

Enantioselective Fluorination of *t*-Butoxycarbonyl Lactones and Lactams Catalyzed by Chiral Pd(II)- Bisphosphine Complexes

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Supporting Information

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(A) General

Catalysts used in this paper were prepared according to the reported procedure.¹ NMR spectra were recorded at 400 MHz for ¹H-NMR, 100.4 MHz for ¹³C-NMR. Chemical shifts were reported downfield from TMS (= 0) for ¹H-NMR. For ¹³C-NMR, chemical shifts were reported in the scale relative to CDCl₃ as an internal reference. ¹⁹F NMR was measured at 376 MHz, and CF₃COOH (TFA) was used as an external standard. FAB-LRMS and FAB-HRMS were taken using *m*-nitrobenzyl alcohol (*m*NBA) as matrix. Flash column chromatography was performed with silica gel. The enantiomeric excesses (ees) were determined by chiral HPLC analysis. Solvents used in this paper were purchased and used directly. Other reagents were purified by usual methods.

(B) NMR data of the substrates examined in this paper

These substrates were prepared based on the reported procedure.²

***tert*-Butyl Tetrahydro-2-oxofuran-3-carboxylate (3a):** Colorless oil; ¹H-NMR (400 MHz, CDCl₃) δ 1.50 (s, 9H), 2.43-2.52 (m, 1H), 2.58-2.66 (m, 1H), 3.44 (dd, *J* = 9.3, 7.3 Hz, 1H), 4.31 (ddd, *J* = 6.9, 8.8, 7.6 Hz, 1H), 4.45 (ddd, *J* = 8.1, 8.8, 5.7 Hz, 1H); ¹³C-NMR (100 MHz, CDCl₃) δ 26.3, 27.7, 46.8, 67.1, 82.8, 166.8, 172.7

***tert*-Butyl Tetrahydro-2-oxo-2H-pyran-3-carboxylate (3b):** Colorless oil; ¹H-NMR (400 MHz, CDCl₃) δ 1.39 (s, 9H), 1.73-1.94 (m, 2H), 2.00-2.17 (m, 2H), 3.36 (t, *J* = 7.7 Hz, 1H), 4.25 (t, *J* = 5.9 Hz, 2H); ¹³C-NMR (100 MHz, CDCl₃) δ 20.8, 22.6, 27.9, 48.3, 69.1, 82.5, 167.7, 168.1.

¹ a) Fujii, A.; Hagiwara, E.; Sodeoka, M. *J. Am. Chem. Soc.* **1999**, *121*, 5450-5458. b) Hamashima, Y.; Yagi, K.; Takano, H.; Tamás, L.; Sodeoka, M. *J. Am. Chem. Soc.* **2002**, *124*, 14530-14531.

² a) Hua, D. H.; Miao, S. W.; Bhatathi, S. N.; Katsuhira, T.; Bravo, A. A. *J. Org. Chem.* **1990**, *55*, 3682-3684. b) Padwa, A.; Kissell, W. S.; Eidell, C. K. *Can. J. Chem.* **2001**, *79*, 1681-1693.

***tert*-Butyl 2-Oxopyrrolidine-3-carboxylate (6a):** White solid; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.35 (s, 9H), 2.15-2.36 (m, 2H), 3.11 (dd, $J = 6.6, 9.3$ Hz, 1H), 3.19-3.38 (m, 2H), 6.15 (brs, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 25.2, 28.0, 40.7, 48.6, 81.9, 169.3, 174.0.

***tert*-Butyl 1-Benzyl-2-oxopyrrolidine-3-carboxylate (6b):** White solid; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.48 (s, 9H), 2.15-2.24 (m, 1H), 2.25-2.34 (m, 1H), 3.20 (ddd, $J = 9.2, 8.3, 5.6$ Hz, 1H), 3.33-3.39 (m, 1H), 4.41 (d, $J = 14.8$ Hz, 1H), 4.51 (d, $J = 14.8$ Hz, 1H), 7.22-7.34 (m, 5H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 22.3, 27.9, 45.2, 46.8, 49.5, 81.8, 127.6, 128.0, 128.6, 136.0, 169.5, 170.3.

***tert*-Butyl 1-Methyl-2-oxopyrrolidine-3-carboxylate (6c):** White solid; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.42 (s, 9H), 2.11-2.21 (m, 1H), 2.23-2.32 (m, 1H), 2.81 (d, $J = 0.72$ Hz, 3H), 3.22-3.30 (m, 2H), 3.42 (td, $J = 9.1, 5.4$ Hz, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 22.3, 28.0, 29.9, 47.9, 49.2, 81.8, 169.7, 170.3.

Dibenzhydryl 2-Oxopyrrolidine-1,3-dicarboxylate (6d): White solid; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 2.11-2.20 (m, 1H), 2.26-2.35 (m, 1H), 3.59 (dd, $J = 9.1, 7.5$ Hz, 1H), 3.68 (ddd, $J = 6.7, 8.0, 10.7$ Hz, 1H), 3.84 (ddd, $J = 5.5, 9.6, 10.6$ Hz, 1H), 6.82 (s, 1H), 6.83 (s, 1H), 7.18-7.31 (m, 16H), 7.36 (d, $J = 7.6$ Hz, 4H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 21.5, 44.7, 50.2, 78.5, 79.6, 126.8, 126.9, 127.0, 127.2, 127.9, 128.0, 128.1, 128.1, 128.5, 128.6, 139.4, 139.5, 139.5, 150.6, 167.4, 168.1.

***tert*-butyl 1-Benzyl-2-oxopiperidine-3-carboxylate (6e):** White solid; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 1.46 (s, 9H), 1.65-1.72 (m, 1H), 1.82-1.88 (m, 1H), 1.98-2.12 (m, 2H), 3.11-3.24 (m, 2H), 3.34 (t, $J = 6.9$ Hz, 1H), 4.40 (d, $J = 14.6$ Hz, 1H), 4.75 (d, $J = 14.6$ Hz, 1H), 7.19-7.29 (m, 5H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 20.6, 25.1, 27.8, 30.7, 46.8, 49.9, 50.1, 81.3, 127.1, 127.7, 128.4, 166.1, 170.2.

(C) Conversion of 4a to 5a

To a solution of **4a** (35 mg, 0.17 mmol) in THF (0.2 mL) was added BnNH_2 (30 μL , 1.5 eq), and the mixture was stirred at ambient temperature for 12 h. 1N HCl was added, and aqueous layer was

extracted with ether (5 mL x 3). Further purification was carried out by flash column chromatography (hexane/ethyl acetate = 1/1) to afford **5a** in 74% yield.

(D) Conversion of **7a** to **7b** (*N*-benzylation)

To a stirred solution of **7a** (27 mg, 0.13 mmol) in THF (2 mL) was added NaH (60% in oil, 6 mg) under ice bath cooling. After 10 minutes, BnBr (30 μ L) was added and the resulting mixture was stirred at room temperature for 12 h. Saturated aqueous NH_4Cl was added for quenching. Aqueous layer was extracted with ether (5 mL x 3) and the combined organic layers were washed with brine. Evaporation and column chromatography (hexane/ethyl acetate = 3/1) gave the desired benzylated product **7b** in 77% yield.

(E) Conversion of **7b** to **8**

To a stirred solution of **7b** (44 mg, 0.15 mmol) in THF (1 mL) was added BH_3 in THF (0.74 mL, 1.0 M, 5 eq), and the solution was stirred equipped with water condenser under reflux condition for 24 h. After completion of the reduction, MeOH (0.1 mL) was added and the mixture was stirred at room temperature for 1 h. MeOH was removed under reduced pressure, and ether (3 mL) and 1N HCl (1 mL) were added. After stirring for 1 h, organic layer was separated and washed with water. The combined layers were treated with 10% NaOH and extracted with ether (3 mL x 3). The combined organic layers were dried over Na_2SO_4 . Removal of the solvent, followed by flash column chromatography (hexane/ethyl acetate = 1/1) afforded **8** as a white powder (18.7 mg, 60%).

(1-Benzyl-3-fluoropyrrolidin-3-yl)methanol (8): Colorless oil; ^1H -NMR (500 MHz, CDCl_3) δ 1.94-2.13 (m, 2H), 2.13 (brs, 1H), 2.59-2.64 (m, 1H), 2.73-2.82 (m, 3H), 3.61-3.75 (m, 4H), 7.23-7.33 (m, 5H); ^{13}C -NMR (100 MHz, CDCl_3) δ 34.1 (d, J = 23.1 Hz), 52.7, 60.1, 61.4 (d, J = 25.6 Hz), 67.0 (d, J = 27.2 Hz), 103.8 (d, J = 178.0 Hz), 127.0, 128.2, 128.7, 138.1; ^{19}F -NMR (470 Hz, CDCl_3) δ -75.0 — -

74.8 (m); FAB-LRMS (*m*NBA) m/z 210 ($M+1$)⁺; HPLC (DAICEL CHIRALPAK AS-H, *n*-hexane/IPA = 9/1, 1.0 mL/min., 254 nm) τ_{minor} 8.5 min, τ_{major} 10.2 min; IR (neat) ν 3353, 2935, 2912, 2803, 1495, 1454, 1381, 1297, 1264, 1204, 1152, 1114, 1051, 986, 900, 744, 699 cm⁻¹.

