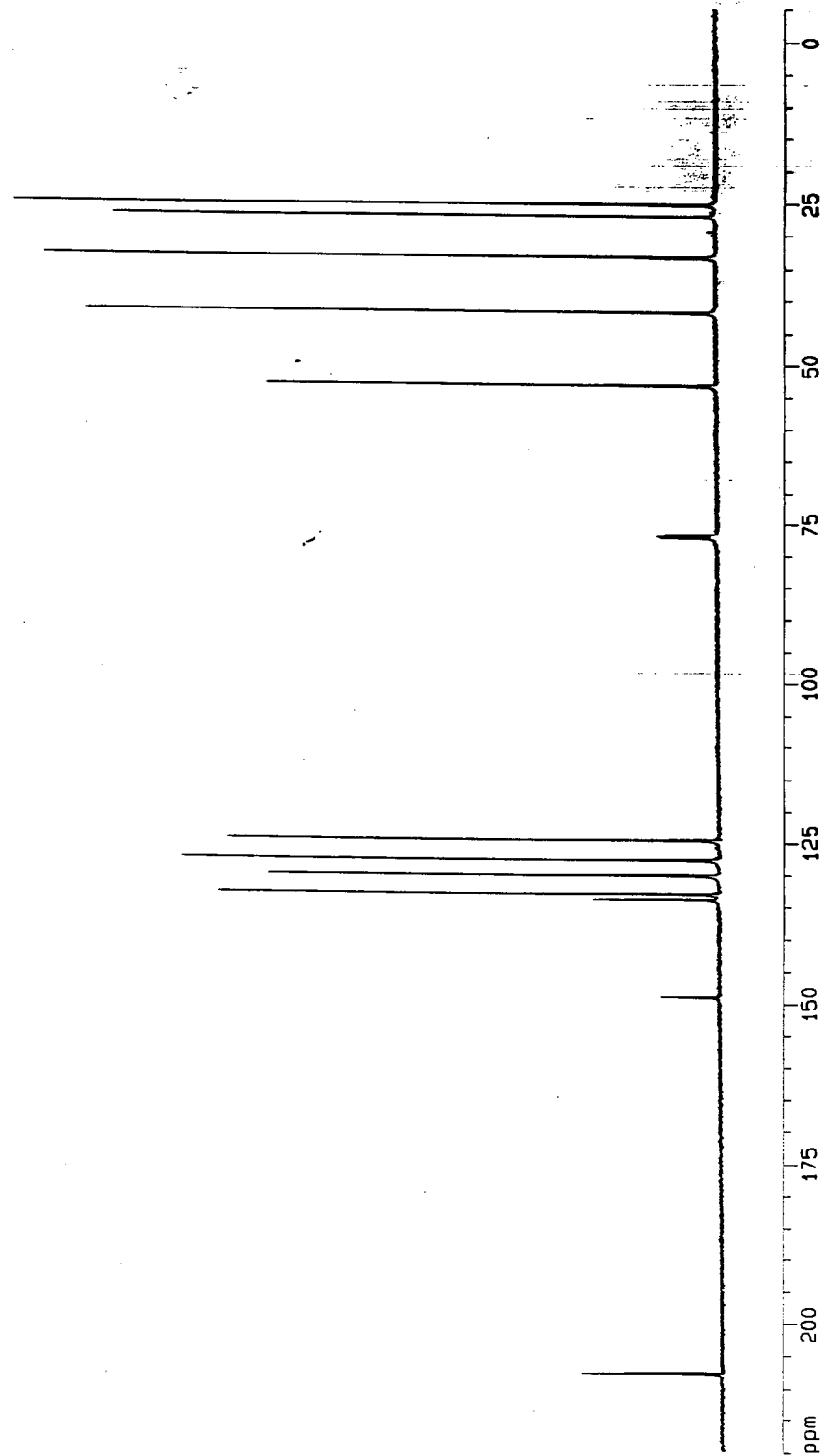
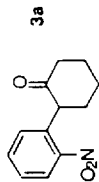
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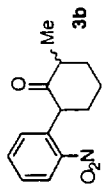
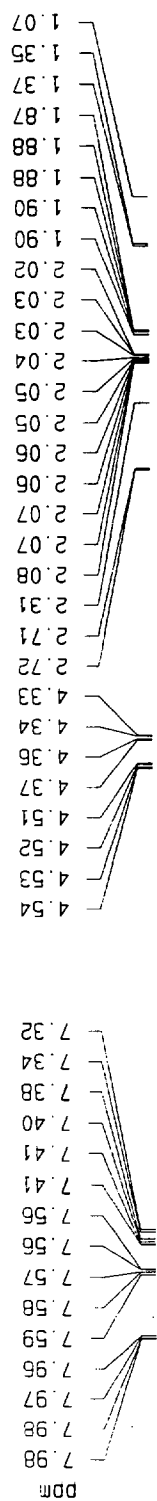
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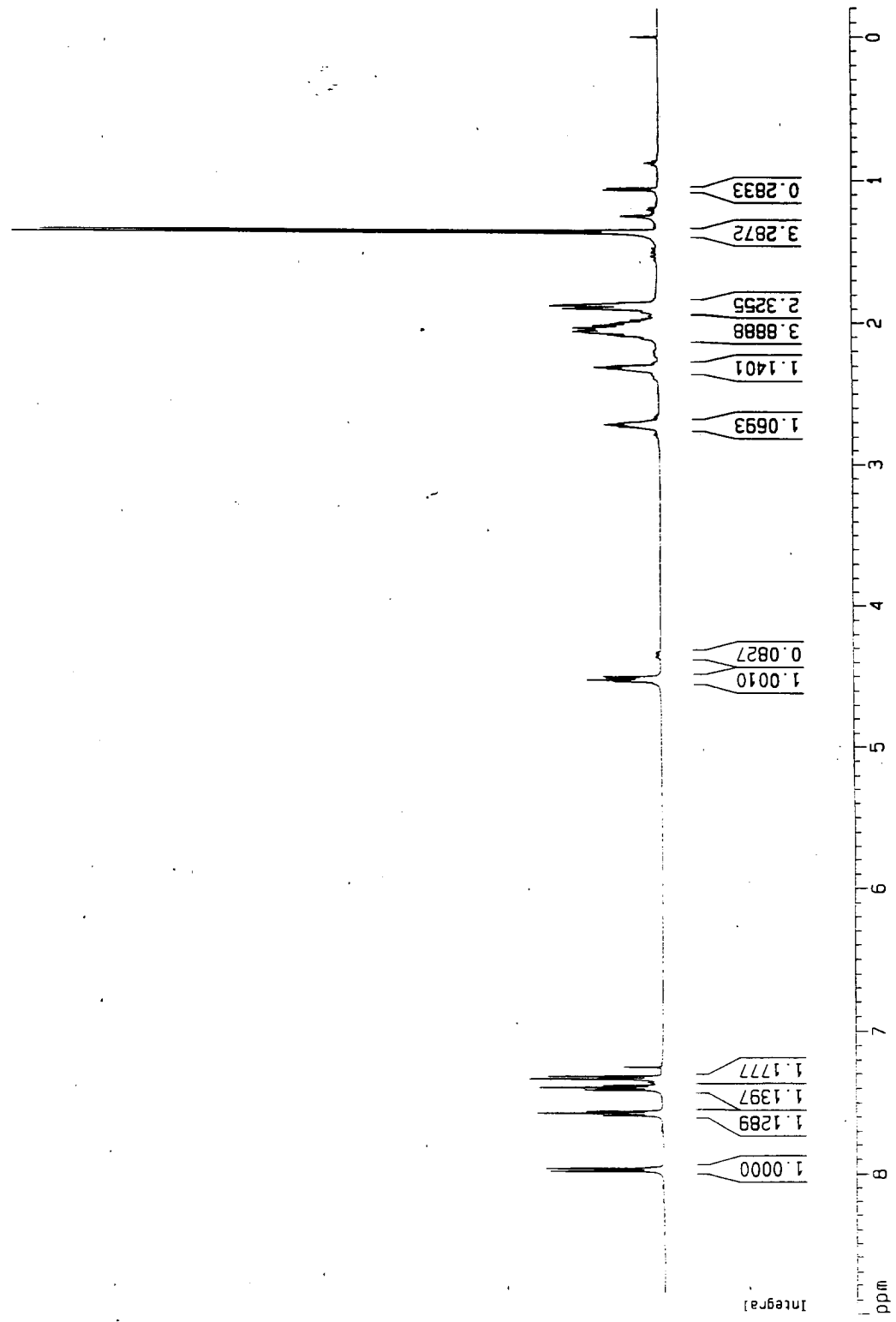
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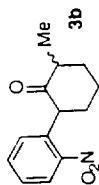
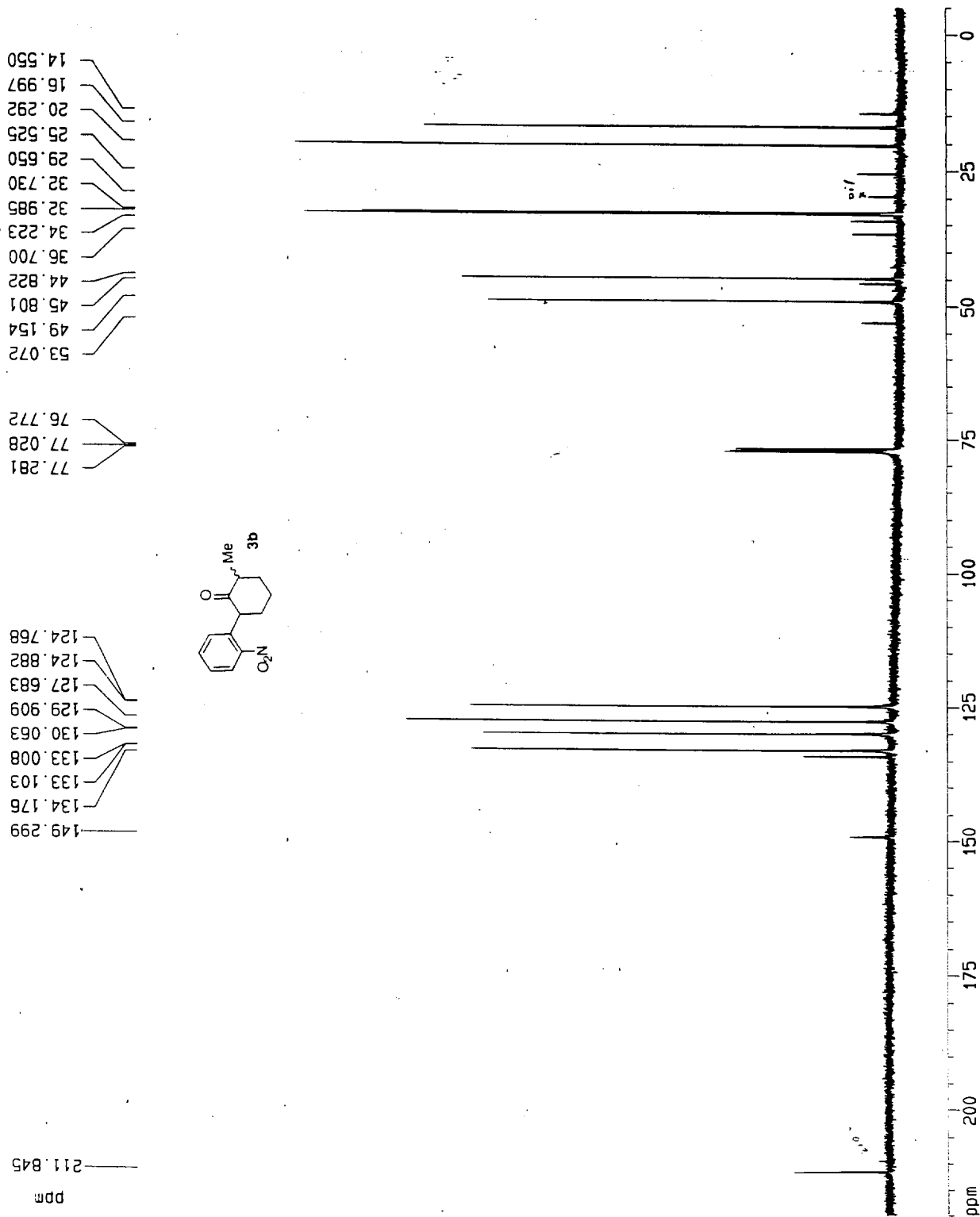


