

Supporting Information

Reductions of Aliphatic and Aromatic Nitriles to Primary Amines using Diisopropylaminoborane

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General Methods. All reactions were performed in oven-dried, argon-cooled glassware. All air and moisture-sensitive compounds were introduced via syringes or cannula through a rubber septum. THF was distilled from sodium-benzophenone. Aminoboranes were not isolated but were characterized and used in solutions. All reactions were analyzed by ^{11}B NMR spectroscopy and were recorded at 80.25 MHz. Chemical shifts are reported relative to external standard $\text{BF}_3\cdot\text{Et}_2\text{O}$ ($\delta = 0$). ^1H NMR and ^{13}C NMR spectra were recorded at 500 MHz (^1H) and 126 MHz (^{13}C). Chemical shifts are reported in δ units relative to CDCl_3 (7.26/77.0).

Preparation of *i*Pr-LAB Reagent 1 M Solution in THF. Diisopropylamine (5.00 g, 7 mL, 50 mmol) was mixed with anhydrous THF (18 mL) in a 100-mL round bottom flask. The solution was cooled to 0 °C (ice bath) and borane dimethylsulfide (5 mL, 10 M, 50 mmol) was added dropwise via syringe, stirred for 1 h at 0 °C and analyzed by ^{11}B -NMR. The analysis showed the solution to be diisopropylamine borane complex (δ -21.1, q, $J = 95.3$ Hz). Then, *n*-butyl lithium in hexanes (20 mL, 2.5 M, 50 mmol) was measured in oven dried graduated cylinder and added dropwise via cannula needle to the solution of amine borane at 0 °C (**CAUTION: Hydrogen evolution**). After stirring at 0 °C for 1 h, an aliquot was taken and analyzed by ^{11}B -NMR (80.25 MHz, THF) which showed the solution to be lithium diisopropylaminoborohydride (δ -23.6, q, $J = 83.4$ Hz). LAB reagent was transferred to an oven-dried, nitrogen cooled ampoule via a cannula needle.

Diisopropylaminoborane prepared from *i*Pr-LAB and Methyl Iodide. To a 50-mL round bottom flask with side arm was added lithium diisopropyl aminoborohydride (prepared from 1.1 equiv *n*-BuLi) (5.0 mL, 1 M in THF, 5.0 mmol) and 3 mL of anhydrous THF. The reaction was cooled to 0 °C (ice bath) and methyl iodide (0.31 mL, 5.0 mmol) was added dropwise

(**CAUTION:** *very exothermic reaction*). After all methyl iodide was added, the ice bath was removed and reaction was stirred at room temperature for 1 h. After 1 h, the ^{11}B -NMR spectrum showed formation of diisopropylaminoborane complex (δ +35, t, J = 125 Hz).

Pure diisopropylaminoboranes prepared from *i*Pr-LAB and TMS-Cl. A 100-mL round-bottom flask equipped with a magnetic stir bar and fitted with rubber septum was charged with lithium diisopropylaminoborohydride (prepared from 1.0 equiv *n*-BuLi) (25 mL, 1M solution in THF 25 mmol). Trimethylsilylchloride (3.20 mL, 25 mmol) was added dropwise over 5 min via syringe while stirring at 25 °C. After 1 h of stirring at 25 °C, an aliquot (0.5 mL) was taken and analyzed via ^{11}B -NMR, which showed the solution to be monomeric diisopropylaminoborane (δ +35.2, t, J =125 Hz).

Lithium Tetraphenylborate A 25-mL round-bottom flask equipped with a magnetic stir bar and fitted with a rubber septum was charged with phenyllithium (2.2 mL, 1.8 M solution in cyclohexane, 4 mmol) and tetrahydrofuran (1.66 mL). Boron trifluoride etherate (0.12 mL, 1 mmol) was added dropwise over 1 min. After 30 min of stirring at 25 °C an aliquot (0.5 mL) was taken and analyzed via ^{11}B -NMR, which showed the solution to be lithium tetraphenylborate (δ +3.0, s).

General Procedure for Competitive Reduction of an Aldehyde or Ester in the Presence of a Nitrile. The following competitive reduction of 4-bromo-2-fluorobenzonitrile and benzonitrile by $\text{BH}_2\text{N}(\text{iPr}_2)$ is representative. A 50-mL round bottom flask, equipped with a magnetic stir bar and fitted with a rubber septum, was charged with 4-bromo-2-fluorobenzonitrile (0.20 g, 1 mmol), mesitylene (0.09 mL, 1 mmol), benzaldehyde (0.106 g, 1 mmol) and THF (10 mL). An aliquot (0.1 mL) was withdrawn from the reaction mixture before the addition of

diisopropylaminoborane to serve as a reference (t_0). Diisopropylaminoborane¹⁰ (8 mL, 0.5 M solution in THF, 4 mmol) made with 1.1 equiv *n*-BuLi and MeI was then added to the flask via syringe. The reaction was monitored by analyzing aliquots via GC. Aliquots (0.1 mL) were diluted with pentane (1 mL) to precipitate diisopropylaminoborane to avoid contamination of the GC column by boron compounds. The percentage of unreacted 4-bromo-2-fluorobenzonitrile and benzaldehyde was determined from the peak areas using mesitylene as the internal standard.