(F) NMR spectra

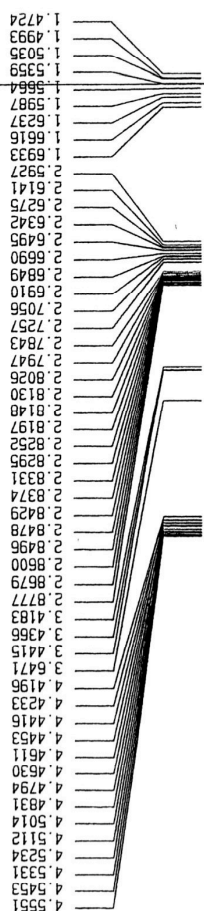
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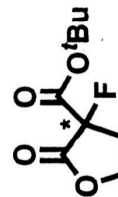
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¹H NMR



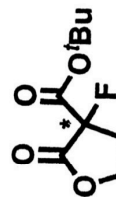
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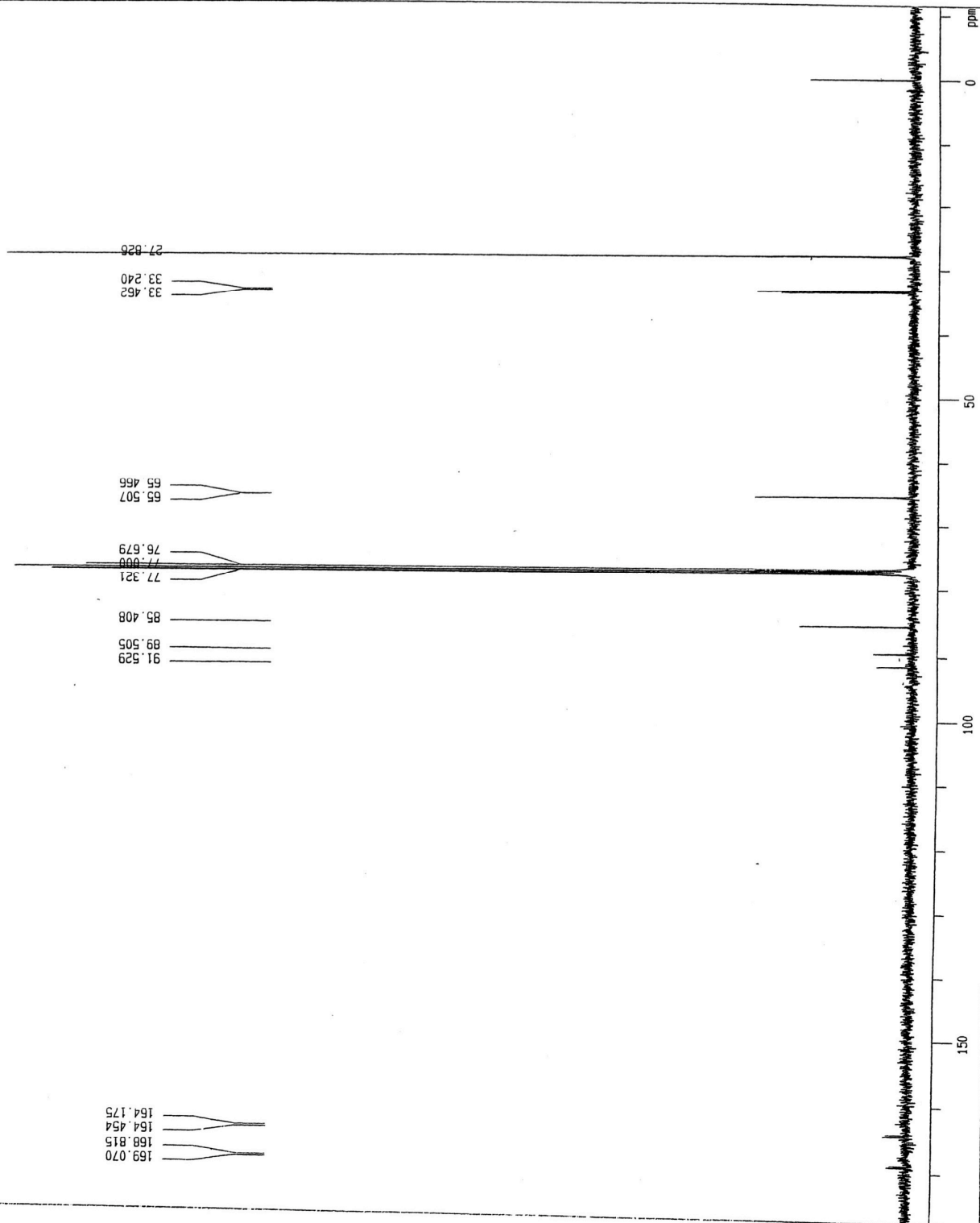
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¹³C NMR



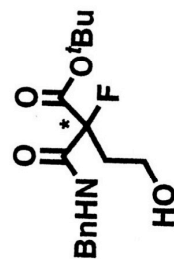
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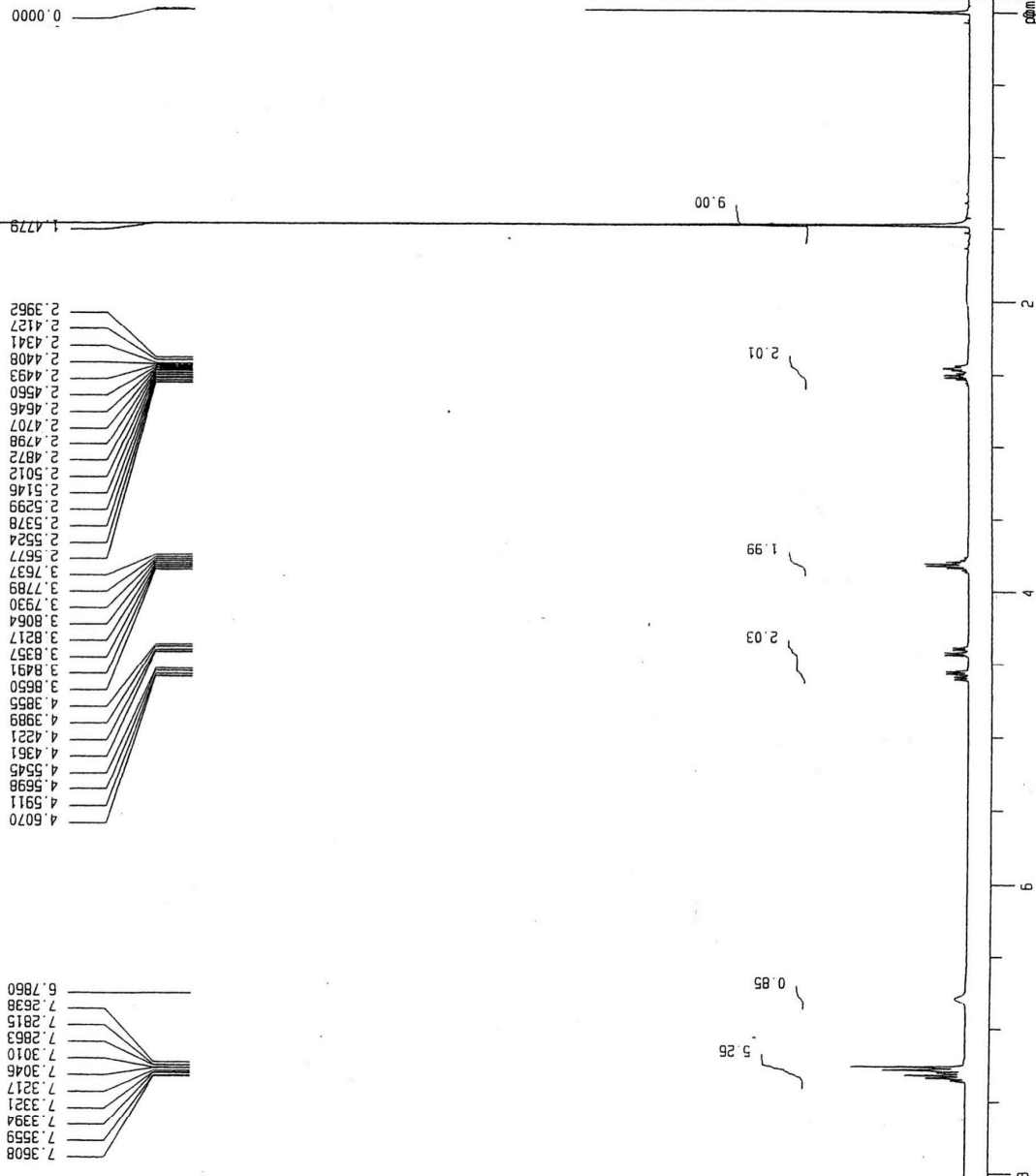
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¹H NMR



5a

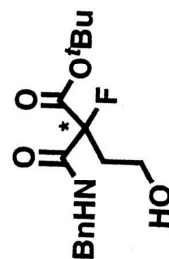


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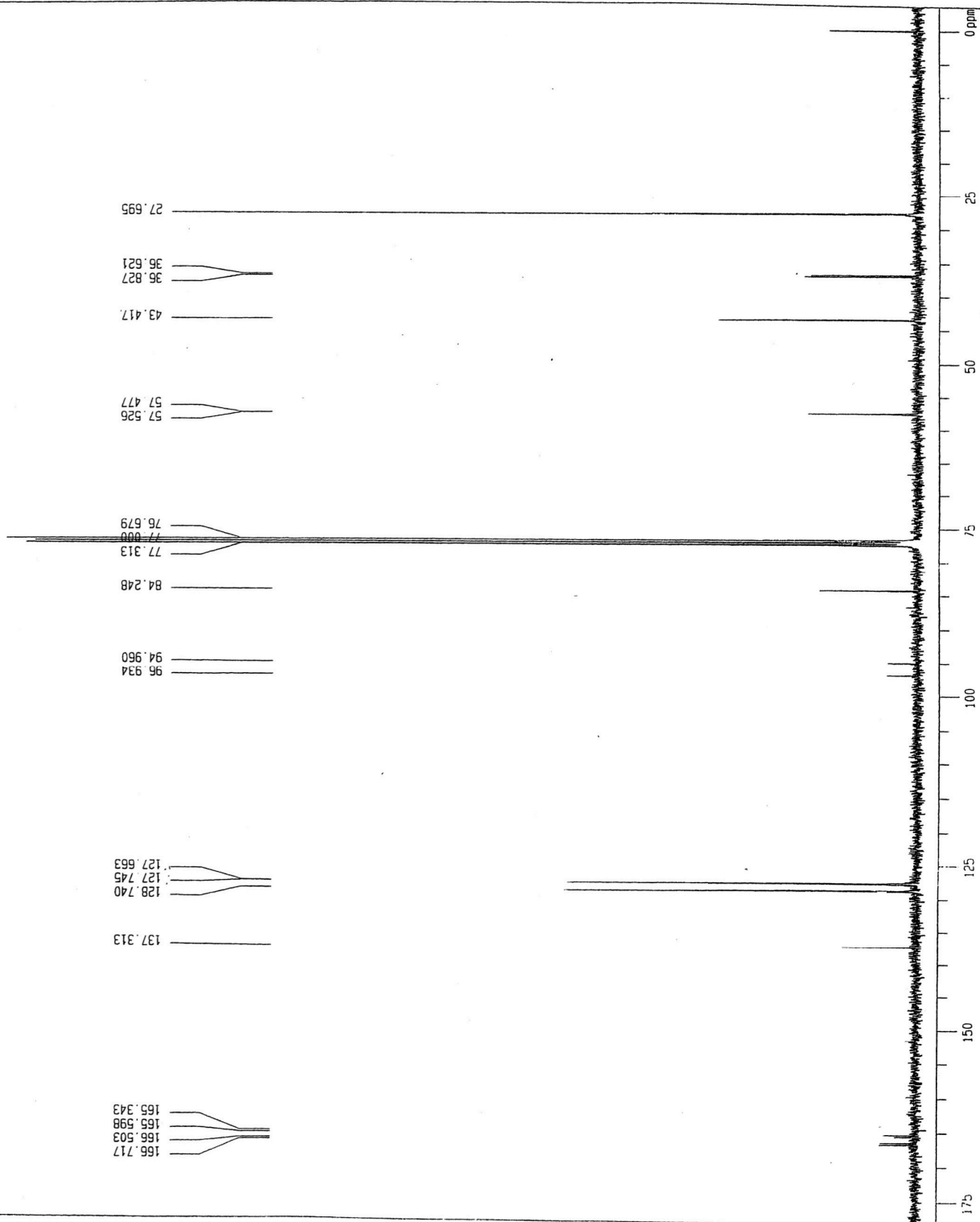
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¹³C NMR