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 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577982 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 27666.71 Hz
 F2 -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.25000 ppm/cm
 HZCM 1414.77527 Hz/cm



0.866
0.878
1.582
1.588
1.606
1.612
1.858
1.885
2.027
2.030
2.054
2.057
2.114
2.120
2.127
2.464
2.476
2.492
2.546
2.551
2.555
2.579
3.818

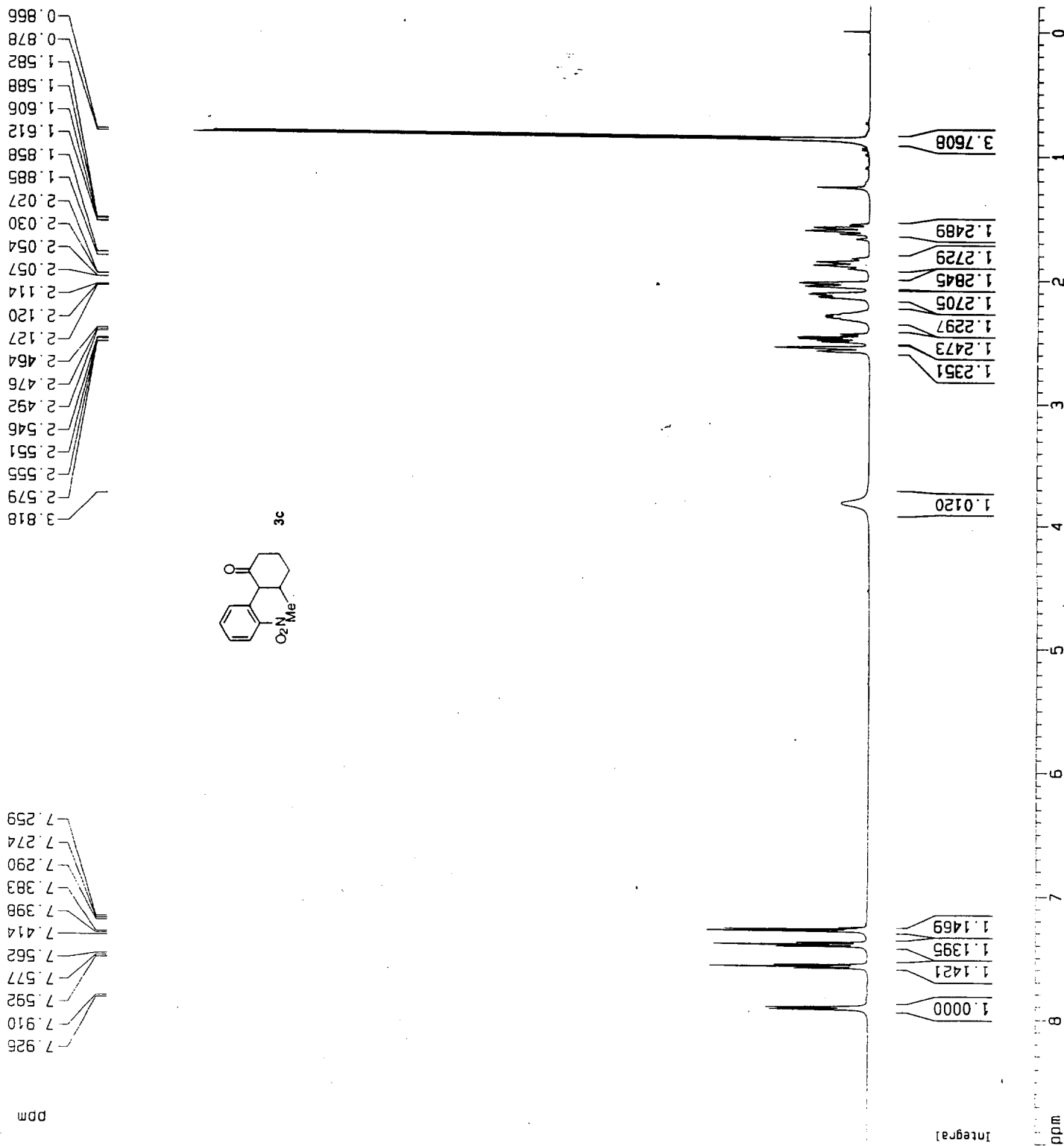
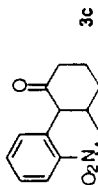
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7.274
7.290
7.383
7.398
7.414
7.562
7.577
7.592
7.910
7.926

Current Data Parameters
NAME default
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 990608
Time 19.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDC13
NS 8
DS 0
SWH 5208.333 Hz
FIDRES 0.158946 Hz
AQ 3.145779 sec
RG 32
DM 96.000 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 120.00 dB
CPDPRG2
PCPD2 100.00 usec
SF02 500.1300000 MHz
NUC2 off
PL2 120.00 dB
P1 1.0000000 sec
P1 5.00 usec
SF01 500.1320118 MHz
NUC1 1H
PL1 0.00 dB

F2 - Processing parameters
SI 16384
SF 500.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 4501.17 Hz
F2P -0.200 ppm
F2 -100.03 Hz
PPHM 0.46000 ppm/cm
HZCM 230.05980 Hz/cm



Current Data Parameters
 NAME default
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990608
 Time 20.22

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 300
 DS 0

SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K

d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB

D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7578007 MHz
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 SSB 0
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 GB 0
 PC 1.40

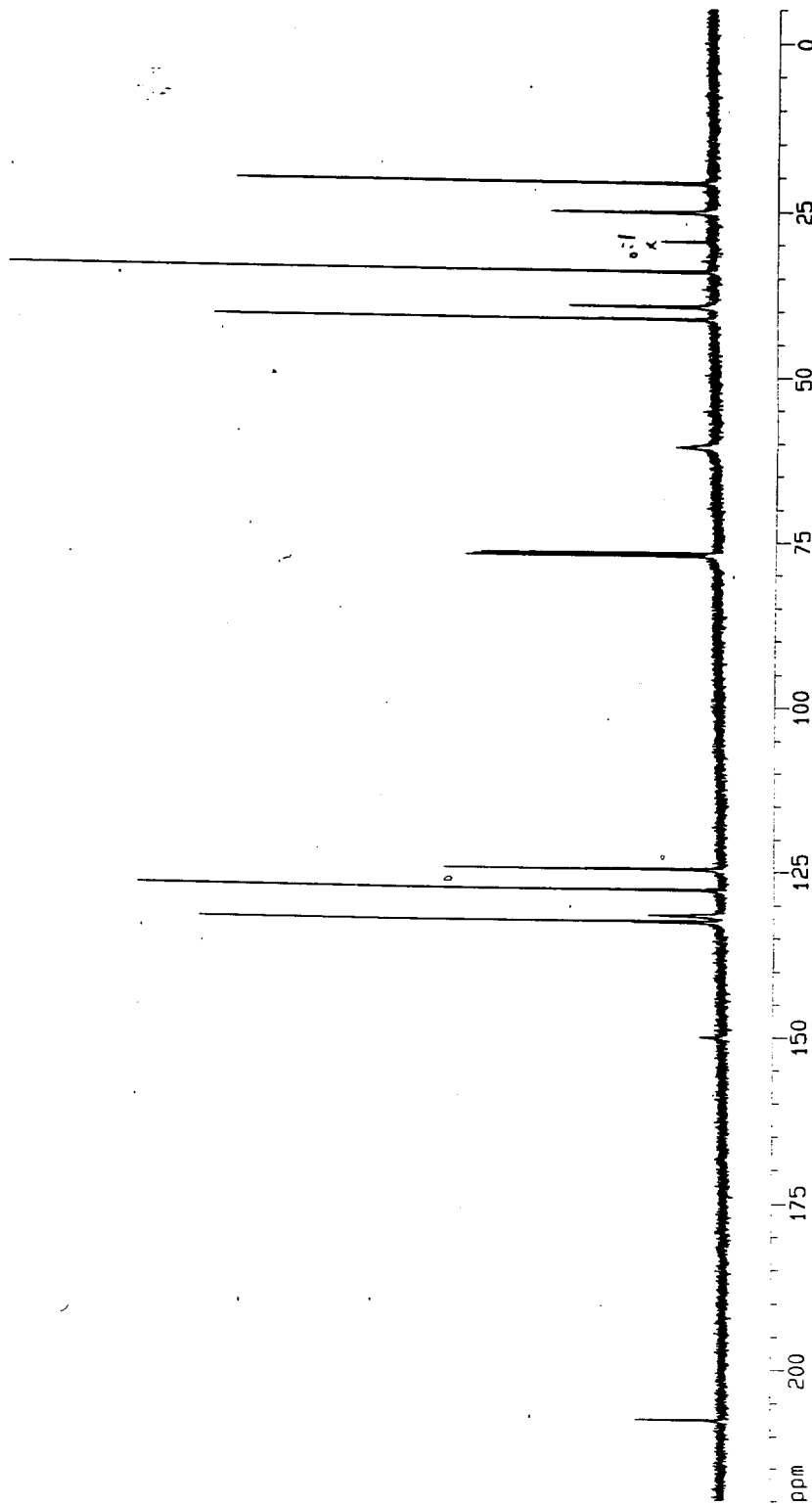
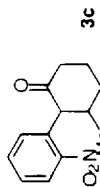
1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 27666.71 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.25000 ppm/cm
 HZCM 1414.77527 Hz/cm

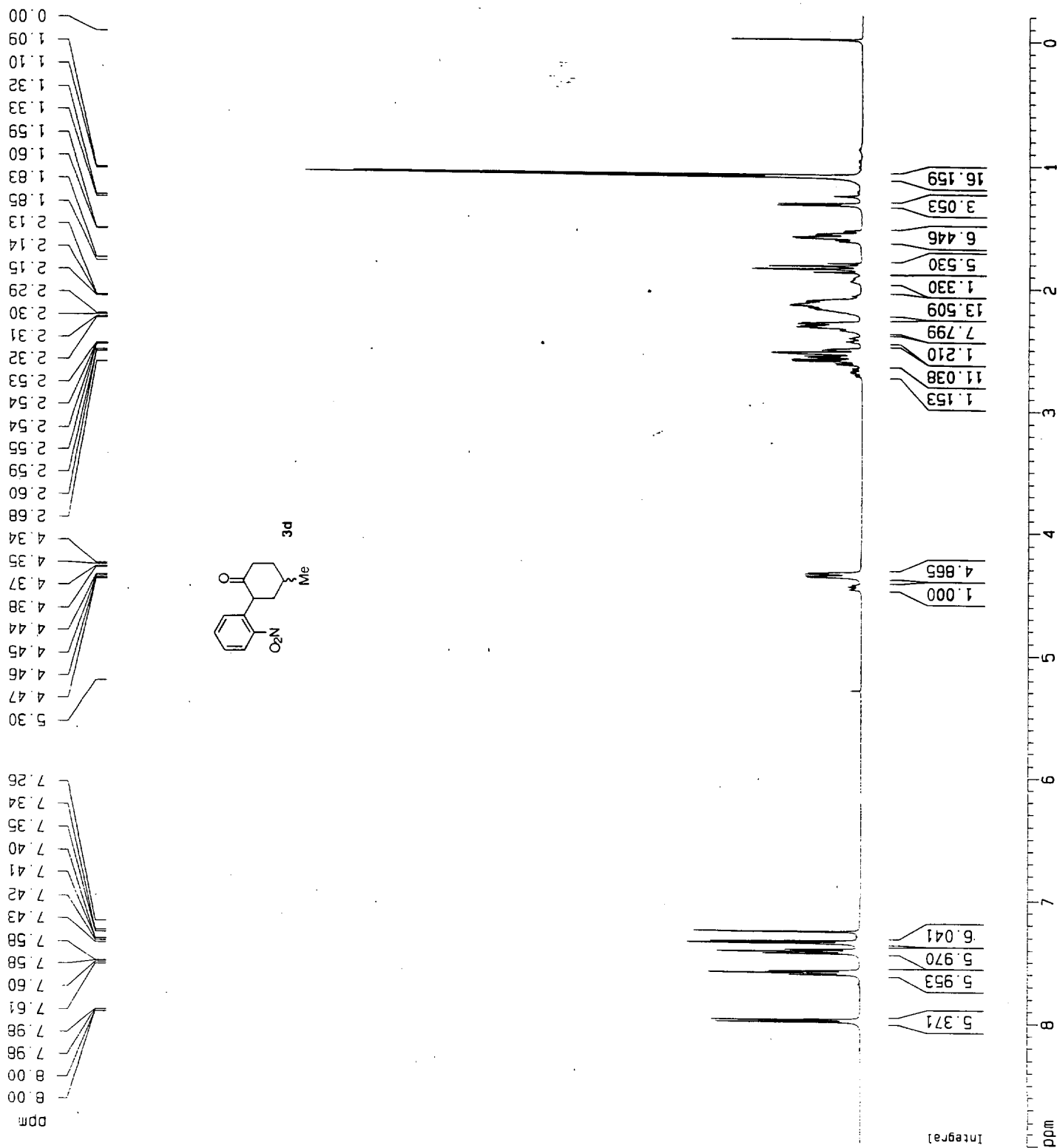
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 29.623
 34.104
 39.377
 41.286
 60.717
 76.762
 77.018
 77.271