Characterization of Compounds from Table 1, 2, and 3

4-Bromo-2-Fluorobenzyl amine (Table 1, Entry 2). Yellowish oil, 90% Yield, ¹H-NMR (500 MHz, CDCl₃) δ 1.80 (bs, 2H), 3.83 (s, 2H), 7.18-7.25 (m, 3H); ¹³C-NMR (125 MHz, CDCl₃) δ 40.1, 119.2, 120.8, 127.5, 129.3, 160.8; exact mass m/z calcd for C₇H₇BrFN, HRMS (70eV) m/z ($M^+ + 1$) calcd. 202.97459, found 202.97558.

4-Bromo-2-Chlorobenzyl amine (Table 1, Entry 3). Yellowish oil, 99% Yield, ¹H-NMR (500 MHz, CDCl₃) δ 2.46 (s, 2H), 3.87 (s, 2H), 7.27 (d, 1H, $J = 4.5$), 7.37 (d, 1H, $J = 8$), 7.51 (s, 1H); ¹³C-NMR (125 MHz, CDCl₃) δ 43.4, 121.0, 130.2, 131.9, 134.1, 138.8; exact mass m/z calcd for C₇H₇BrClN, HRMS (70eV) m/z ($M^+ + 1$) calcd. 218.94504, found 218.94403.

5-Bromo-2-Fluorobenzyl amine (Table 1, Entry 4). Yellowish oil, 95% Yield, ¹H-NMR (500 MHz, CDCl₃) δ 2.42 (s, 2H), 3.87 (s, 2H), 6.92 (t, 1H, $J = 9$), 7.33 (m, 1H), 7.49 (m, 1H); ¹³C-NMR (125 MHz, CDCl₃) δ 40.0, 117.2, 117.4, 131.7, 132.3, 159.1, 161.0; exact mass m/z calcd for C₇H₇BrFN, HRMS (70eV) m/z ($M^+ + 1$) calcd. 202.97459, found 202.97563.

3-Bromobenzyl amine (Table 1, Entry 5). Yellowish oil, 94% Yield, ¹H-NMR (500 MHz, CDCl₃) δ 2.85 (bs, 2H), 3.80 (s, 2H), 7.16 (t, 1H, $J = 7.5$), 7.23 (d, 1H, $J = 7.5$), 7.38 (d, 1H, $J =$

8), 7.45 (s, 1H); ^{13}C -NMR (125 MHz, CDCl_3) δ 45.3, 122.7, 126.1, 130.2, 144.0; exact mass m/z calcd for $\text{C}_7\text{H}_8\text{BrN}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 184.98401, found 184.98389.

2-Bromobenzyl amine (Table 1, Entry 6). Yellowish oil, 94% Yield, ^1H -NMR (500 MHz, CDCl_3) δ 2.85 (s, 2H), 3.80 (s, 2H), 7.13 (t, 1H, $J = 7.5$), 7.30 (t, 1H, $J = 7.5$), 7.39 (d, 1H, $J = 7.75$), 7.54 (d, 1H, $J = 8$); ^{13}C -NMR (125 MHz, CDCl_3) δ 46.4, 123.5, 127.8, 128.7, 129.4, 132.8, 141.0, 163.2; exact mass m/z calcd for $\text{C}_7\text{H}_8\text{BrN}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 184.98401, found 184.98441.

3-Iodobenzyl amine (Table 1, Entry 7). Yellowish oil, 95% Yield, ^1H -NMR (500 MHz, CDCl_3) δ 1.50 (s, 2H), 3.80 (s, 2H), 7.04 (t, 1H, $J = 7.5$), 7.25 (d, 1H, $J = 7.5$), 7.55 (d, 1H, $J = 8$), 7.66 (s, 1H); ^{13}C -NMR (125 MHz, CDCl_3) δ 45.8, 126.4, 130.3, 135.8, 136.2, 145.7; exact mass m/z calcd for $\text{C}_7\text{H}_8\text{IN}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 232.97014, found 232.97124.

4-Fluorobenzyl amine (Table 1, Entry 8). Yellowish oil, 97% Yield, ^1H -NMR (500 MHz, CDCl_3) δ 1.45 (s, 2H), 3.85 (s, 2H), 7.19 (m, 2H), 7.29 (m, 2H); ^{13}C -NMR (125 MHz, CDCl_3) δ 44.9, 115.1, 115.3, 127.0, 137.8, 160.9, 162.8; exact mass m/z calcd for $\text{C}_7\text{H}_8\text{FN}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 125.06408, found 125.06413.

Benzyl amine (Table 1, Entry 9). Yellowish oil, 60% Yield, ^1H -NMR (500 MHz, CDCl_3) δ 3.25 (s, 2H), 3.85 (s, 2H), 7.25-7.36 (m, 4H); ^{13}C -NMR (125 MHz, CDCl_3) δ 45.8, 127.4, 127.6, 128.8, 141.4; exact mass m/z calcd for $\text{C}_7\text{H}_9\text{N}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 107.07350, found 107.07346.