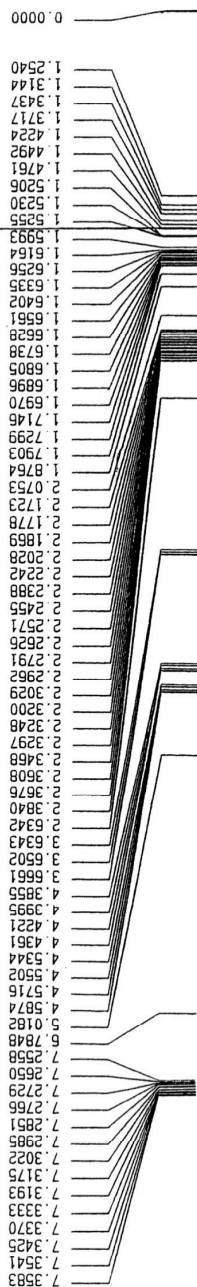
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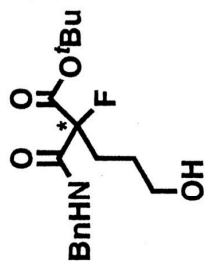
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¹H NMR



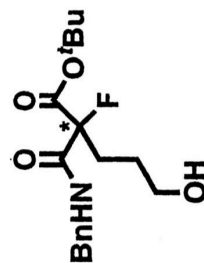
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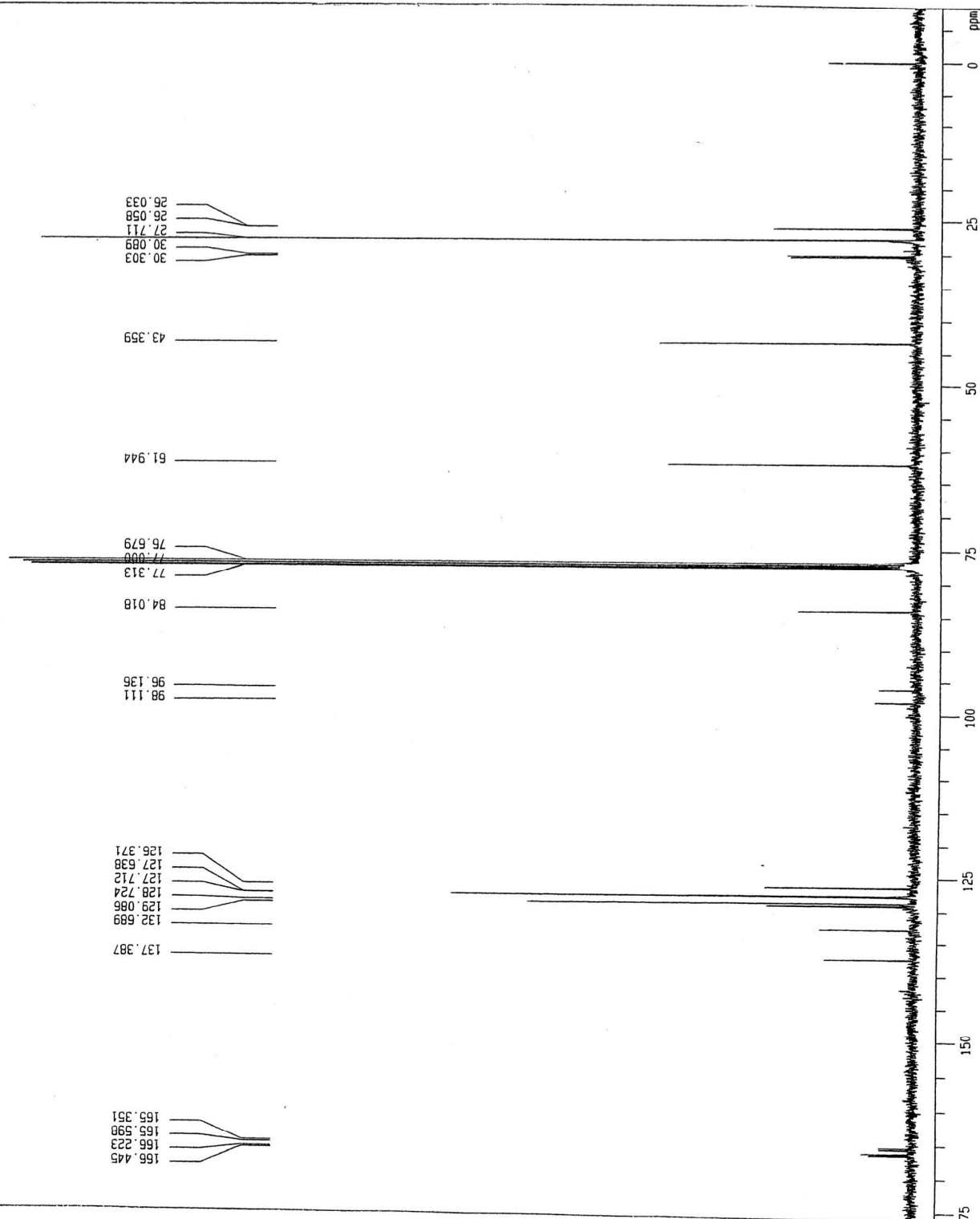
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¹³C NMR



5b

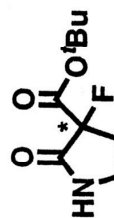


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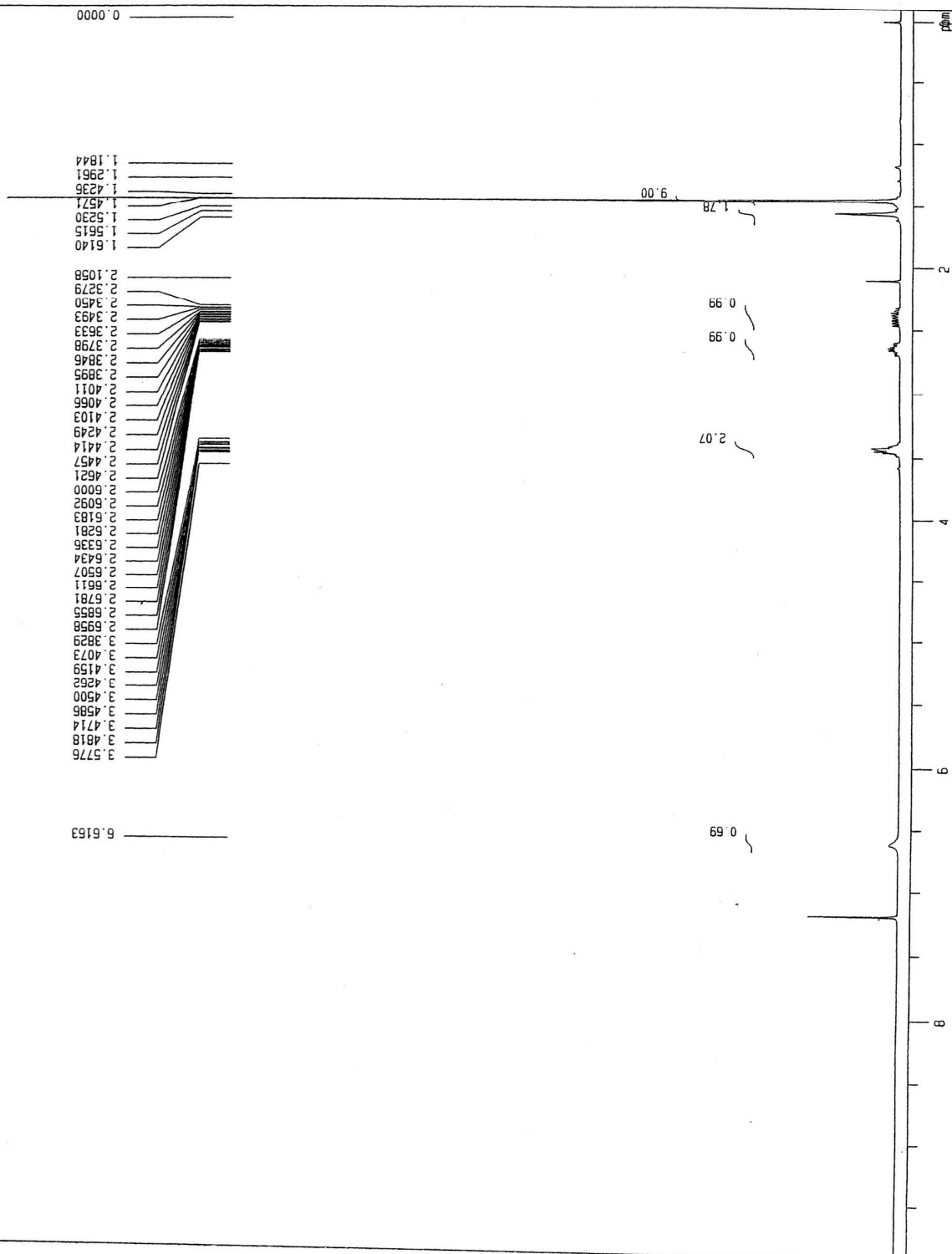
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¹H NMR