124.726
 127.702
 131.791
 132.615
 132.785

207.779

ppm





Current Data Parameters
 NAME DEFAULT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990529
 Time 12.47
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 128
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300116 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm

Current Data Parameters
 NAME DEFAULT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990529
 Time 11.40
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.0258036 sec
 RG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7577958 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

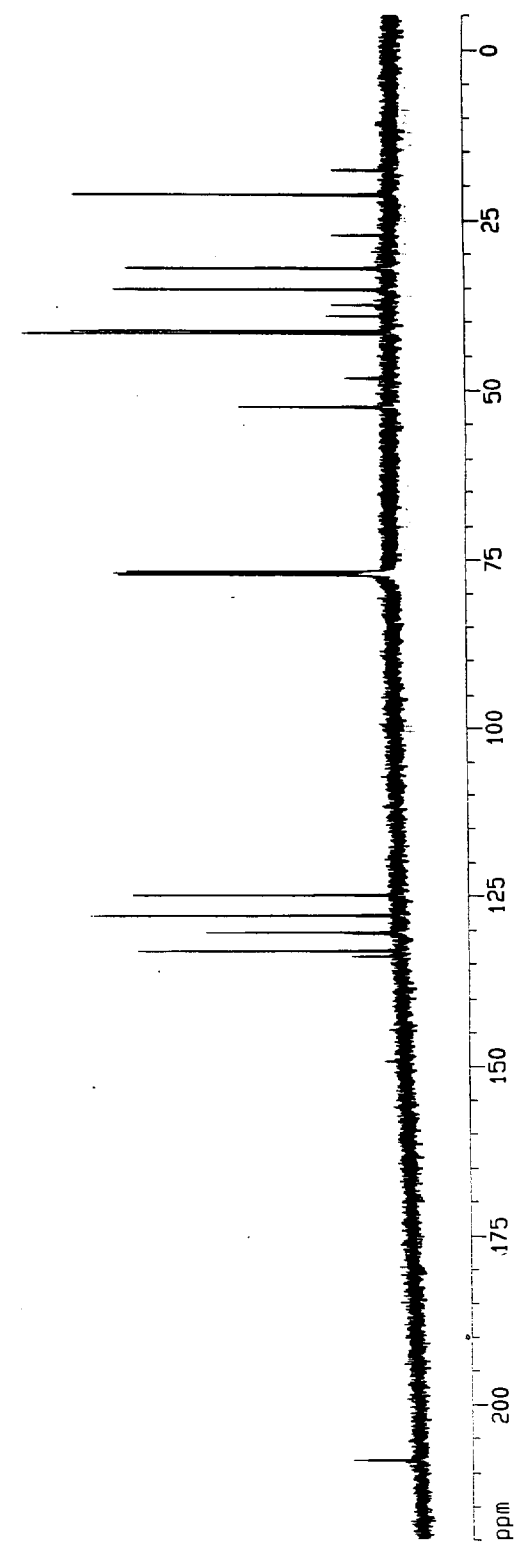
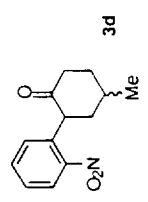
1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 27666.71 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.25000 ppm/cm
 HZCM 1414.77527 Hz/cm

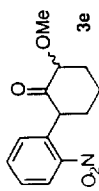
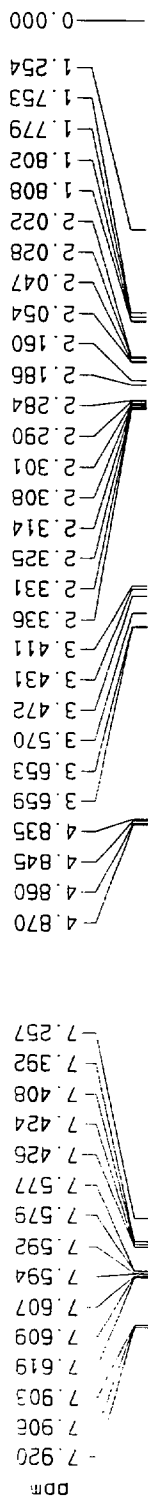
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21.303
27.206
32.088
32.178
35.143
37.497
39.205
41.306
41.690
48.178
52.429

76.745
77.000
77.253

124.911
127.777
130.326
130.592
133.097
133.904

208.274
ppm





Current Data Parameters
 NAME DEFAULT
 EXPNO 1
 PROCNO 1

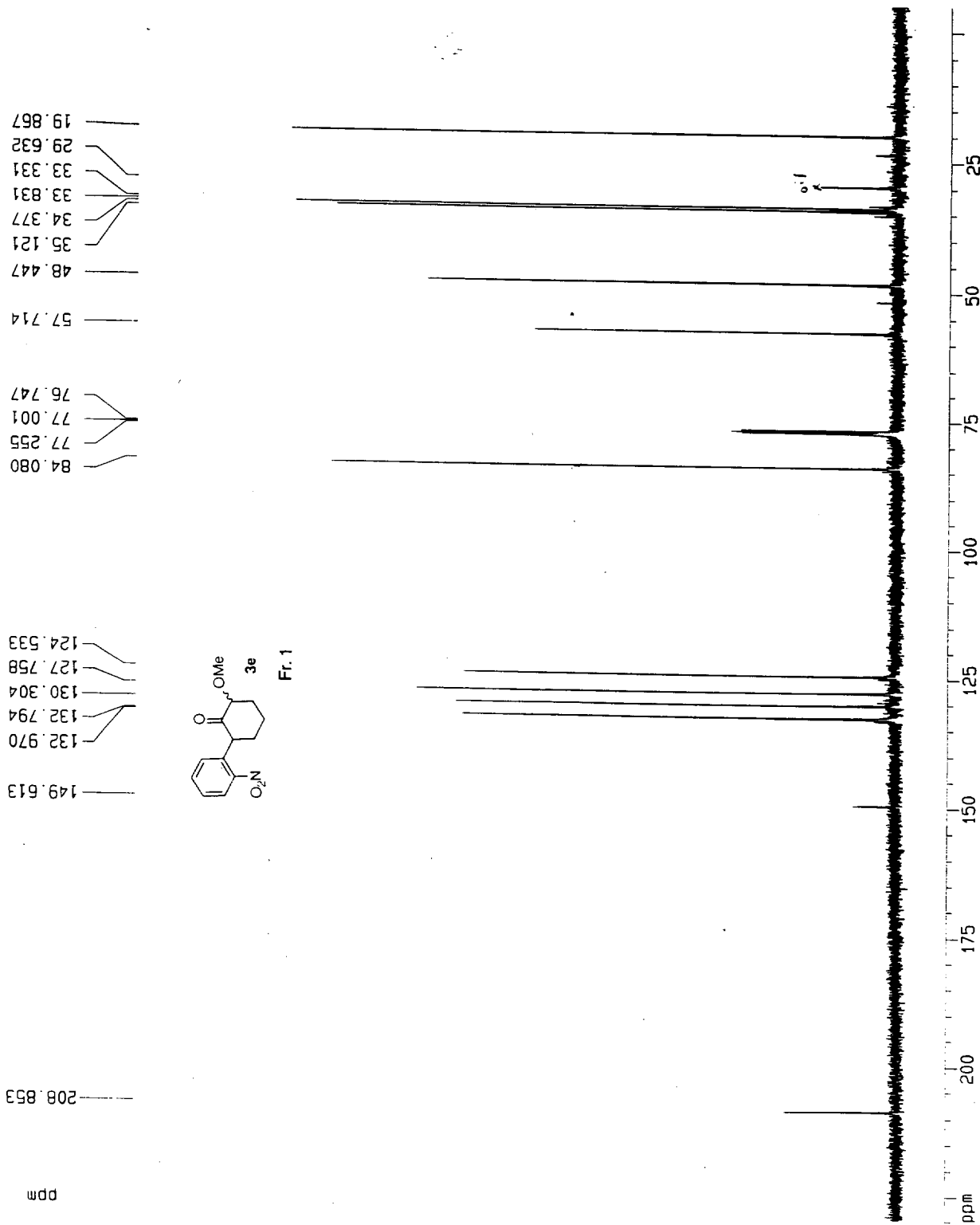
F2 - Acquisition Parameters
 Date_ 990609
 Time 16.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 32
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 120.00 dB
 CPDPRG2
 PCPD2 100.00 usec
 SF02 500.1300000 MHz
 NUC2 off
 PL2 120.00 dB
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

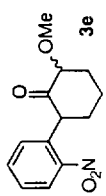
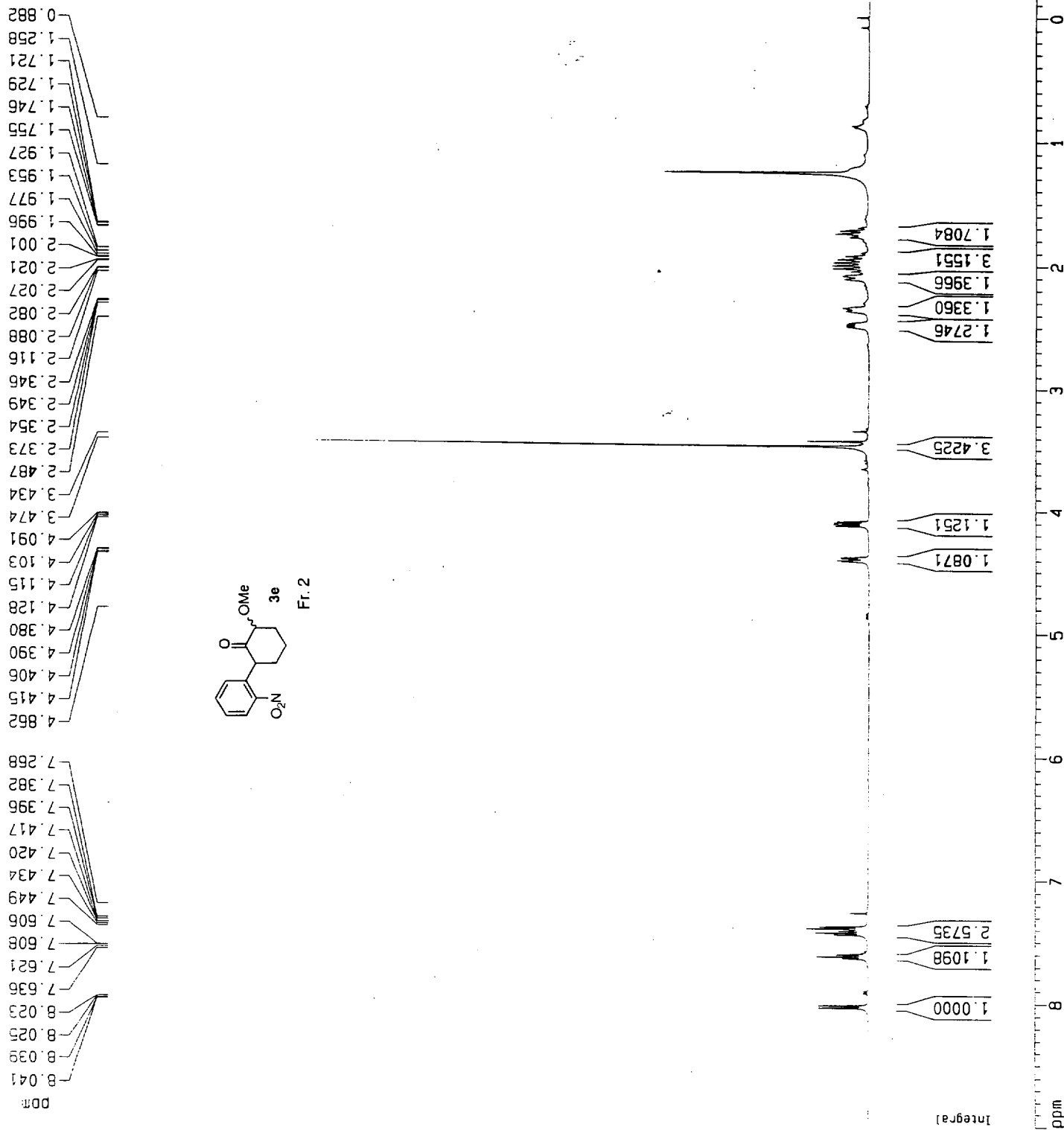
F2 - Processing parameters
 SI 16384
 SF 500.1300110 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm



Current Data Parameters
 NAME dEFAUL1
 EXPNO
 PROCNO
 F2 - Acquisition Param
 Date_ 990501
 Time 16.41
 INSTRUM spect
 PROBHD 5 mm GNP 11
 PULPROG zgpg
 TD 65531
 SOLVENT CDCl3
 NS 301
 DS 1
 SWH 39682.531
 FIDRES 0.605501
 AQ 0.8258031
 RG 2041
 DM 12.601
 DE 7.51
 TE 300.1
 d11 0.0300001
 PL12 20.01
 CPDPRG2 waltz11
 PCPD2 100.01
 SF02 500.1320001
 NUC2 11
 PL2 120.01
 D1 2.0000001
 P1 5.01
 SF01 125.7736211
 NUC1 131
 PL1 0.01
 F2 - Processing param
 SI 32761
 SF 125.7577991
 WDW EI
 SSB 1
 LB 1.01
 GB 1
 PC 1.41
 1D NMR plot parameters
 CX 20.01
 F1P 230.001
 F1 28924.21
 F2P -5.001
 F2 -628.71
 PPMCH 11.75001
 HZCN 1477.65401





Fr. 2

Current Data Parameters
 NAME defAult
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990609
 Time 16.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.145779 sec
 RG 32
 DM 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D11 0.03000000 sec
 PL12 120.00 dB
 CPDPRG2
 PCPD2
 SF02 500.1300000 MHz
 NUC2 off
 PL2 120.00 dB
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300056 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

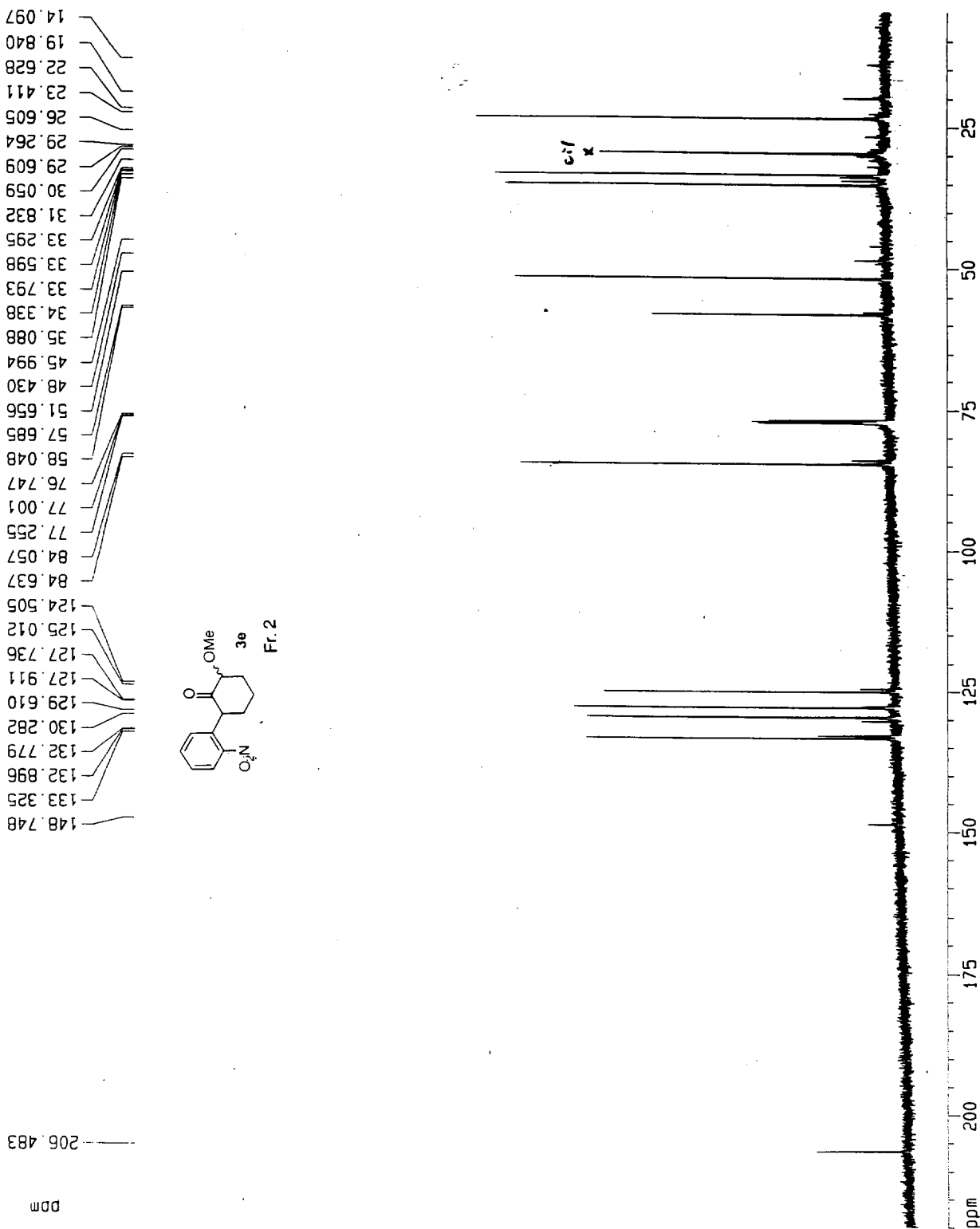
10 NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2 -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm

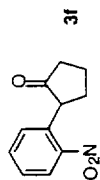
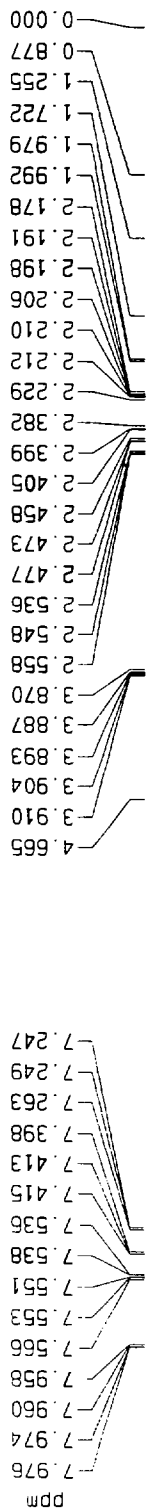
Current Data Parameters
 NAME dEFAULT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990609
 Time 17.11
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpgc
 TD 65536
 SOLVENT CDCl3
 NS 300
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8258036 sec
 RG 1024
 DW 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 O1 2.00000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 32768
 SF 125.7578019 MHz
 WOH EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 27666.72 Hz
 F2P 5.000 ppm
 F2 628.79 Hz
 PPHCM 10.75000 ppm/cm
 HZCM 1351.89636 Hz/cm