[4-(aminomethyl)phenyl]methanol (Table 1, Entry 10). Yellowish oil, 80% Yield, ^1H -NMR (500 MHz, CDCl_3) δ 2.94 (bs, 2H), 3.69 (s, 2H), 4.56 (s, 2H), 7.15 (d, 2H, $J = 8$), 7.24 (m, 2H);

^{13}C -NMR (125 MHz, CDCl_3) δ 45.9, 64.4, 127.3, 128.4, 140.5, 141.5; exact mass m/z calcd for $\text{C}_8\text{H}_{11}\text{NO}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 137.08406, found 137.08511.

2-Chloro-6-Fluorobenzyl amine (Table 2, Entry 2). Yellowish oil; 70 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 1.80 (bs, 2H), 3.99 (s, 2H), 6.99 (m, 1H), 7.17 (m, 2H); ^{13}C -NMR (125 MHz, CDCl_3) δ 37.6, 114.4, 125.6, 128.9, 129.0, 160.4; exact mass m/z calcd for $\text{C}_7\text{H}_7\text{ClFN}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 159.02511, found 159.02601.

4-Dimethylaminebenzylamine (Table 2, Entry 3). Yellowish oil, 93 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 1.80 (bs, 2H), 2.94 (s, 6H), 3.77 (s, 2H), 6.73 (d, 2H, $J = 8.5$), 7.20 (d, 2H, $J = 8.5$) ; ^{13}C -NMR (125 MHz, CDCl_3) δ 40.8, 45.8, 113.0, 128.3, 131.2, 150.0; exact mass m/z calcd for $\text{C}_9\text{H}_{14}\text{N}_2$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 151.12161, found 151.12298.

4-Methoxybenzyl amine (Table 2, Entry 4). Yellowish oil; 80 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 2.42 (bs, 2H), 3.87 (s, 2H), 6.92 (d, 2H, $J = 9$ Hz), 7.33 (d, 1H); ^{13}C -NMR (125 MHz, CDCl_3) δ 45.5, 55.4, 114.1, 129.7, 132.5, 158.8; exact mass m/z calcd for $\text{C}_8\text{H}_{11}\text{NO}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 137.08406, found 137.08511.

p-Tolylmethanamine (Table 2, Entry 5). Colorless oil, 80 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 2.15 (bs, 2H), 2.34 (s, 3H), 3.80 (s, 2H), 7.15 (d, 2H, $J = 8$), 7.20 (d, 2H, $J = 8.5$); ^{13}C -NMR (125 MHz, CDCl_3) δ 21.0, 45.8, 127.2, 129.3, 136.5, 139.8; exact mass m/z calcd for $\text{C}_8\text{H}_{11}\text{N}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 122.09806, found 122.09643.

(4-(allyloxy)phenyl)methanamine (Table 2, Entry 6). Colorless oil, 85 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 2.17 (bs, 2H), 3.70 (s, 2H), 4.45 (d, 2H, $J = 7$), 5.21 (d, 1H, $J = 10$), 5.36

(d, 1H, $J=10$), 6.00 (octet, 1H, $J = 5$), 6.83 (d, 2H, $J = 8.5$), 7.15 (d, 1H, $J = 8$); ^{13}C -NMR (125 MHz, CDCl_3) δ 45.5, 68.8, 114.9, 117.6, 128.5, 133.5, 135.0, 157.8

2-(4-Methoxyphenyl)ethanamine (Table 3, Entry 2). Yellowish oil, 94 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 2.67 (t, 2H, $J = 7$), 2.93 (t, 2H, $J = 7$), 3.79 (s, 3H), 6.84 (d, 2H, $J = 6.5$), 7.10 (d, 2H, $J = 8.5$); ^{13}C -NMR (125 MHz, CDCl_3) δ 39.1, 43.7, 55.2, 113.9, 129.8, 131.8, 157.9; exact mass m/z calcd for $\text{C}_9\text{H}_{13}\text{NO}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 152.10823, found 152.10699.

Phenethylamine oxalate (Table 3, Entry 3). White Solid; 83 % crude yield of amine; ^1H -NMR (500 MHz, MeOH-d_4) δ 2.93 (t, 2H, $J = 8$), 3.13 (t, 2H, $J = 8$), 7.25 (m, 3H), 7.32 (m, 2H); ^{13}C -NMR (125 MHz, CDCl_3) δ 33.2, 40.4, 126.8, 128.3, 128.5, 136.6, 164.8

N-(hex-5-ynyl)benzamide (Table 3, Entry 4). Yellowish oil; 80 %; ^1H -NMR (500 MHz, CDCl_3) δ 1.61 (m, 2H), 1.74 (m, 1H), 1.96 (t, 1H, $J = 2.5$), 2.24 (dt, 2H, $J = 2.5, 6$), 3.47 (q, 2H, $J = 6$), 6.47 (bs, 1H), 7.41 (t, 2H, $J = 8$), 7.48 (t, 1H, $J = 7$), 7.75 (d, 2H, $J = 7.5$); ^{13}C -NMR (125 MHz, CDCl_3) δ 18.0, 25.7, 28.6, 39.5, 68.8, 84.0, 126.9, 128.6, 131.5, 134.6, 167.8 exact mass m/z calcd for $\text{C}_{13}\text{H}_{15}\text{NO}$, HRMS (70eV) m/z ($\text{M}^+ + 1$) calcd. 202.12394, found 202.12264.