7a



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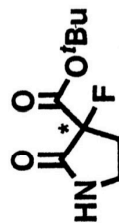
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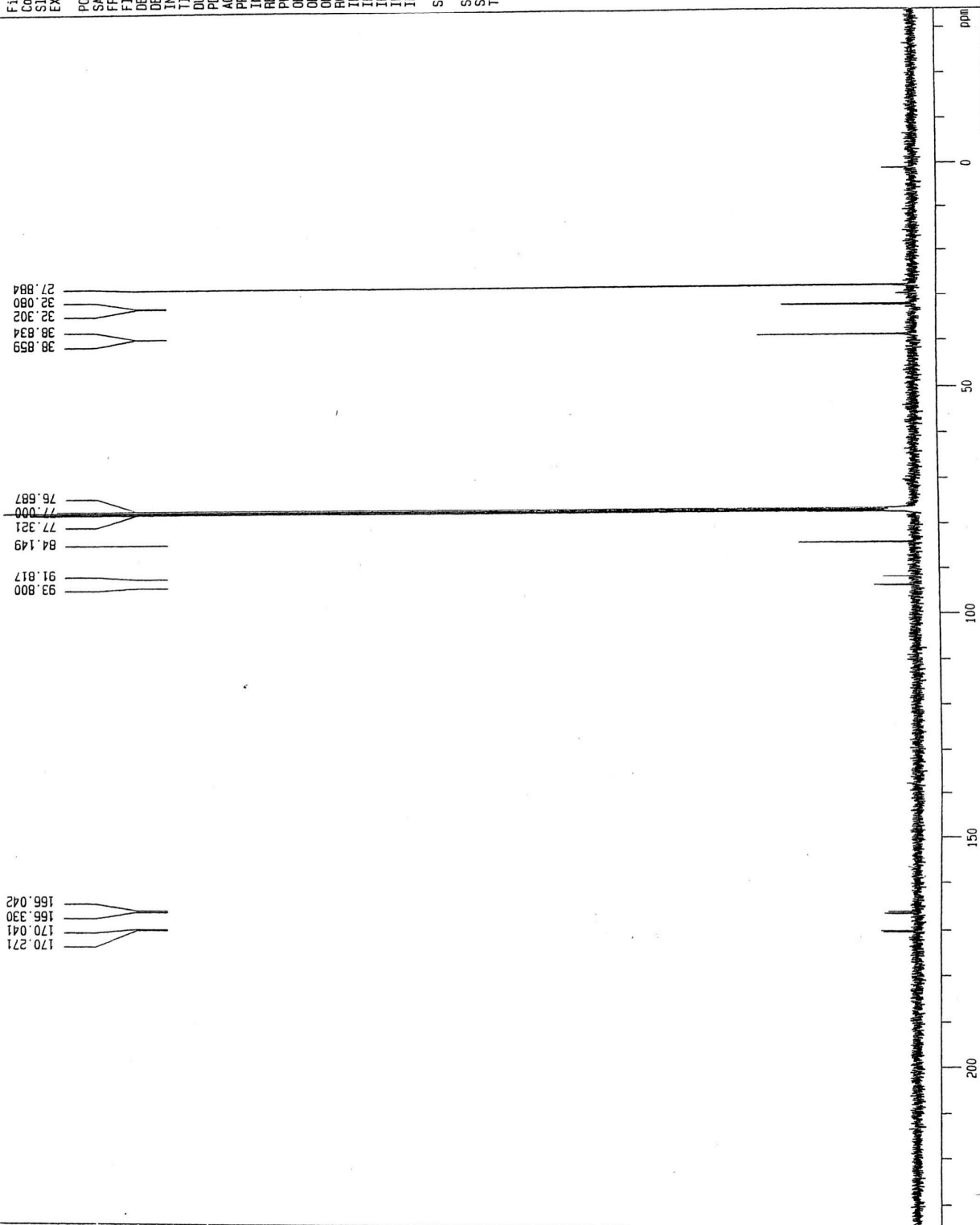
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¹³C NMR



7a

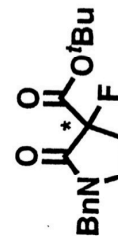


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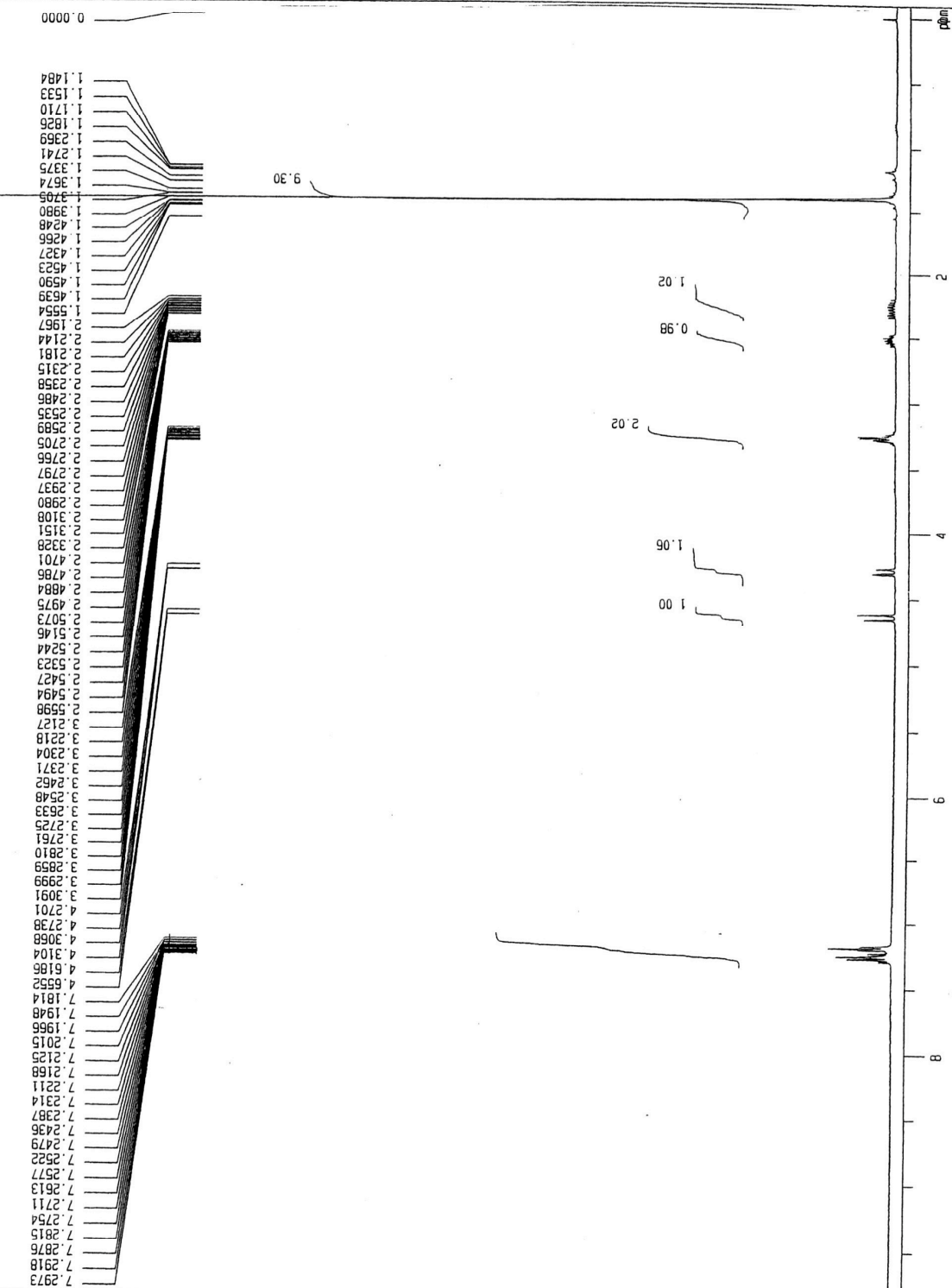
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¹H NMR



7b

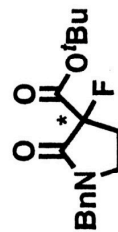


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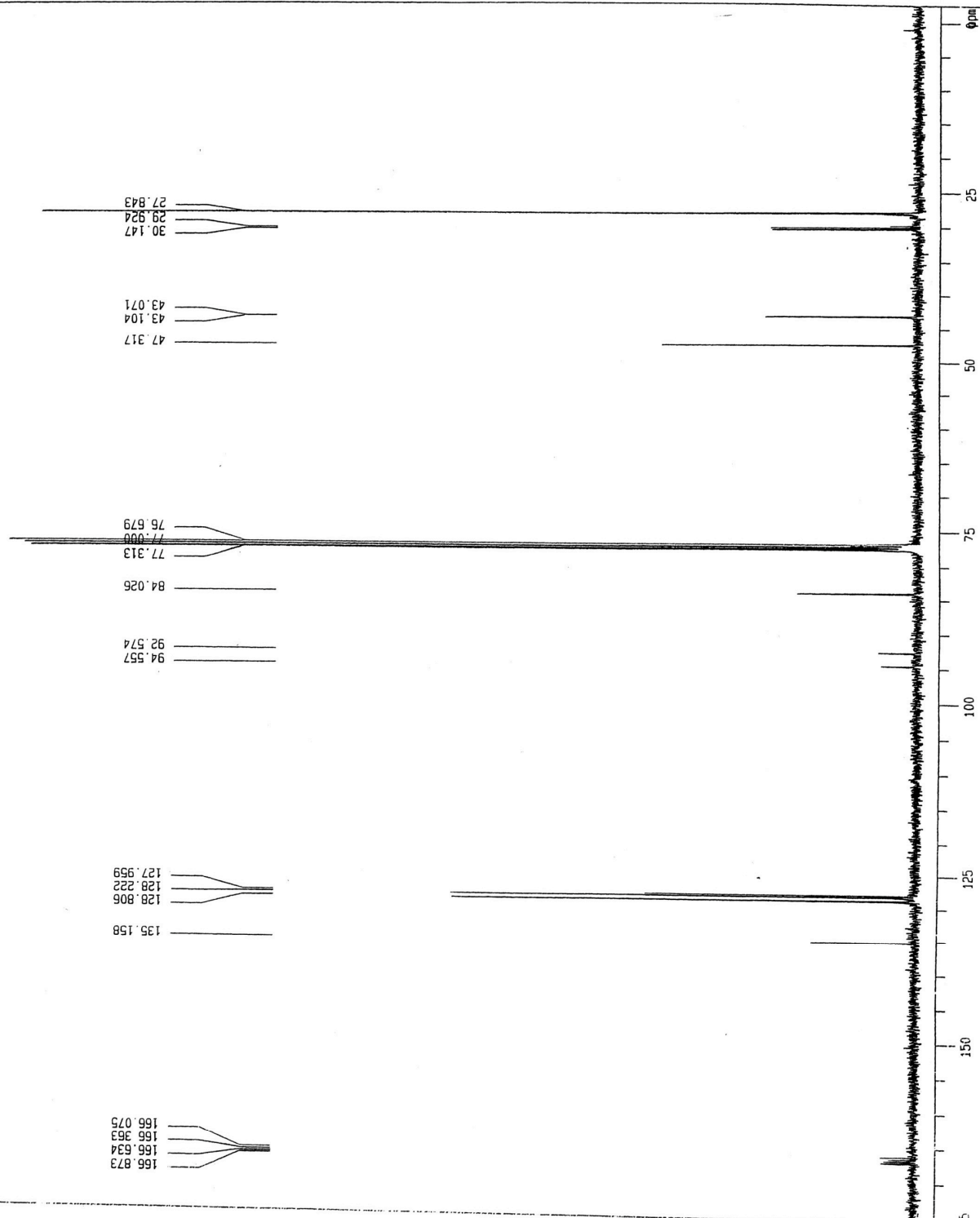
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¹³C NMR