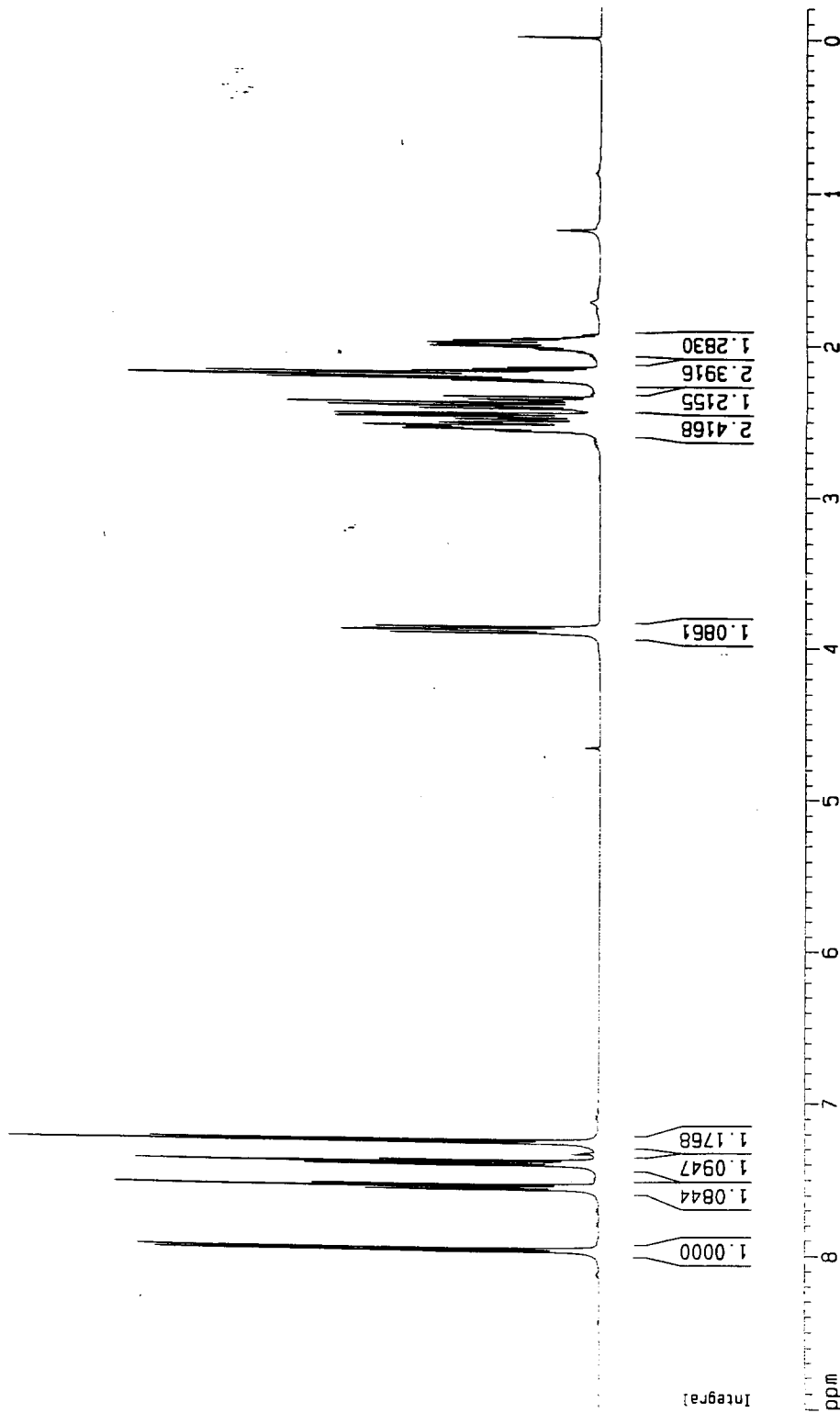


Current Data Parameters
 NAME DEFAULT
 EXPNO 1
 PROCNO 1

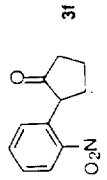
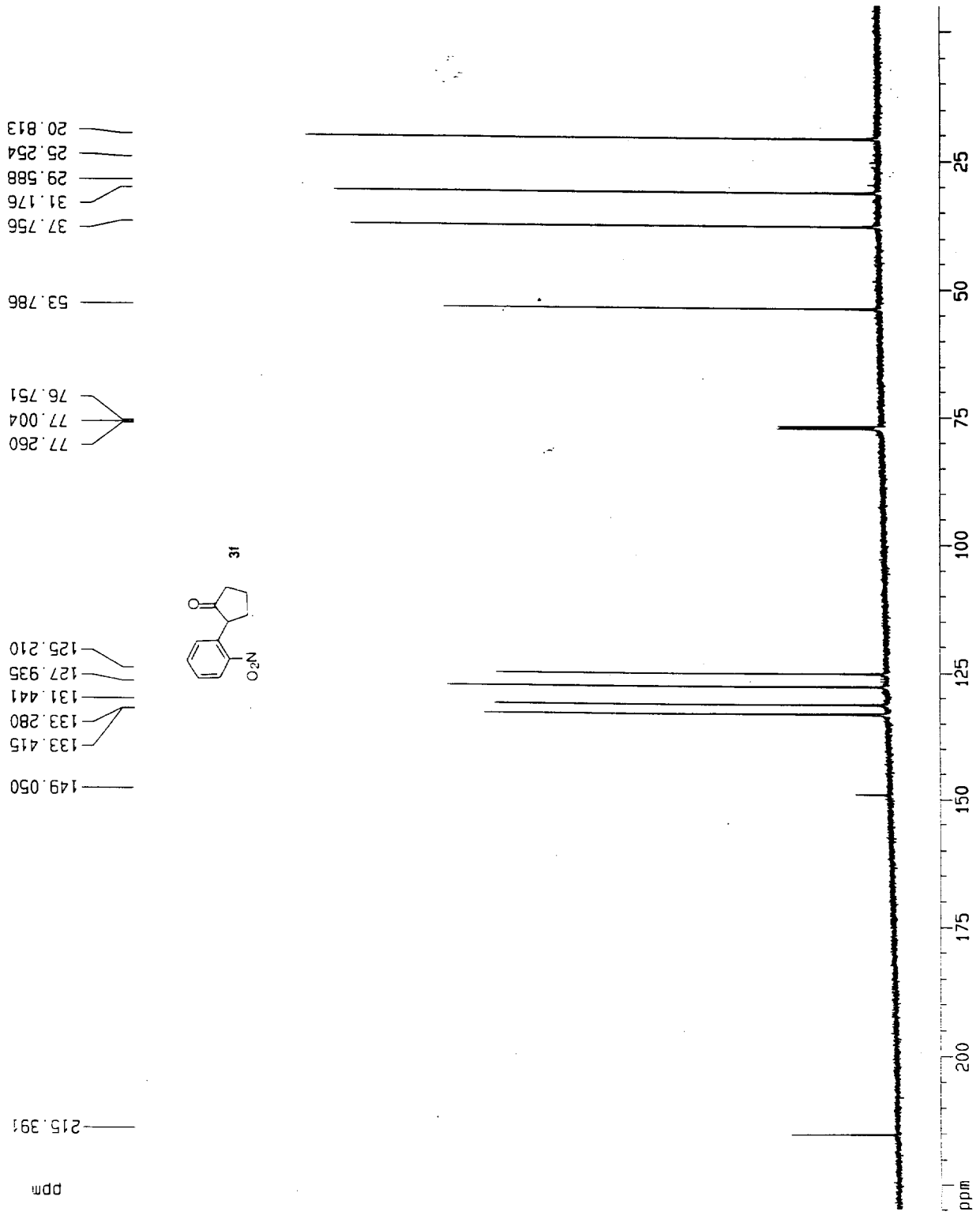
F2 - Acquisition Parameters
 Date_ 990608
 Time 20.40
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 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 OS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 32
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.03000000 sec
 PL12 120.00 dB
 CPDPRG2
 PCPD2 100.00 usec
 SF02 500.1300000 MHz
 NUC2 off
 PL2 120.00 dB
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

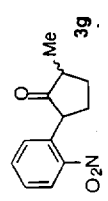
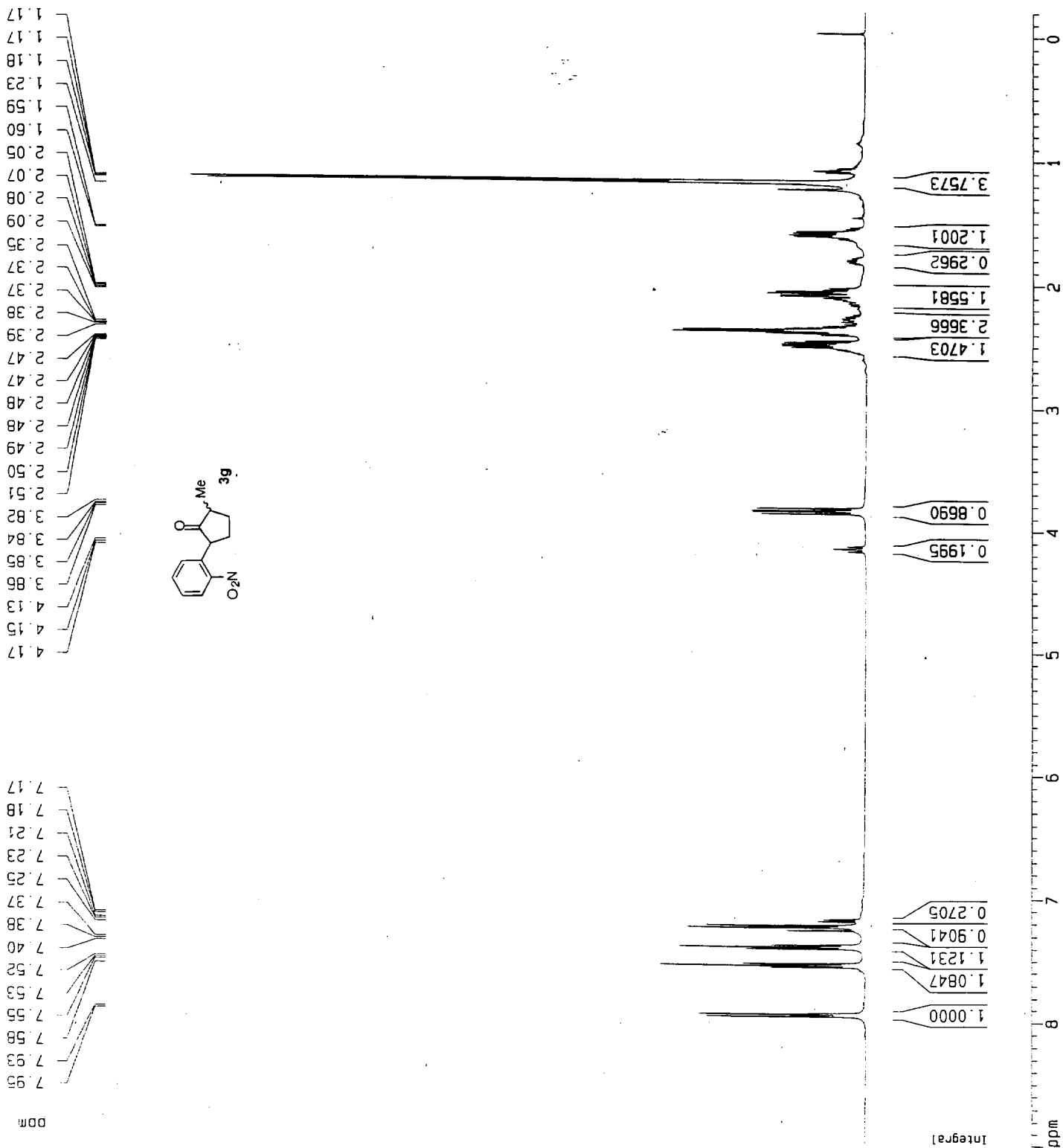
F2 - Processing parameters
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 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm



Current Data Parameter
 NAME dEFAULT
 EXPNO
 PROCNO
 F2 - Acquisition Param
 Date_ 99060
 Time 20.5
 INSTRUM spec
 PROBH0 5 mm QNP 1
 PULPROG zgpg
 TD 6553
 SOLVENT CDC1
 NS 30
 DS
 SMH 39682.53
 FIDRES 0.60550
 AQ 0.825803
 RG 102
 DM 12.60
 DE 7.5
 TE 300.1
 d11 0.0300001
 PL12 20.01
 CPDPRG2 waltz11
 PCPD2 100.01
 SF02 500.1320001
 NUC2 11
 PL2 120.01
 D1 2.00000001
 P1 5.01
 SF01 125.773621
 NUC1 13C
 PL1 0.01
 F2 - Processing param
 SI 32766
 SF 125.757805
 WDW EY
 SSB ()
 LB 1.01
 GB ()
 PC 1.41
 1D NMR plot parameters
 CX 20.01
 F1P 230.001
 F1 28924.21
 F2P -5.001
 F2 -628.71
 PPMCH 11.75001
 HZCM 1477.65401





Current Data Parameters
 NAME DEFAULT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990608
 Time 18.19
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl₃
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 32
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300181 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm

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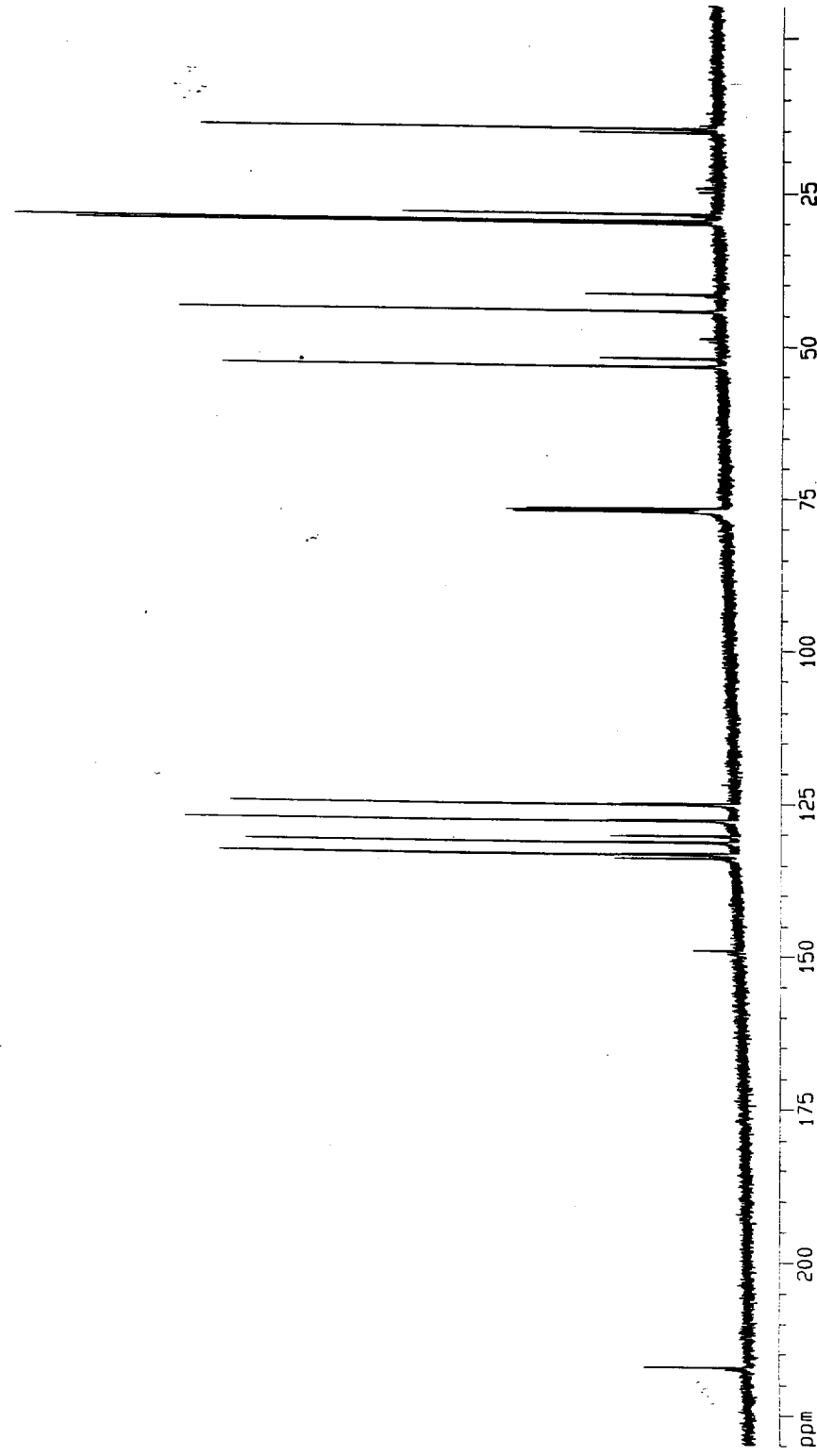
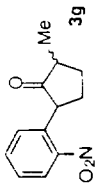
Current Data Parameter
NAME          dEFAU
EXPND
PROCNO