Dodecylamine (Table 3, Entry 5). White solid; 99 % yield; ^1H -NMR (500 MHz, CDCl_3) δ 0.83 (t, 3H, $J = 7$), 1.21 (m, 20H), 2.63 (t, 2H, $J = 7.5$); ^{13}C -NMR (125 MHz, CDCl_3) δ 13.8, 22.5, 26.8, 29.2, 29.3, 29.5, 31.8, 32.7, 41.5

Undecan-1-amine (Table 3, Entry 6). Yellowish oil; 95% yield; ^1H -NMR (500 MHz, CDCl_3) δ 0.87 (t, 3H, $J = 7.5$), 1.25 (m, 16H), 1.55 (m, 2H), 2.65 (m, 2H); ^{13}C -NMR (125 MHz, CDCl_3) δ

14.0, 22.6, 29.3, 29.6, 32.9; exact mass m/z calcd for $C_{11}H_{25}N$, HRMS (70eV) m/z ($M^+ + 1$)
calcd. 171.1987, found 171.12013.

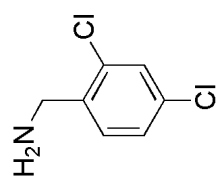
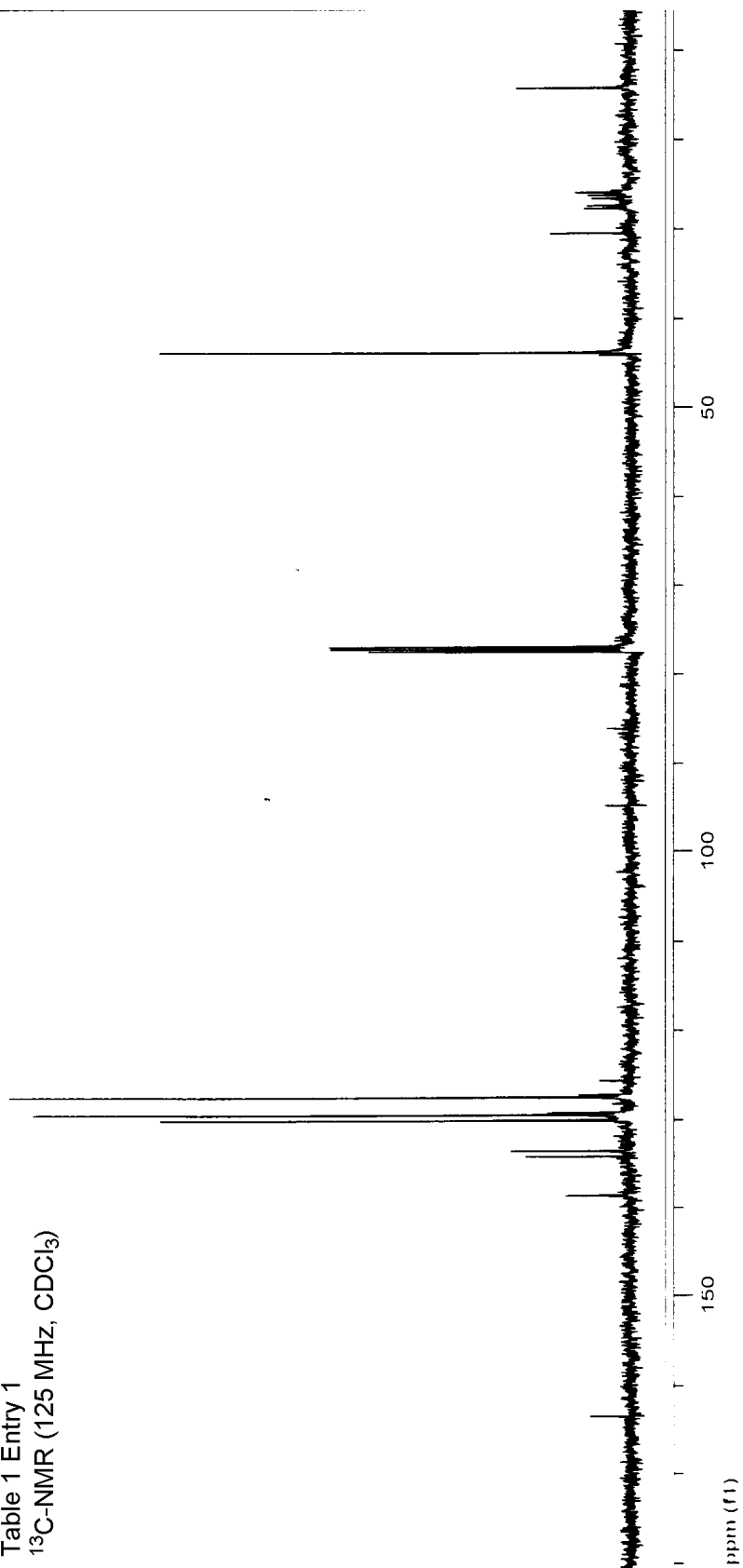


Table 1 Entry 1
 ^{13}C -NMR (125 MHz, CDCl_3)