7b



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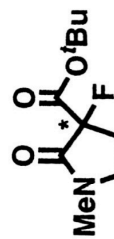
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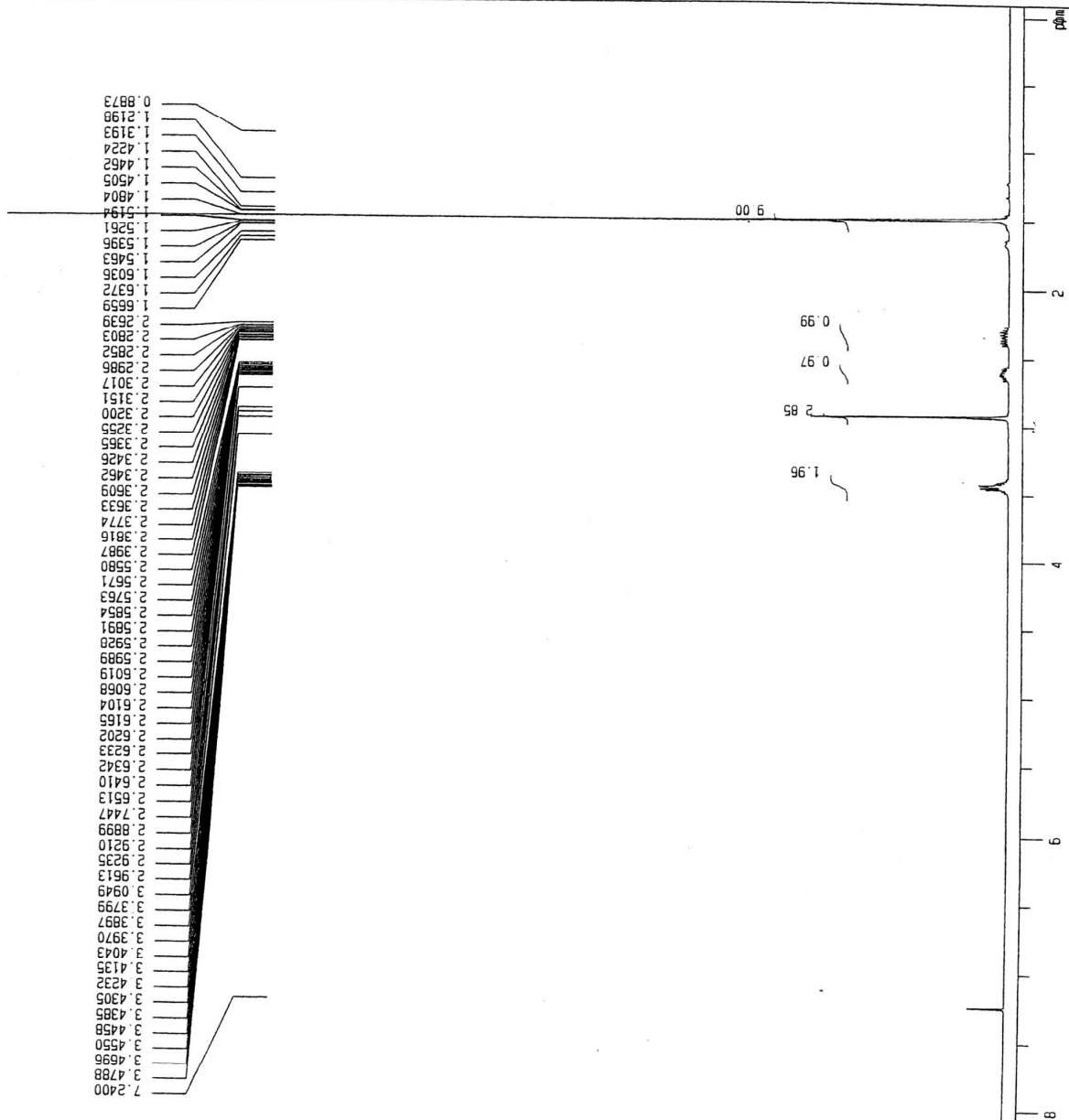
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¹H NMR



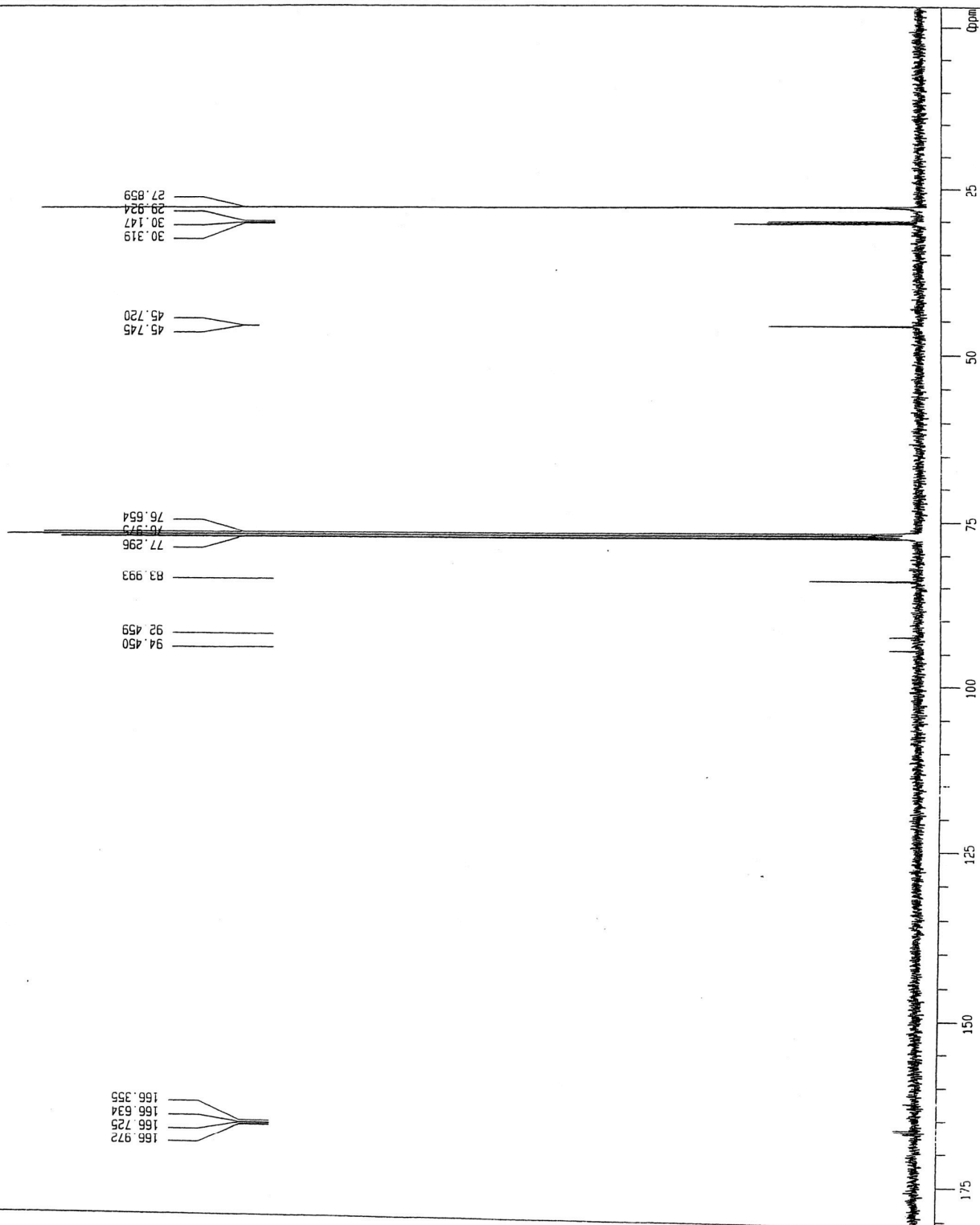
7c



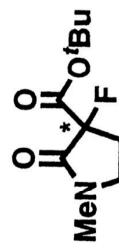
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 INIYL : 36.9 usec
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 DUMMY : 1 times
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 RESOL : 0.83 Hz
 PW1 : 5.85 usec
 CBNUC : ¹³C
 CBFHQ : 100.40 MHz
 OBSET : 135500.00 Hz
 RGAIN : 30
 1H : 399.65 MHz
 IRFRQ : 134300.00 Hz
 IRSET : 50.0 usec
 IRRPW : 0
 TRNS : 0
 SCANS : 1000 times
 SLVNT : CDCL₃
 SPINNT : 11 Hz
 TEMP : -22.8 °C