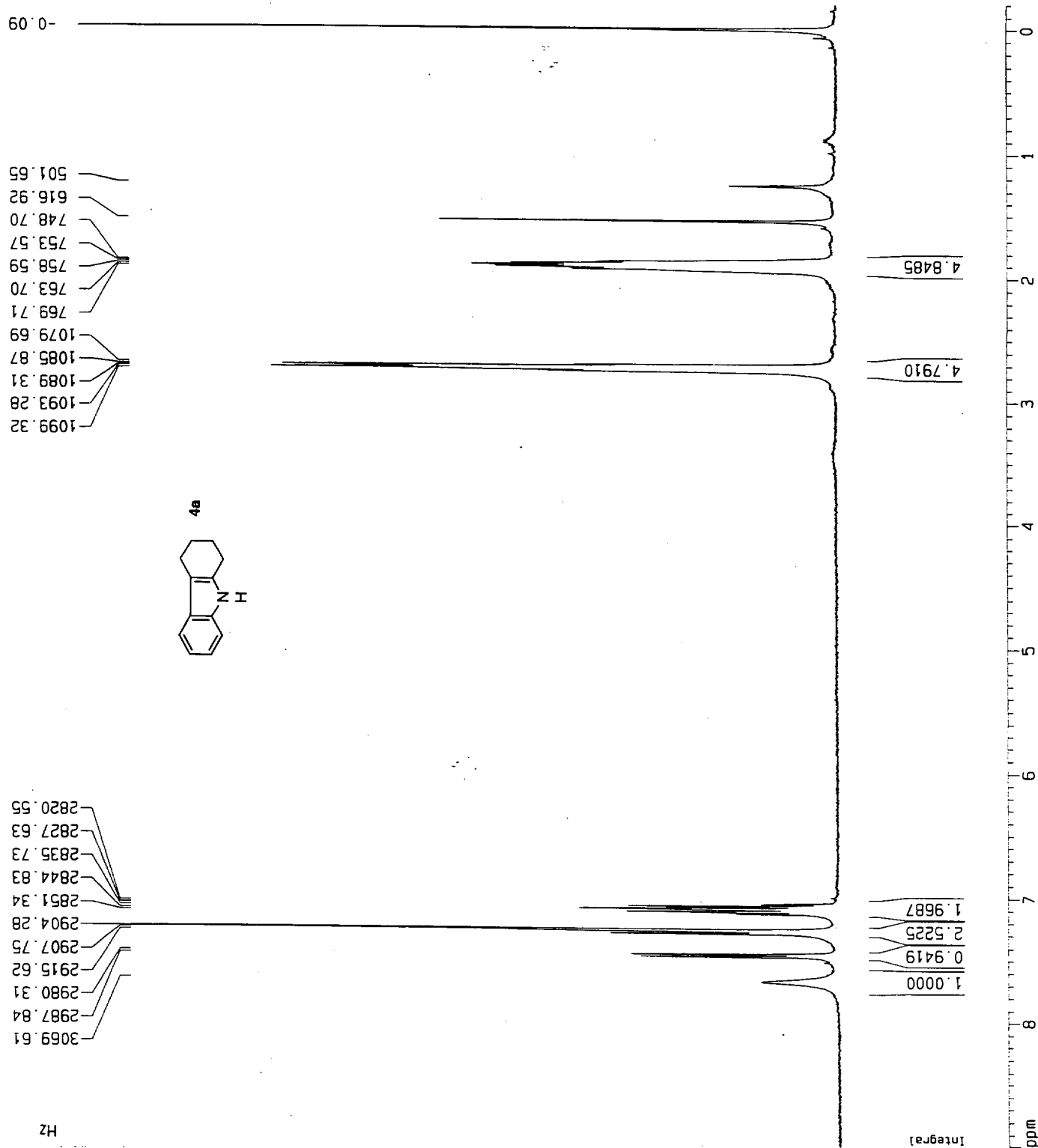
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Time_        18:
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PULPROG      zgpg
TD           655
SOLVENT      CDC
NS           5
DS
SMH          39682.5
FIDRES      0.6055
AQ          0.82580
RG          10
DM          12.6
DE          7.5
TE          300
d11         0.0300000
PL12        20.0
PCPDPRG2    waltz16
PCPD2       100.0
SF02        500.132000
NUC2         1
PL2         120.0
P1          2.0000000
P1          5.0
SF01        125.773621
NUC1         13
PL1         0.0

F2 - Processing parameters
SI          3276
SF          125.757801
WDW         EM
SSB         0
LB          1.0
GB          0
PC          1.41

1D NMR plot parameters
CX          20.01
F1P         230.001
F1          28924.2100
F2P         -5.001
F2          -626.71
PPMCH       11.75001
HZCM        1477.65401
    