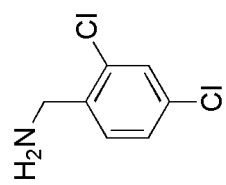
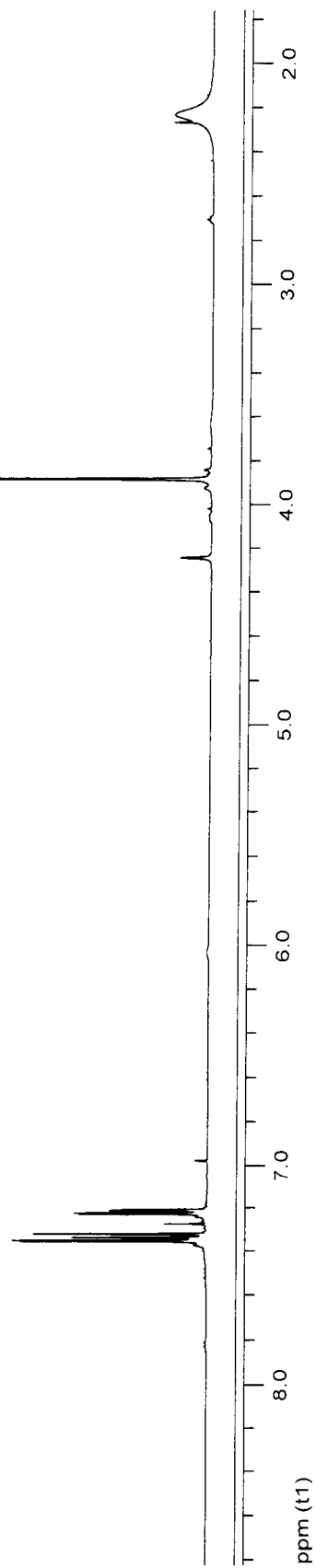


Table 1 Entry 1
¹H-NMR (500 MHz, CDCl₃)



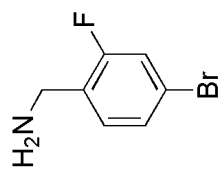
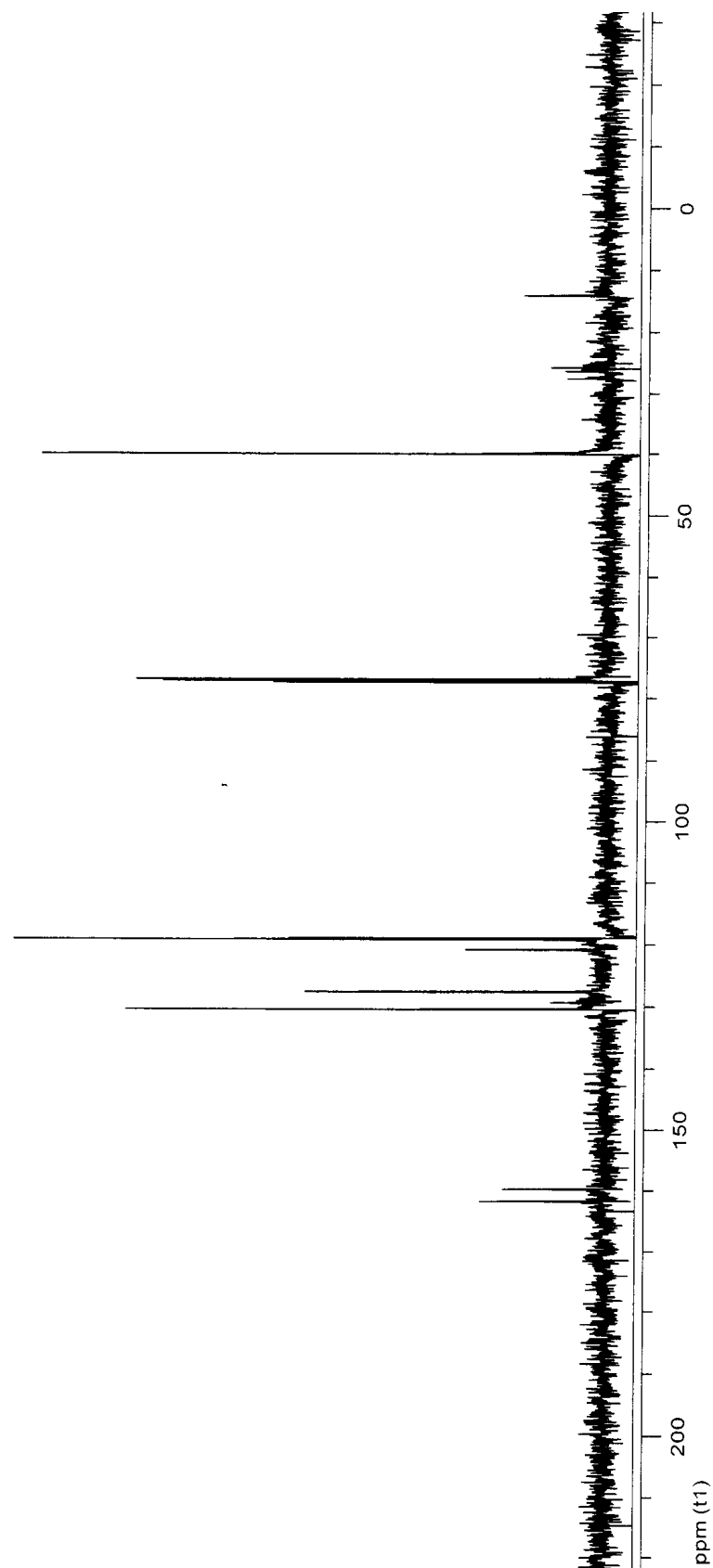