¹³C NMR



7c

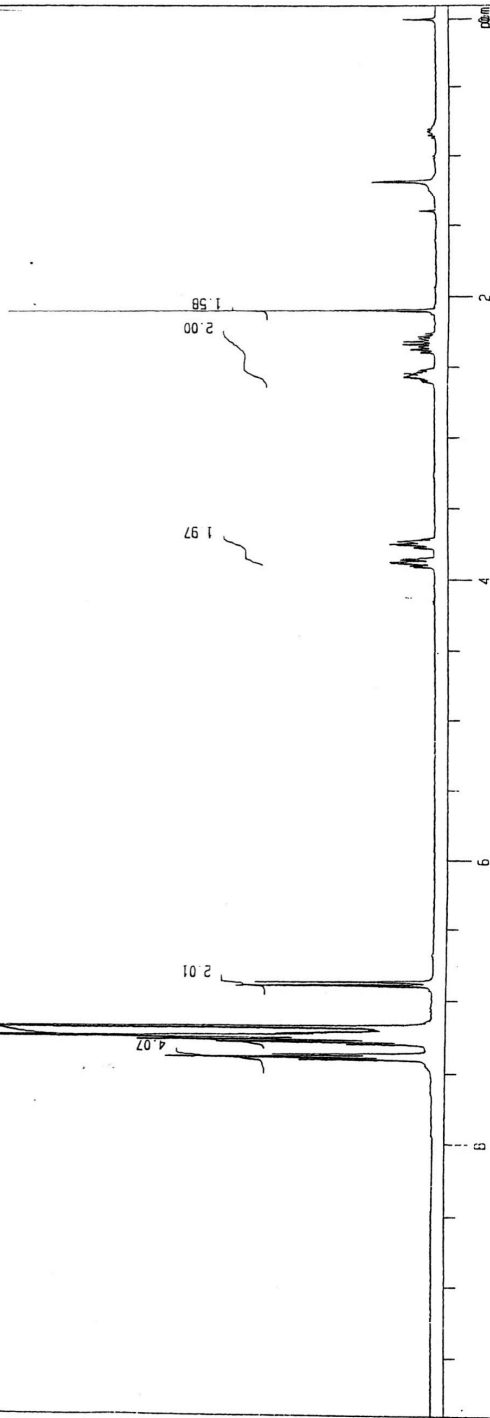
Date : Thu Feb 16 19:45:32 2006

Filename : LoadingFID.imdata
 Comment : 060228_fm137_1H
 SliceHistory : non
 EXMODE :

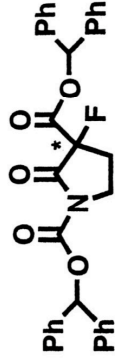
POINT 32768 points
 SAMPD 32768 points
 FREQ 7993.6 Hz
 F1 F2 4000 Hz
 DELAY 50.0 usec
 DEATH 72.0 usec
 INVT 125.1 usec
 TIMES 16 times
 DUMMY 1 times
 PD 2.9007 sec
 ACQTM 4099.2769 msec
 PRECL 10.00000 msec
 INITI 1000.00000 msec
 RESOL 0.24 Hz
 PM1 6.10 usec
 DBNLJ 399.65 MHz
 DBFRQ 134300.00 Hz
 DBSET 23
 RGAIN 16 times
 SCANS 16 times
 SLVNT CDCL3
 SPINNT 10 Hz
 TEMP 22.4 C

4.1481
 3.9107
 3.9022
 3.8845
 3.8802
 3.8760
 3.8619
 3.8528
 3.7747
 3.7606
 3.7558
 3.7338
 3.7289
 3.7100
 3.6074
 2.5982
 2.5884
 2.5787
 2.5701
 2.5634
 2.5512
 2.5439
 2.5348
 2.5244
 2.5158
 2.3975
 2.3755
 2.3566
 2.3407
 2.3187
 2.2992
 2.2840
 2.2644
 2.1009

7.4029
 7.3858
 7.3675
 7.2943
 7.2766
 7.2571
 7.2485
 7.2363
 7.2186
 7.2119
 7.1979
 7.1844
 7.1789
 6.8879
 6.8629



¹H NMR



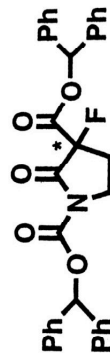
7d

Date : Thu Feb 16 19:42:25 2006

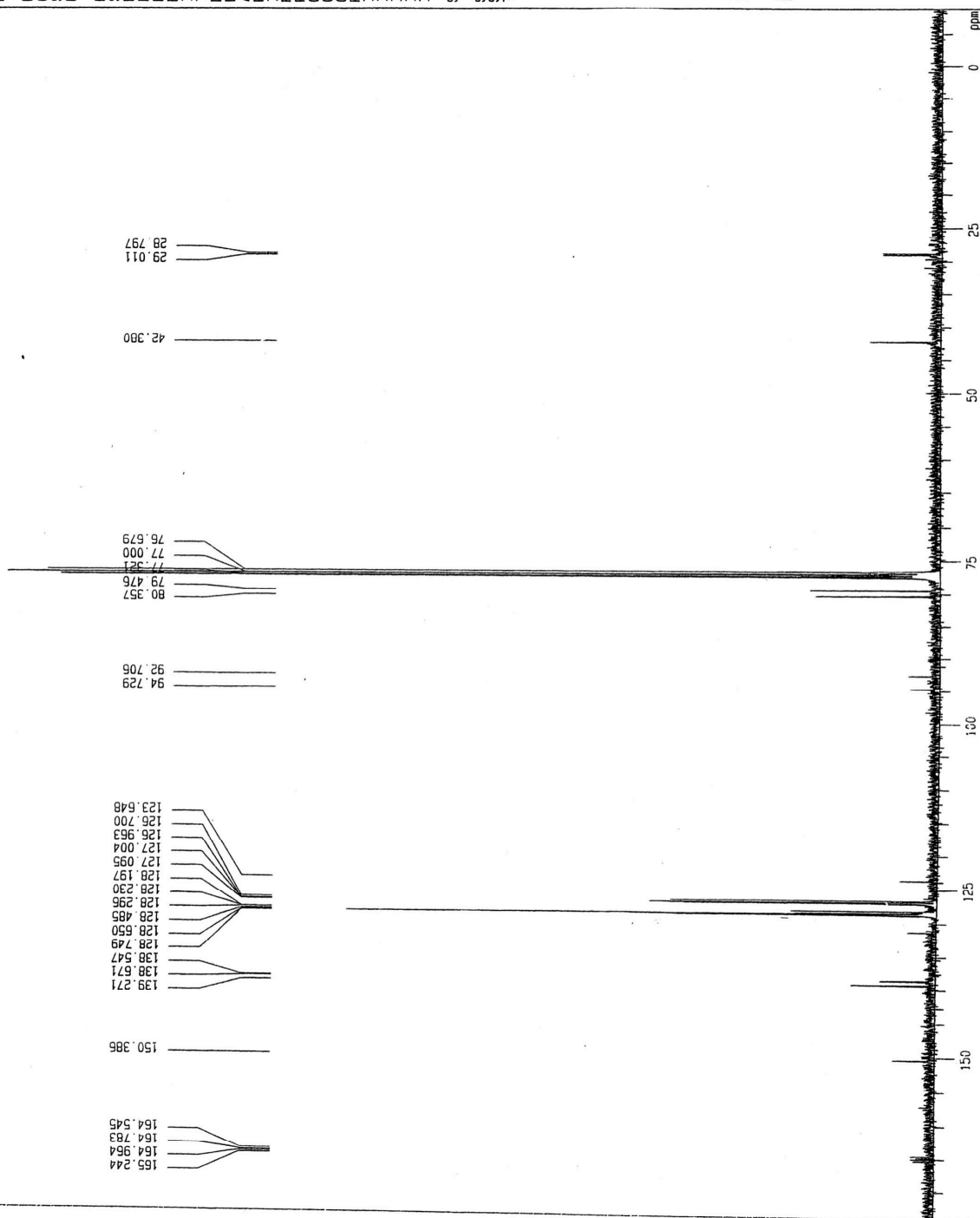
Filename : LoadingF10.mdata
 Comment : 060228_fm137
 SliceHistory : bcm
 EXMODE :

POINT 32768 points
 SAMPD 32768 points
 FREQ 27100.3 Hz
 F1LTH 13550 Hz
 DELAY 14.8 usec
 DEADT 19.2 usec
 INVL 35.9 usec
 TIMES 1000 times
 DUMMY 1 times
 PD 1.7909 sec
 ACQTM 1209.1393 msec
 PREDL 10.00000 msec
 INVT 1000.0000 msec
 RESOL 0.83 Hz
 PM1 5.85 usec
 13C 100.40 MHz
 135500.00 Hz
 29
 1H 399.65 MHz
 134300.00 Hz
 50.0 usec
 0
 SCANS 1000 times
 SLVNT CDCL3
 SPINNING 10 Hz
 TEMP 23.2 C

¹³C NMR



7d

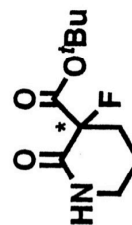


Date : Mon Nov 14 10:34:13 2005
 FileName : Loading10.nmdata
 Comment : 051126_fm97
 SliceHistory :
 EXMODE : non