```

14.735
15.042
15.343
24.277
28.555
28.890
29.653
30.084
41.593
44.347
52.024
53.330
76.749
77.002
77.257
125.038
125.151
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127.877
130.389
131.343
133.136
133.269
133.856
217.184



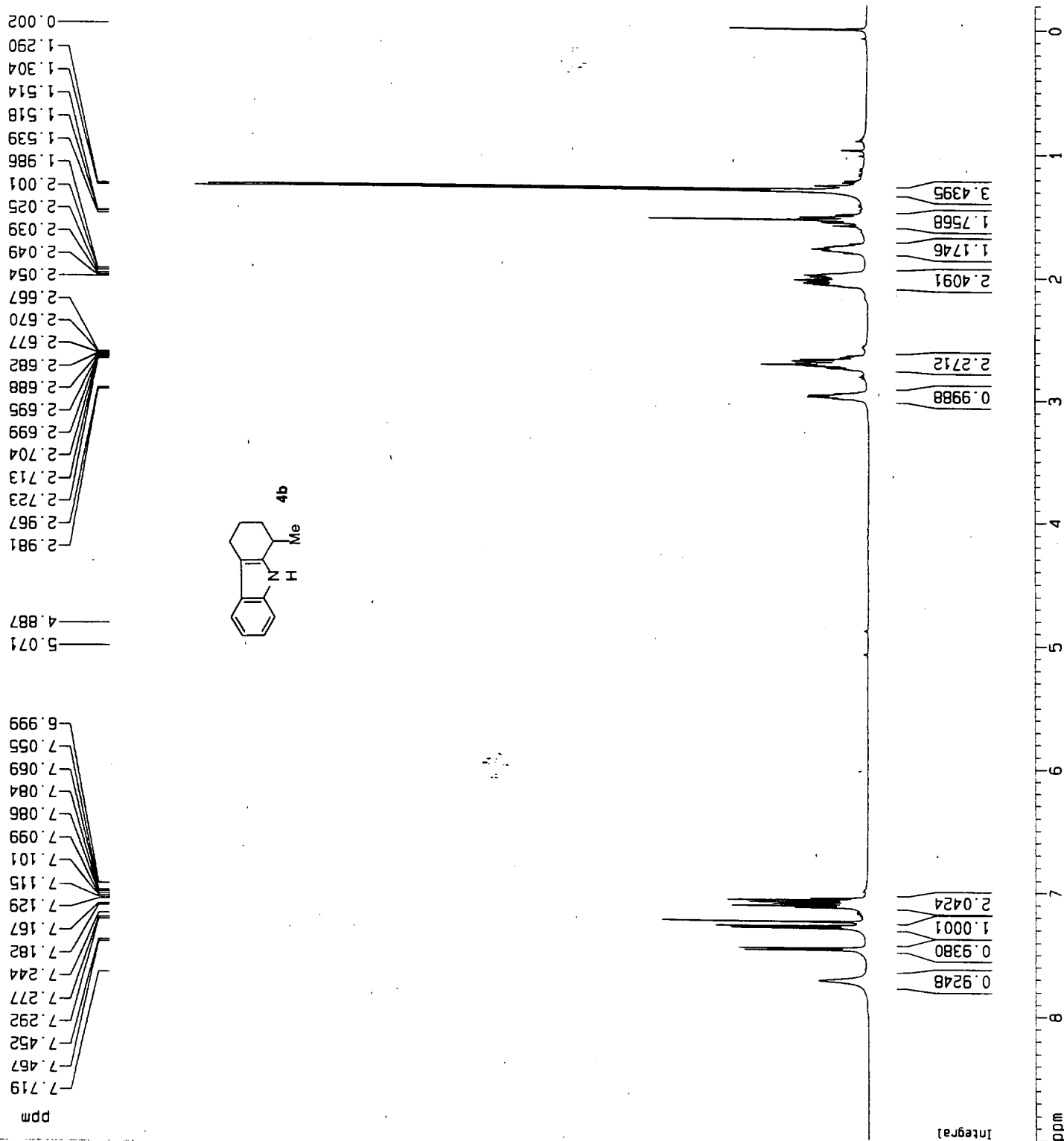


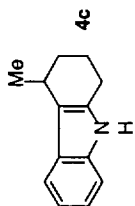
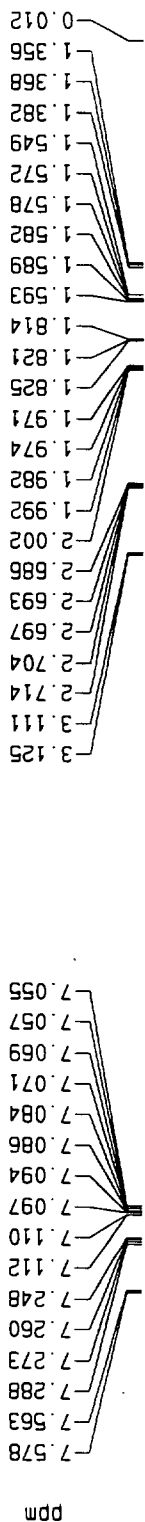
Current Data Parameters
 NAME default
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 PROCNO 1

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 PULPROG zg
 TD 32768
 SOLVENT COC13
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 128
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300215 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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 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05981 Hz/cm



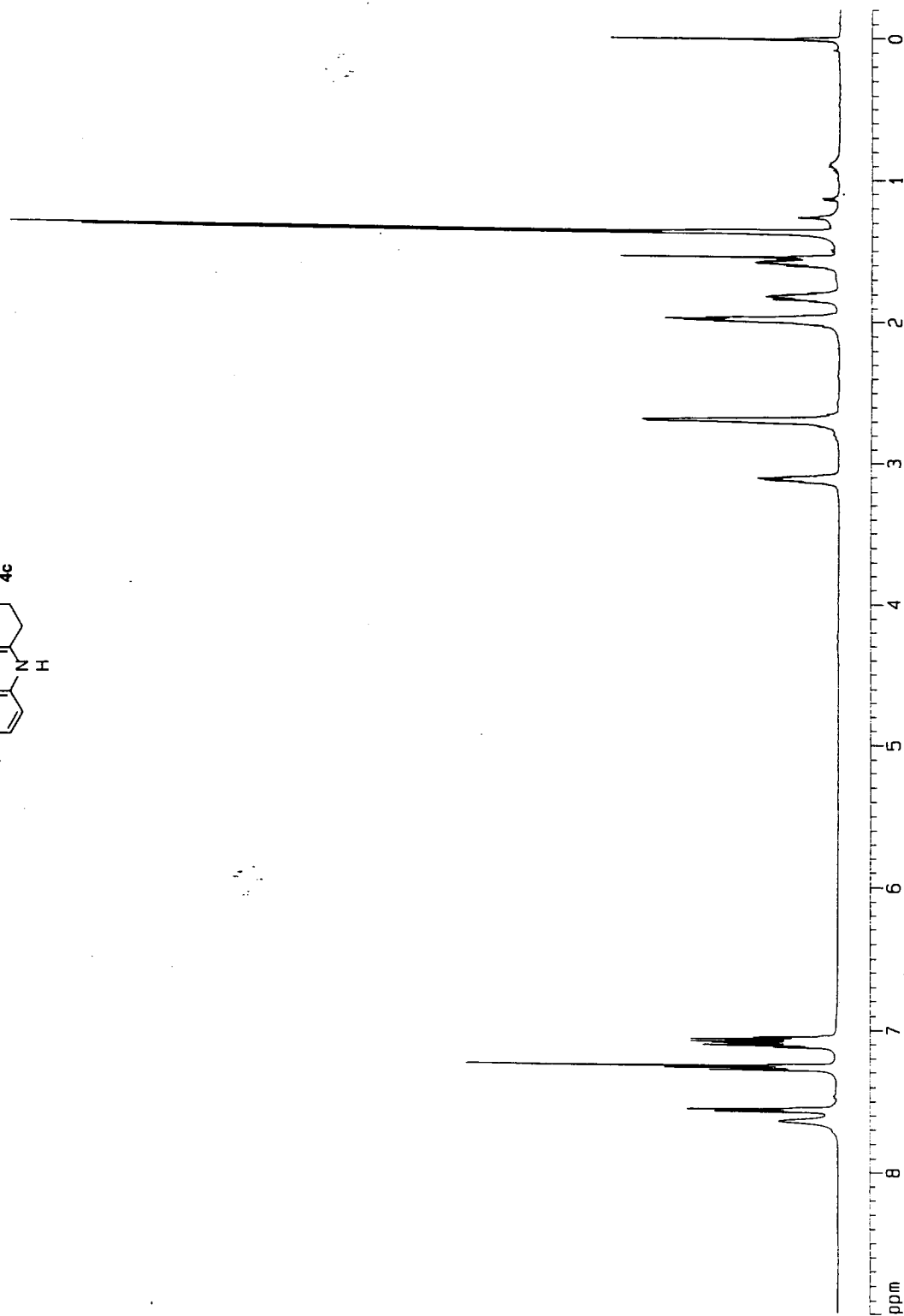


Current Data Parameters
 NAME dEfaUlt
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.145779 sec
 RG 128
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300135 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm

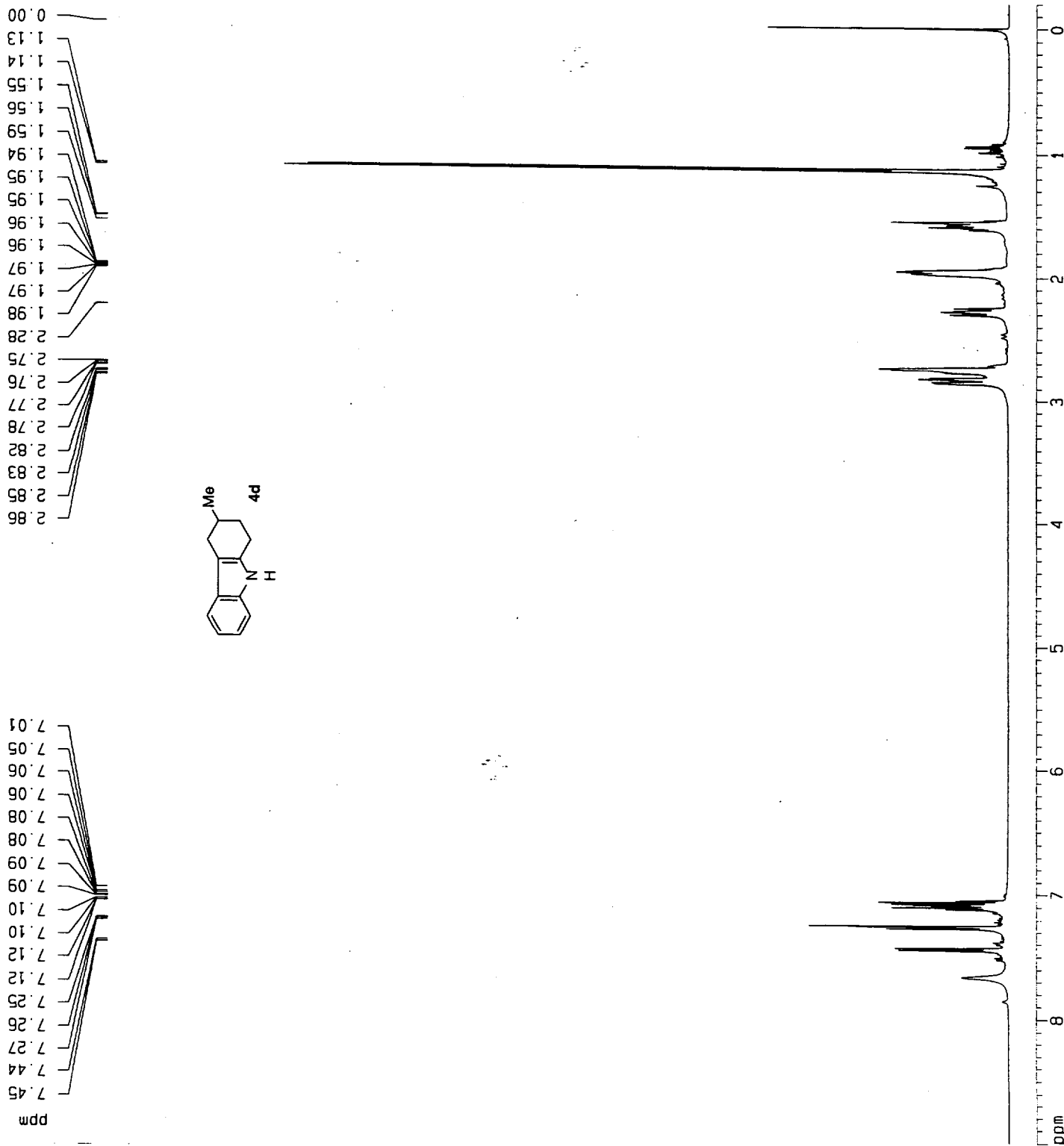


Current Data Parameters
 NAME default
 EXPND 1
 PROCND 1

F2 - Acquisition Parameters
 Date_ 990605
 Time 18.11
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 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 128
 DM 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300183 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05980 Hz/cm



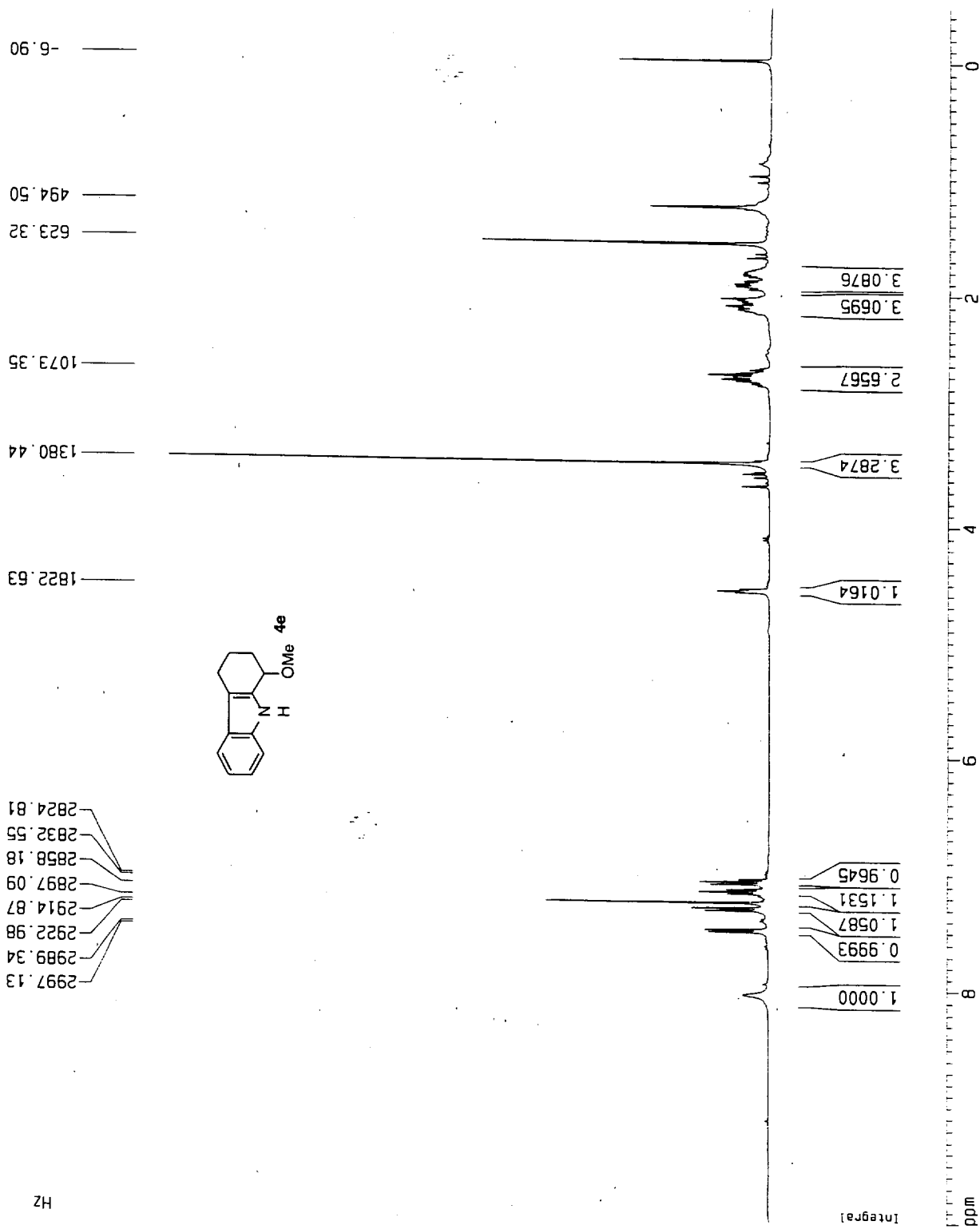
Current Data Parameters
 NAME default
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990605
 Time 13.25
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 161.3
 DW 119.200 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 8.50 usec
 PL1 0.00 dB
 SF01 400.1319246 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300177 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.007 ppm
 F1 4004.18 Hz
 F2P -0.476 ppm
 F2 -190.45 Hz
 PPMCM 0.52416 ppm/cm
 HZCM 209.73154 Hz/cm



HZ

Integral

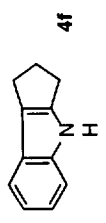
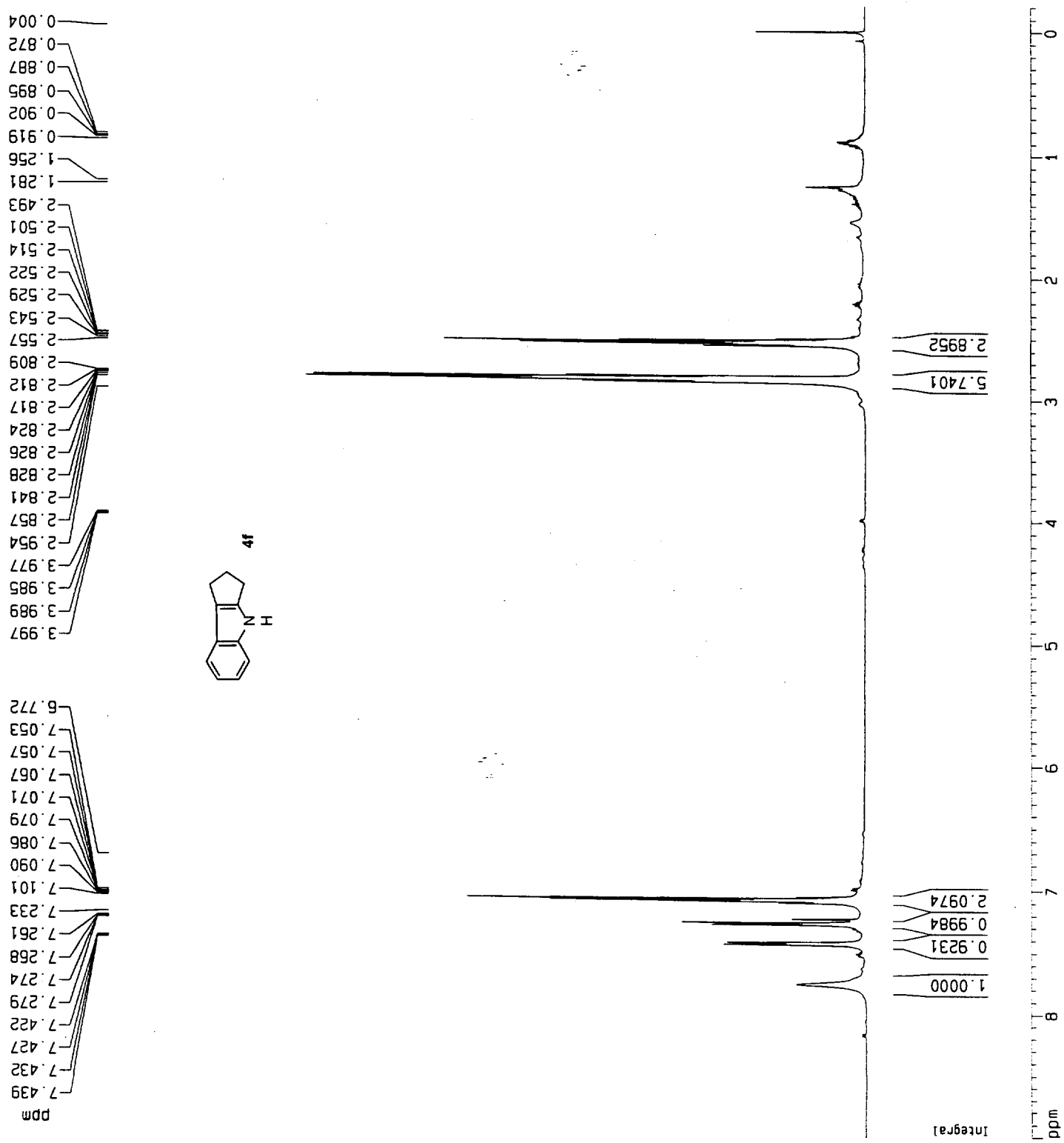
ppm

Current Data Parameters
 NAME default
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990609
 Time_ 17.21
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 128
 DM 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 5.00 usec
 SF01 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300269 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05581 Hz/cm