Table 1 Entry 2
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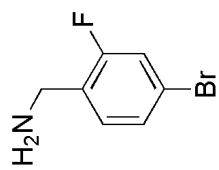
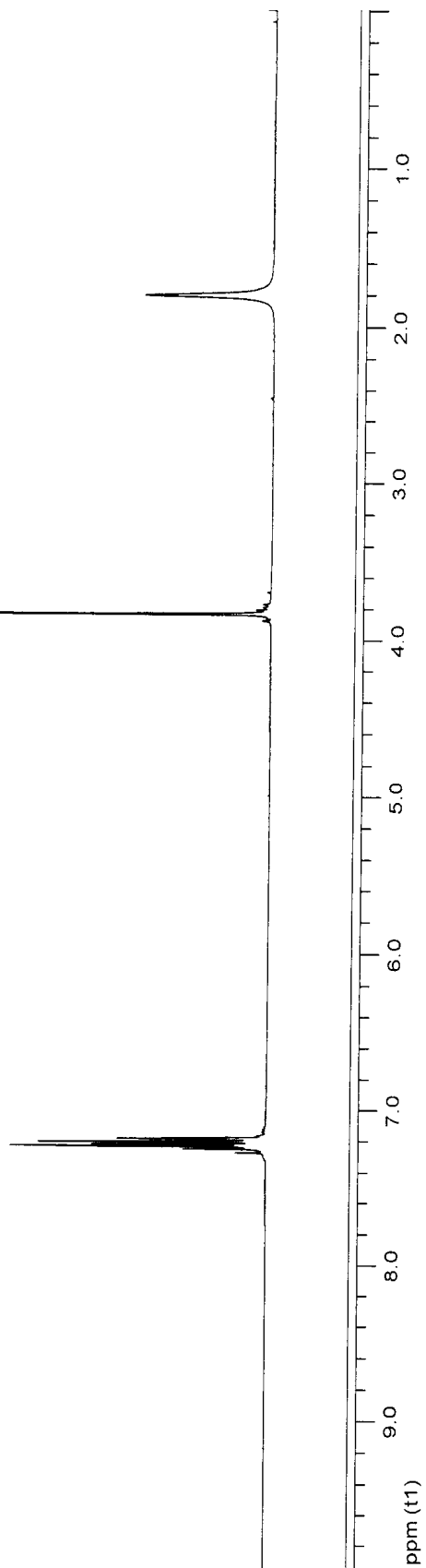


Table 1 Entry 2
¹H-NMR (500 MHz, CDCl₃)

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S13



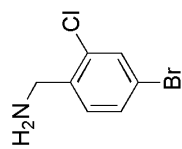
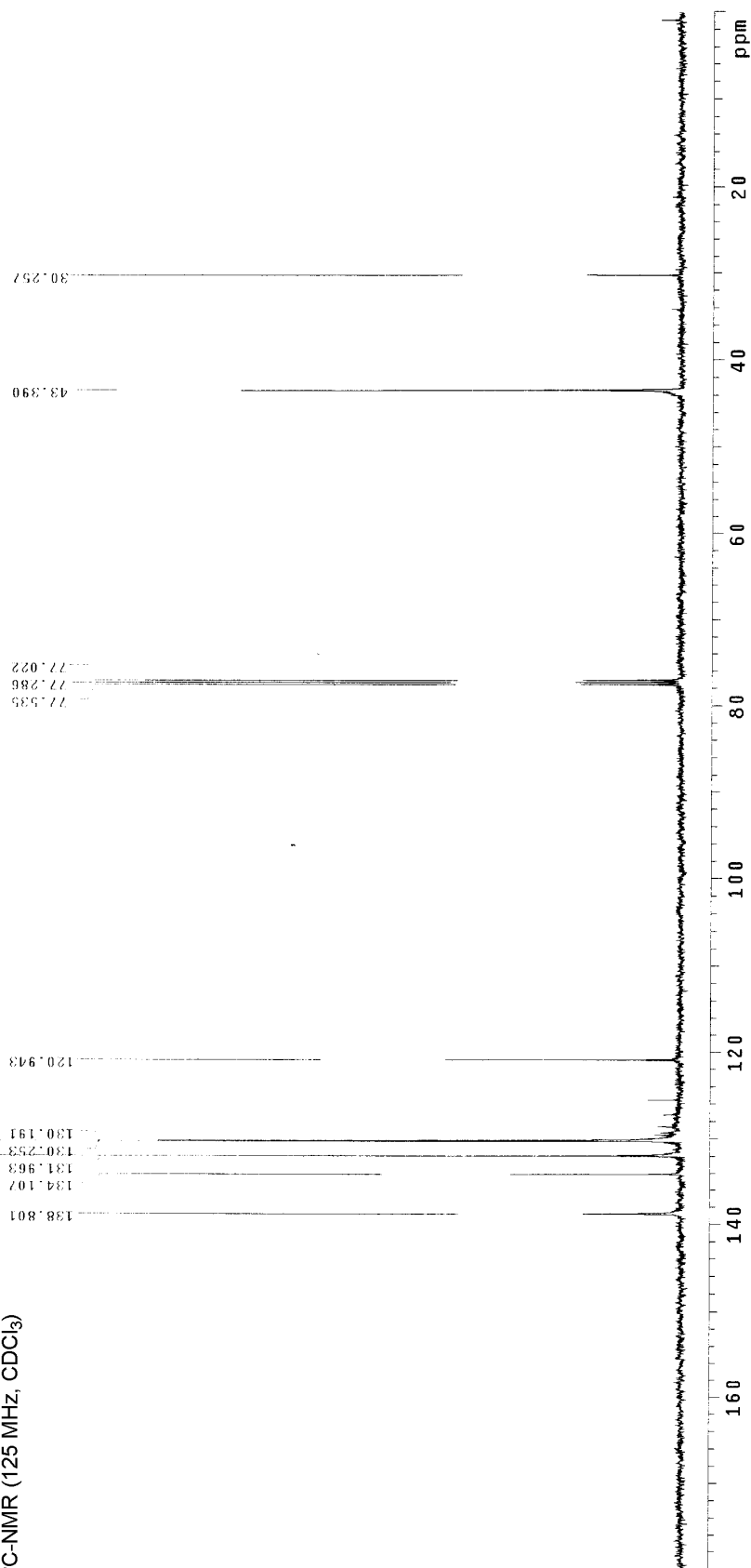