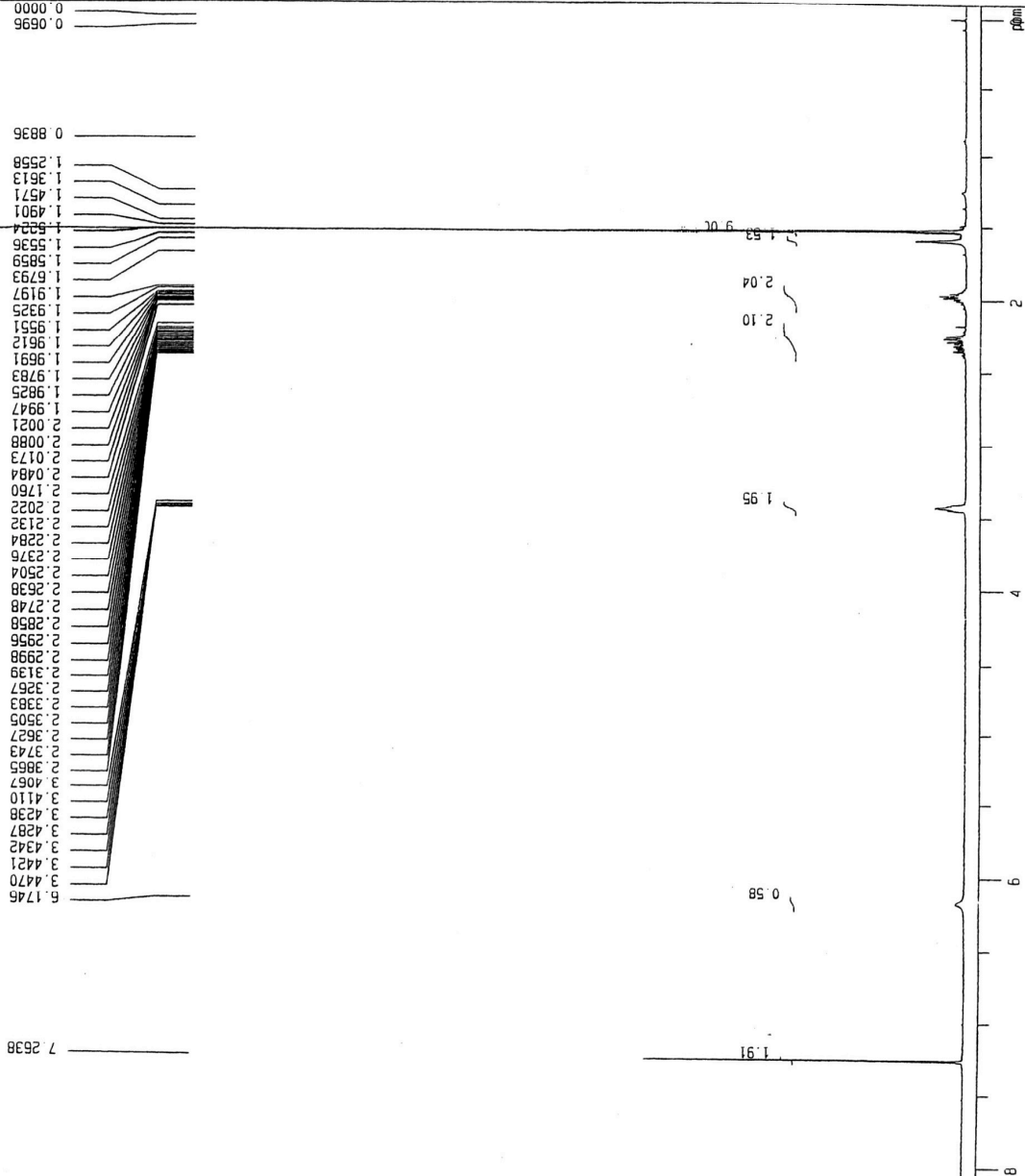
POINT : 32768 points
 SAMPO : 32768 points
 FREQ : 7993.6 Hz
 FILTR : 4000 Hz
 DELAY : 50.0 usec
 DEADT : 72.0 usec
 INTVL : 125.1 usec
 TIMES : 16 times
 DUMMY : 1 times
 PD : 2.9007 sec
 ACQTM : 4099.2769 msec
 PREDL : 10.00000 msec
 INIWT : 1000.0000 msec
 RESOL : 0.24 Hz
 PM1 : 6.10 usec
 OBNUC : ¹H
 OBFRQ : 399.65 MHz
 OBSET : 134300.00 Hz
 REAIN : 26

SCANS : 16 times
 SLVNT : CDCL3
 SPINNING : 12 Hz
 TEMP : 20.5 C

¹H NMR



7e



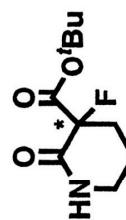
Date : Wed Nov 16 17:37:27 2005

Filename : LoadingFID.nddata
 Comment : 051128_7195_13Cco
 SliceHistory :
 EXMODE : bcm

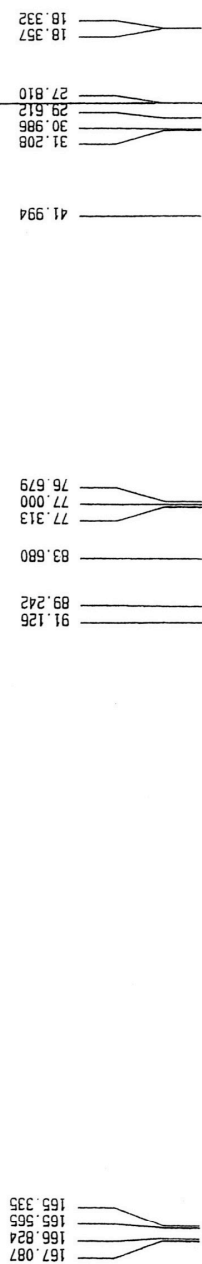
POINT : 32768 points
 SAMPD : 32768 points
 FREQU : 27100.3 Hz
 FILTR : 13550 Hz
 DELAY : 14.8 usec
 DEADT : 19.2 usec
 INTVL : 36.9 usec
 TIMES : 1000 times
 DUMMY : 1 times
 PD : 1.7909 sec
 ACQTM : 1209.1393 msec
 PREDL : 10.00000 msec
 INTWT : 1000.0000 msec
 RESOL : 0.83 Hz
 PFI : 5.85 usec
 13C : 100.40 MHz
 OBFRQ : 135500.00 Hz
 OBSET : 29
 FBALN : 1H
 IRNUC : 399.65 MHz
 IRFREQ : 134300.00 Hz
 IRSET : 50.0 usec
 IRRPW : 0
 TRNS : 1000 times

SCANS : 1000 times
 SLVNT : CDCl3
 SPINNING : 11 Hz
 TEMP : 22.3 C

¹³C NMR



7e



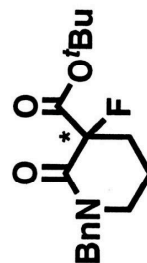
Date : Mon Dec 12 13:12:12 2005

FileName : LoadingID mmdata
 Comment : 051224_fm95y_13C
 SliceHistory : bcm
 EXMODE :

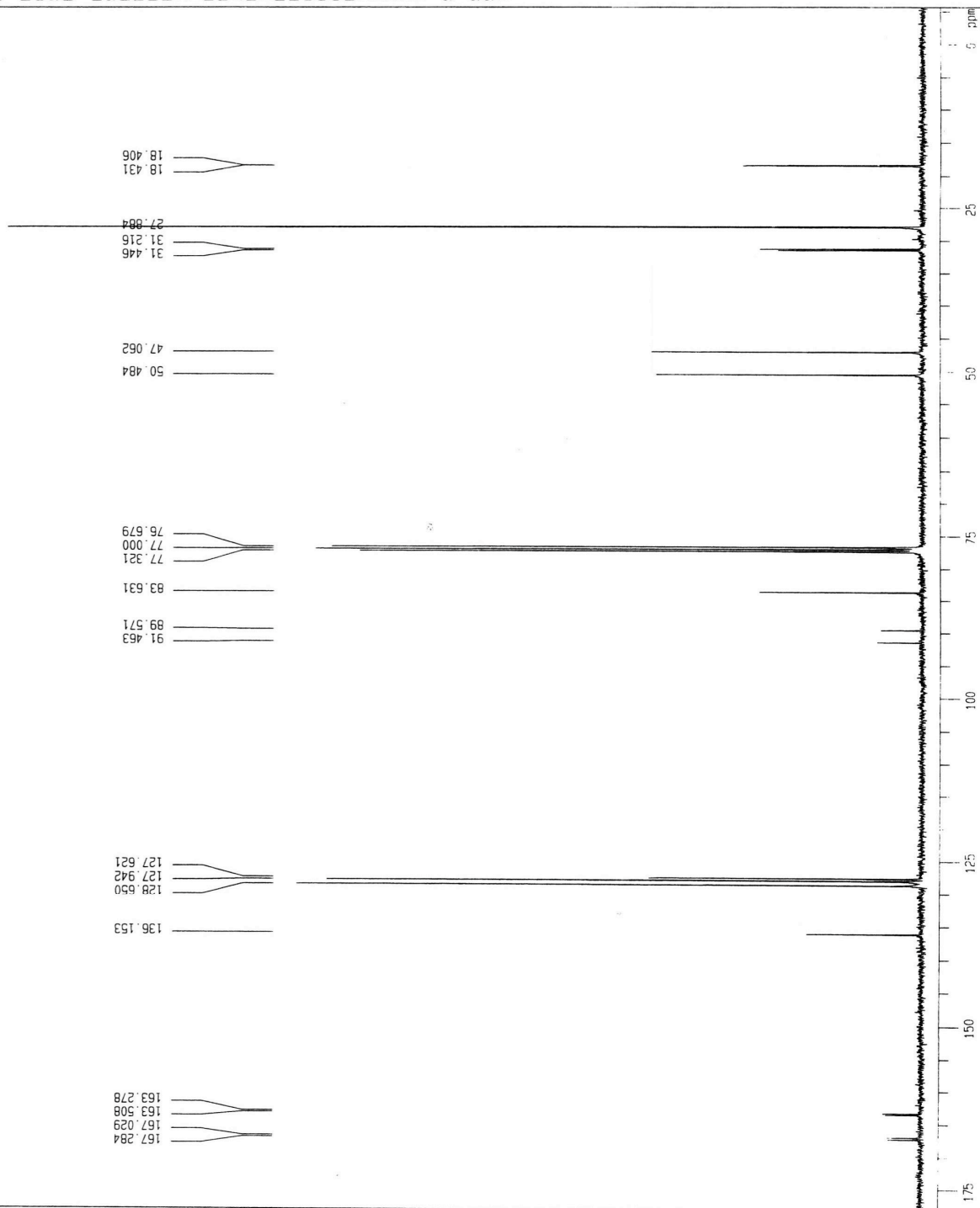
POINT : 32768 points
 SAMPD : 32768 points
 FREQ : 27100.3 Hz
 FIDR : 13550 Hz
 DELAY : 14.8 usec
 DEADT : 19.2 usec
 INYVL : 36.9 usec
 TIMES : 1000 times
 DUMMY : 1 times
 PD : 1.7909 sec
 ACQTM : 1209.1393 msec
 PREDL : 10.00000 msec
 INIWT : 1000.0000 msec
 RESOL : 0.83 Hz
 PM1 : 5.85 usec
 13C : 100.40 MHz
 GBNUC : 135500.00 Hz
 GBFRO : 30
 RGAIN : 399.65 MHz
 TRFRO : 134300.00 Hz
 TRSET : 50.0 usec
 TRPRM : 0
 TRRNS :