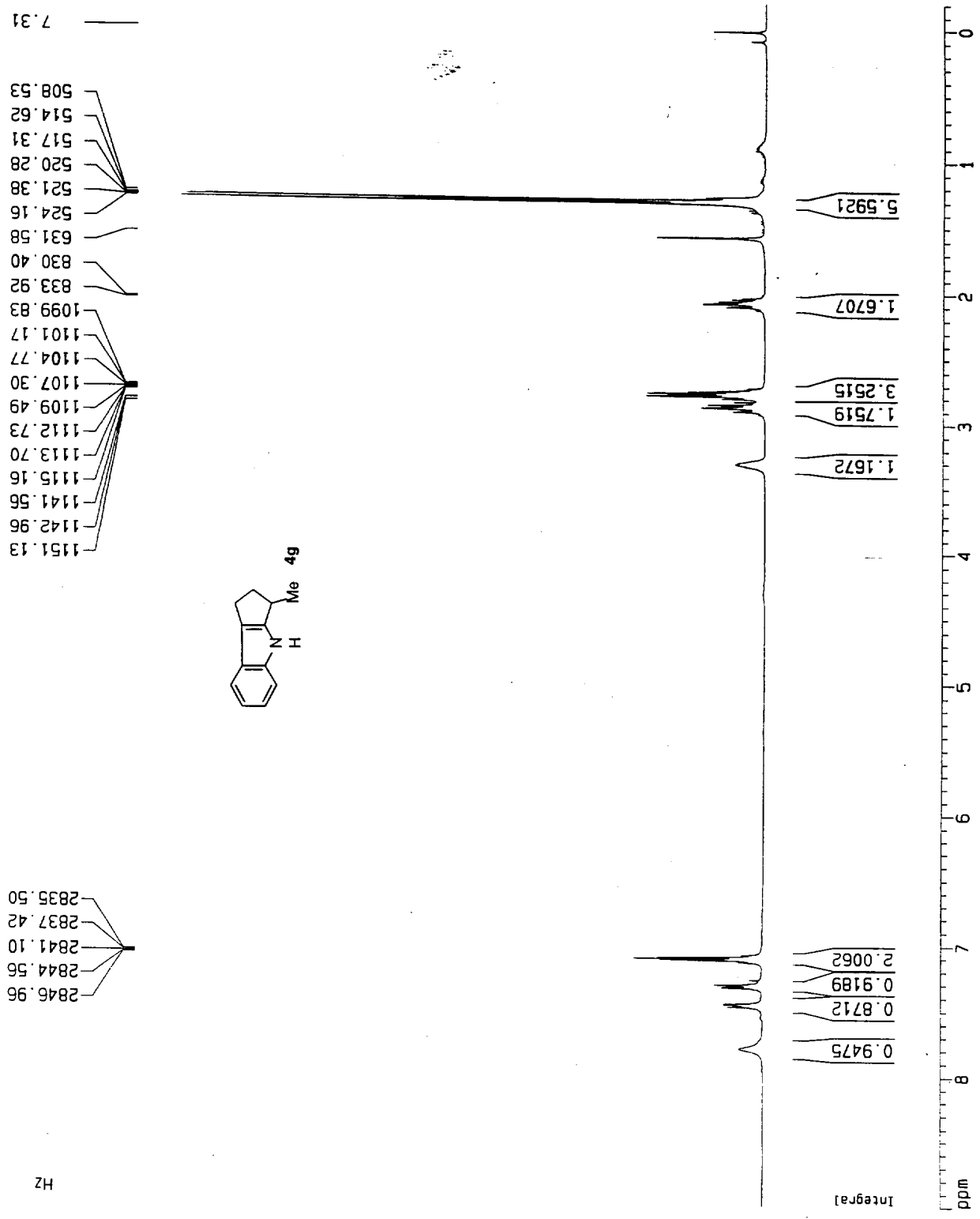
Current Data Parameters
 NAME default
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990603
 Time 17.34
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 101.6
 DW 119.200 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec

CHANNEL f1 -----
 NUC1 1H
 P1 8.50 usec
 PL1 0.00 dB
 SF01 400.1319246 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300072 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 3601.17 Hz
 F2P -0.200 ppm
 F2 -80.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 184.05980 Hz/cm



HZ

Integral

2.705
2.696
2.678
2.661
2.032
2.020
1.629
1.547
1.348
1.341
1.251
1.158
1.122
1.106
0.952
0.926
0.913
0.879
0.874
0.865
0.861
0.826
0.694

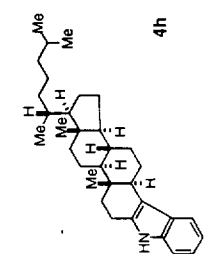
7.703
7.687
7.673
7.415
7.253
7.237
7.059
7.044
7.030
7.005
6.989
6.975

Current Data Parameters
 NAME dEFAULT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990612
 Time 17.30
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5208.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.145779 sec
 RG 128
 DW 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 120.00 dB

Processing parameters
 CPOPRG2
 PCPD2 100.00 usec
 SFDS 500.1300000 MHz
 NUC2 off
 PL2 120.00 dB
 D1 1.0000000 sec
 P1 5.00 usec
 SFO1 500.1320118 MHz
 NUC1 1H
 PL1 0.00 dB

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.000 ppm
 F1 4501.17 Hz
 F2P -0.200 ppm
 F2 -100.03 Hz
 PPMCM 0.46000 ppm/cm
 HZCM 230.05981 Hz/cm



2.0805
1.0400
1.0501
3.8343
3.1907
1.8383
4.8808
2.0768
1.6025
3.5902
1.1712
1.5492

0.9797
0.8763
1.0670

Integral



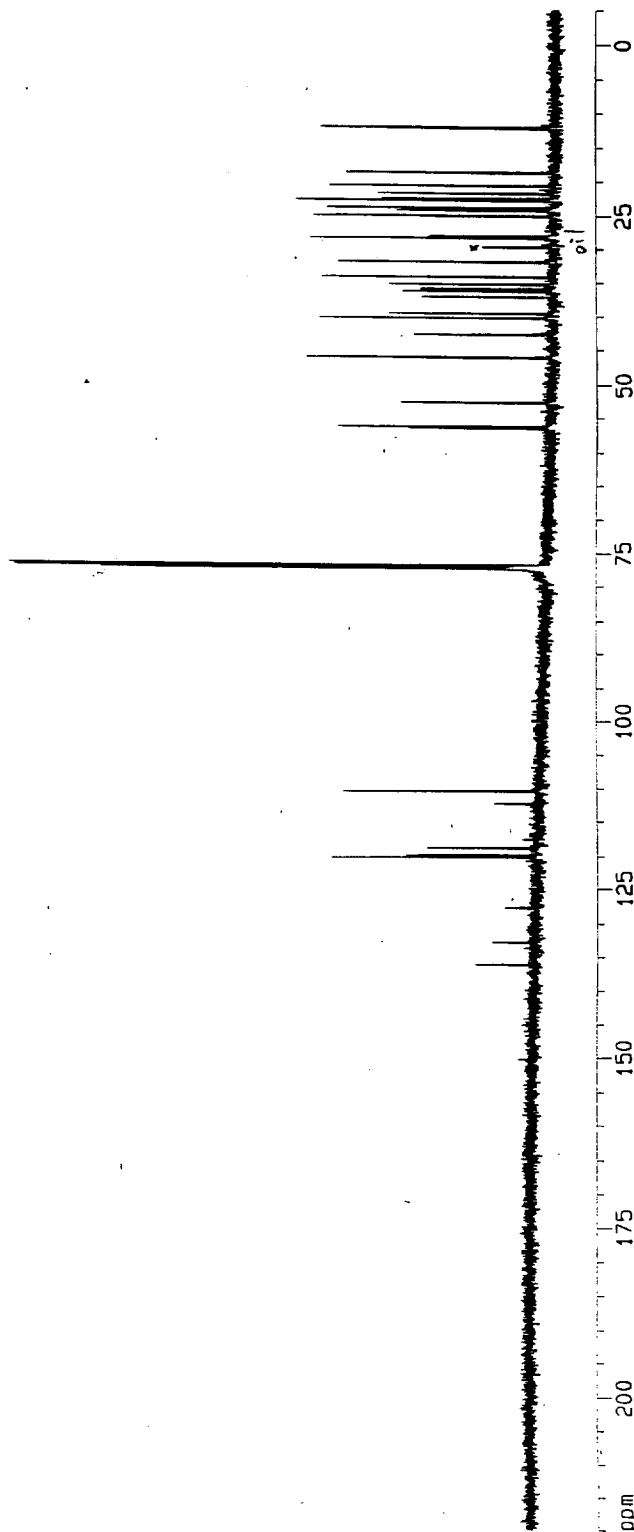
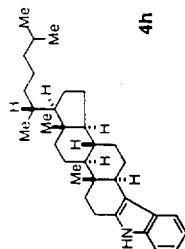
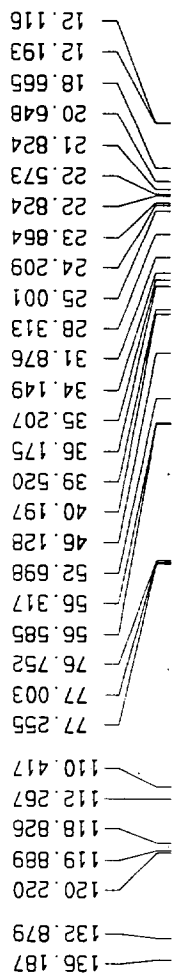
DDF

Current Data Parameters
 NAME dEFAUL1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 990612
 Time 19.08
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg
 TO 65536
 SOLVENT CDCl3
 NS 2024
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.605507 Hz
 AQ 0.8256036 sec
 RG 2048
 DH 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 PL12 20.00 dB
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 500.1320005 MHz
 NUC2 1H
 PL2 120.00 dB
 D1 2.0000000 sec
 P1 5.00 usec
 SF01 125.7736214 MHz
 NUC1 13C
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 125.7577934 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 27666.71 Hz
 F2P -5.000 ppm
 F2 -628.79 Hz
 PPMCM 11.25000 ppm/cm
 HZCM 1414.77515 Hz/cm.



DDM