Table 1 Entry 3
¹³C-NMR (125 MHz, CDCl₃)



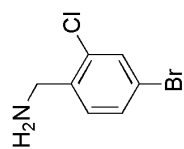
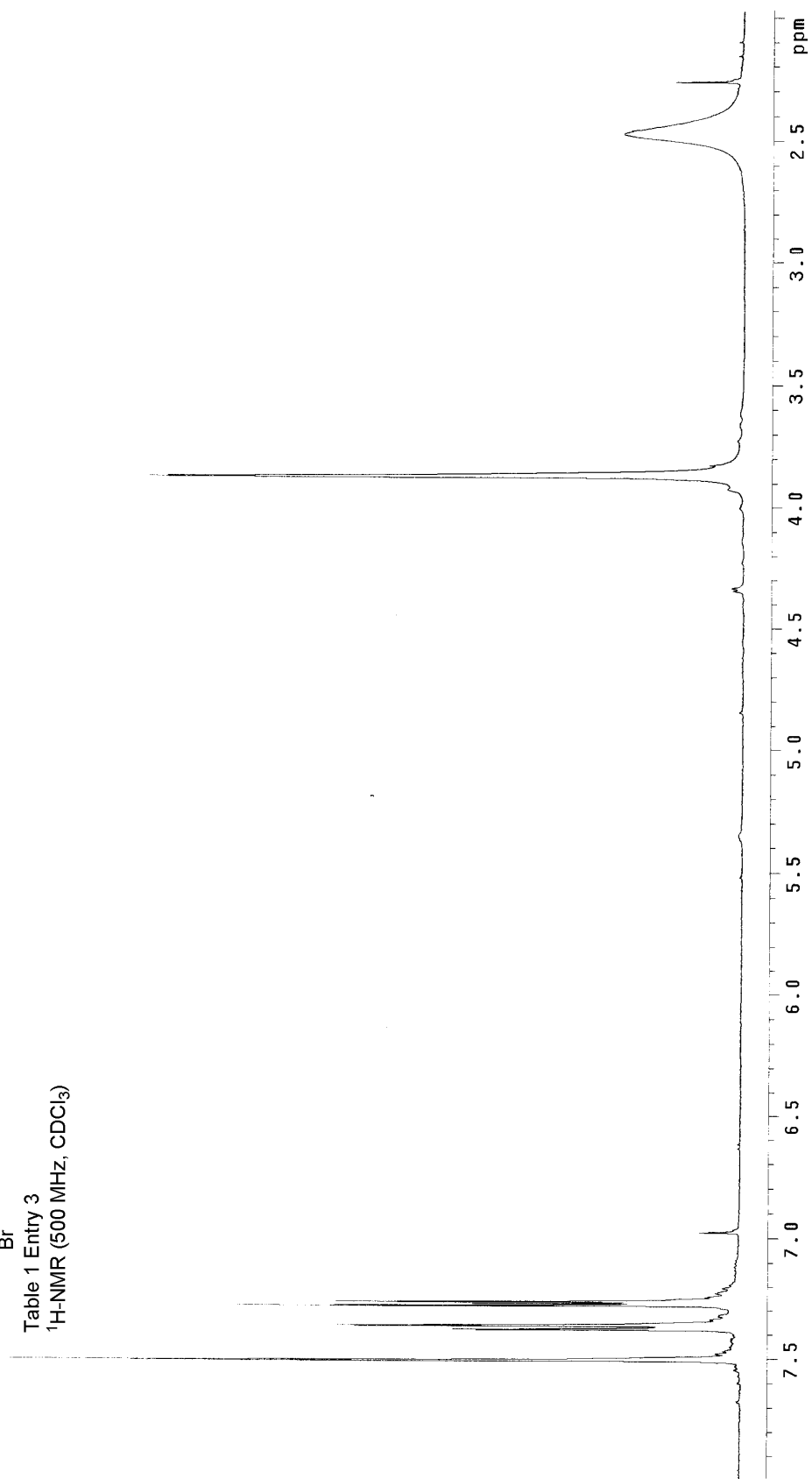