SCANS : 1000 times
 SLVNT : CDCL3
 SPINNING : 10 Hz
 TEMP : 23.0 C

¹³C NMR

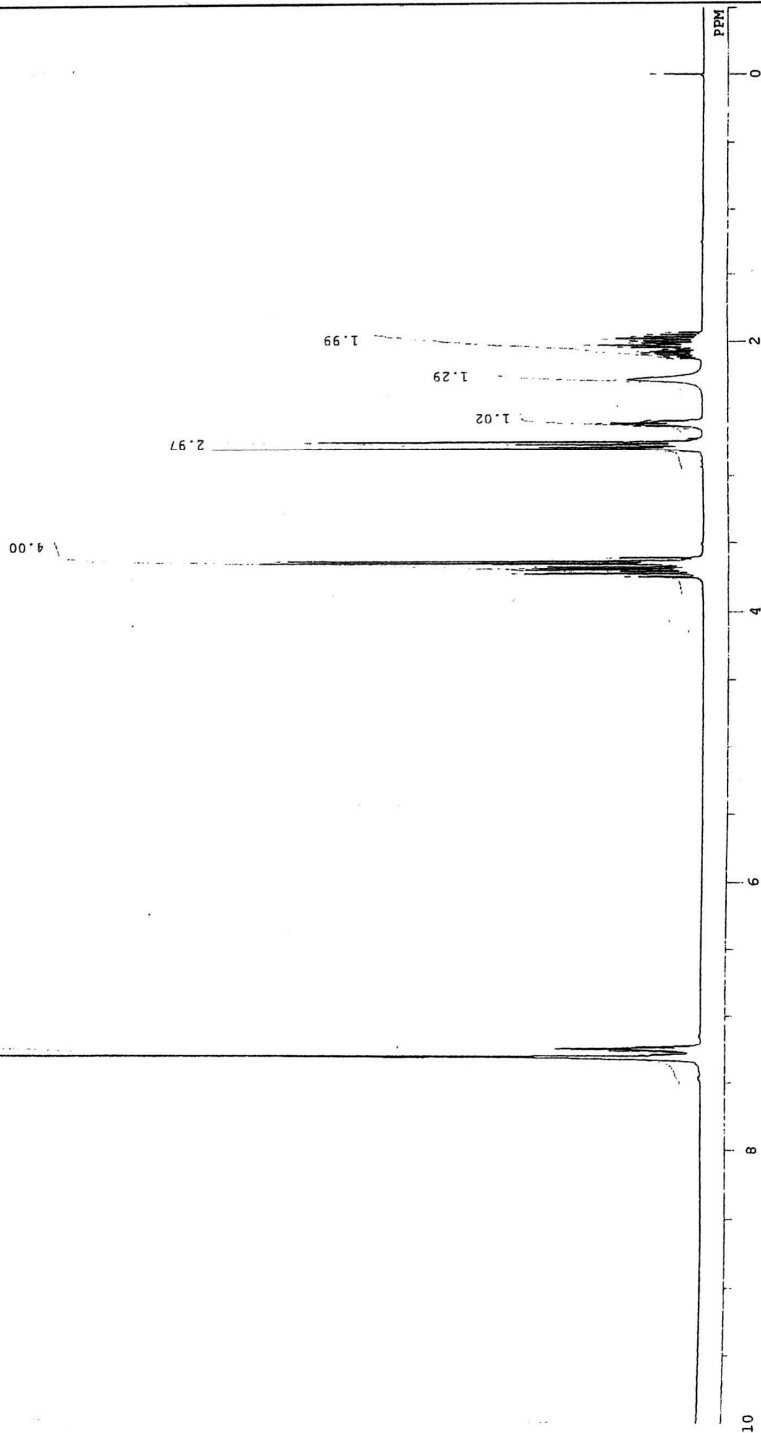


N-Bn-7e



J:\753-792-1h-500MHz.als
 Single Pulse Experiment
 22-SEP-2006 16:12:27
 1H
 single_pulse.exp
 OBRQ 500.00 MHz
 OBSE 162.00 kHz
 OBFN 416.02 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 16
 ACQTM 2.1823 sec
 PD 3.0000 sec
 FWH 7.00 usec
 1H
 IRRUC 23.5 c
 CTEMP CDCL3
 SLVNT
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 17

DEFL 0.0000
 COMET 1.9386
 DATTN 1.9532
 OBRUC 1.9670
 EXNOD 1.9826
 OBRQ 1.9972
 OBSE 2.0000
 OBFN 2.0137
 POINT 2.0201
 FREQU 2.0284
 SCANS 2.0348
 ACQTM 2.0430
 PD 2.0586
 FWH 2.0632
 IRRUC 2.0742
 CTEMP 2.0852
 SLVNT 2.0897
 EXREF 2.0998
 BF 2.1136
 RGAIN 2.1181
 DEFL 2.1282
 COMET 2.2840
 DATTN 2.5945
 OBRUC 2.5964
 EXNOD 2.6046
 OBRQ 2.6064
 OBSE 2.6129
 OBFN 2.6147
 POINT 2.6202
 FREQU 2.6229
 SCANS 2.6257
 ACQTM 2.6284
 PD 2.6303
 FWH 2.6385
 IRRUC 2.6403
 CTEMP 2.6596
 SLVNT 2.7292
 EXREF 2.7420
 BF 2.7539
 RGAIN 2.7695
 DEFL 2.7732
 COMET 2.7842
 DATTN 2.7878
 OBRUC 2.7988
 EXNOD 2.8172
 OBRQ 3.6124
 OBSE 3.6252
 OBFN 3.6325
 POINT 3.6380
 FREQU 3.6499
 SCANS 3.6564
 ACQTM 3.6756
 PD 3.6893
 FWH 3.7003
 IRRUC 3.7132
 CTEMP 3.7278
 SLVNT 3.7526
 EXREF 7.2348
 BF 7.2422
 RGAIN 7.2440
 DEFL 7.2522
 COMET 7.2559
 DATTN 7.2614
 OBRUC 7.2687
 EXNOD 7.2770
 OBRQ 7.2907
 OBSE 7.2953
 OBFN 7.2999
 POINT 7.3081
 FREQU 7.3164
 SCANS 7.3246
 ACQTM 7.3347

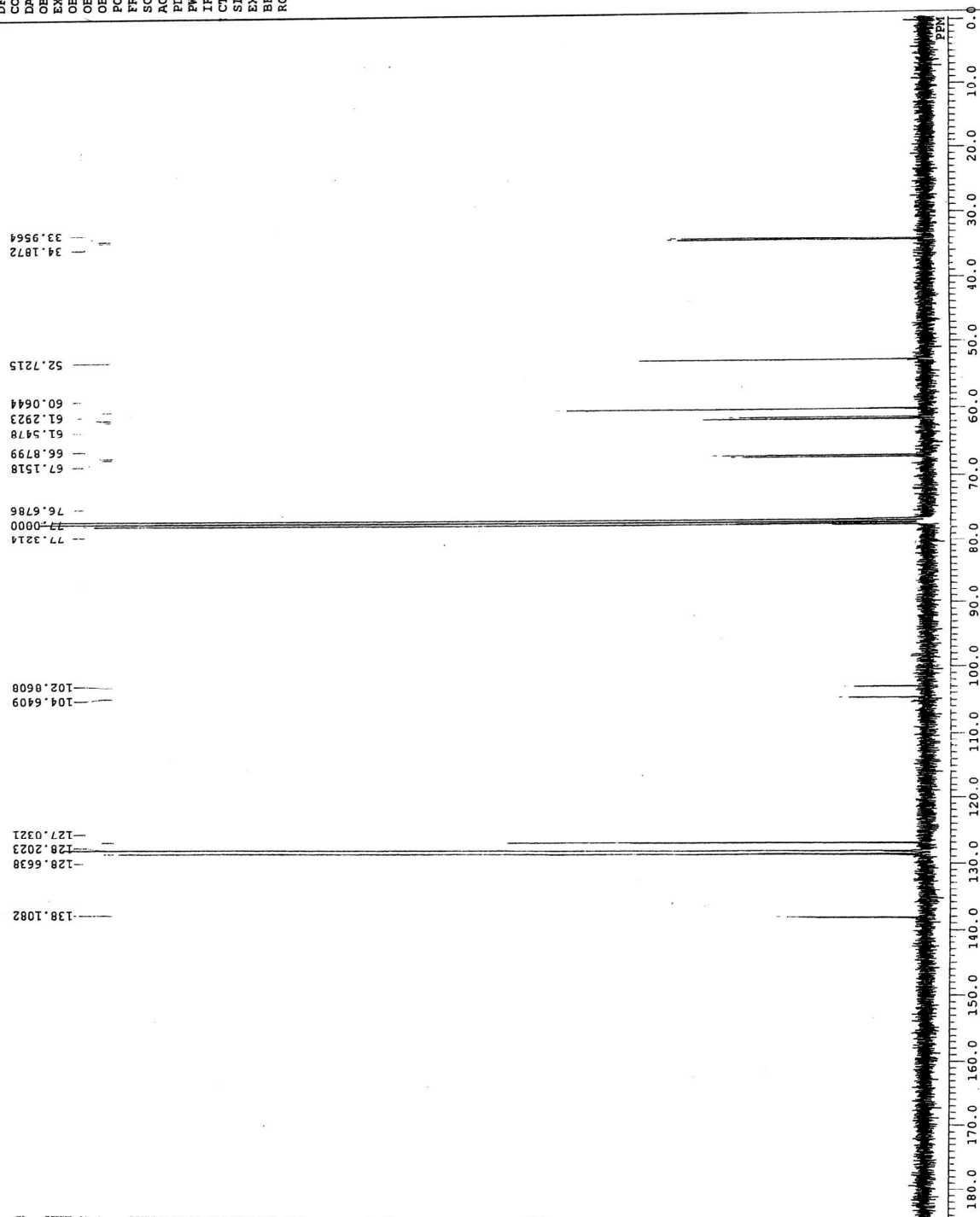


¹H NMR



8

DEFILE J:\753-13c.als
 COMMT Sat Aug 05 17:52:09 2006
 DATIN 13C
 EXMOD BCM
 OBFRQ 99.45 MHz
 OBSET 94.00 KHz
 OFIN 10309.00 Hz
 POINT 32768
 FREOU 26881.70 Hz
 SCANS 2307
 ACQTM 1.2190 sec
 PD 1.7810 sec
 PWI 6.00 usec
 IRNUC 1H
 CTEMP 22.9 c
 SIVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 25



¹³C NMR



8