Table 1 Entry 3
¹H-NMR (500 MHz, CDCl₃)



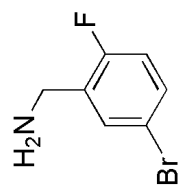
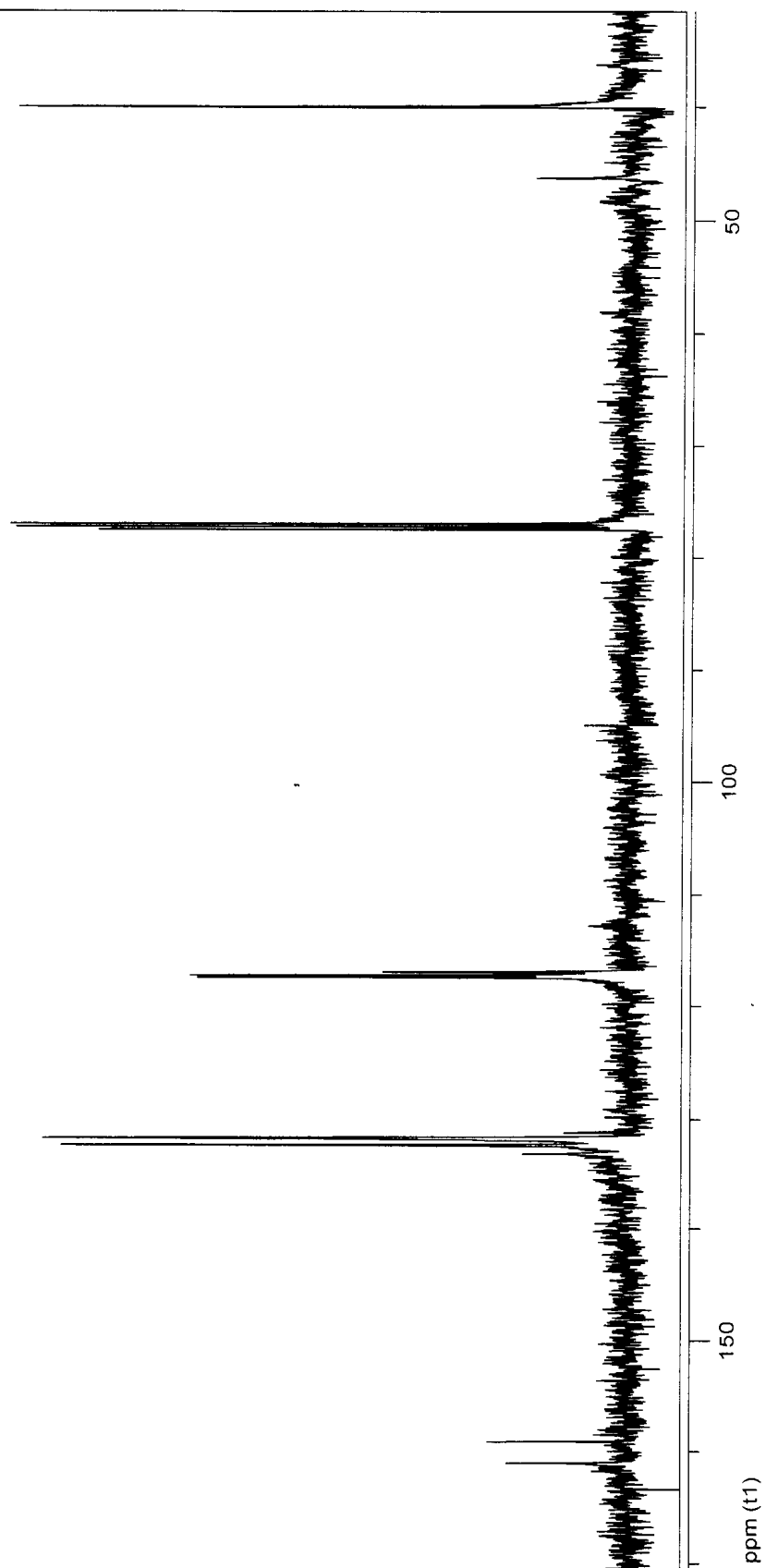


Table 1 Entry 4
¹³C-NMR (125 MHz, CDCl₃)



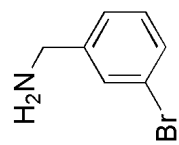


Table 1 Entry 5
 ^{13}C -NMR (125 MHz, CDCl_3)

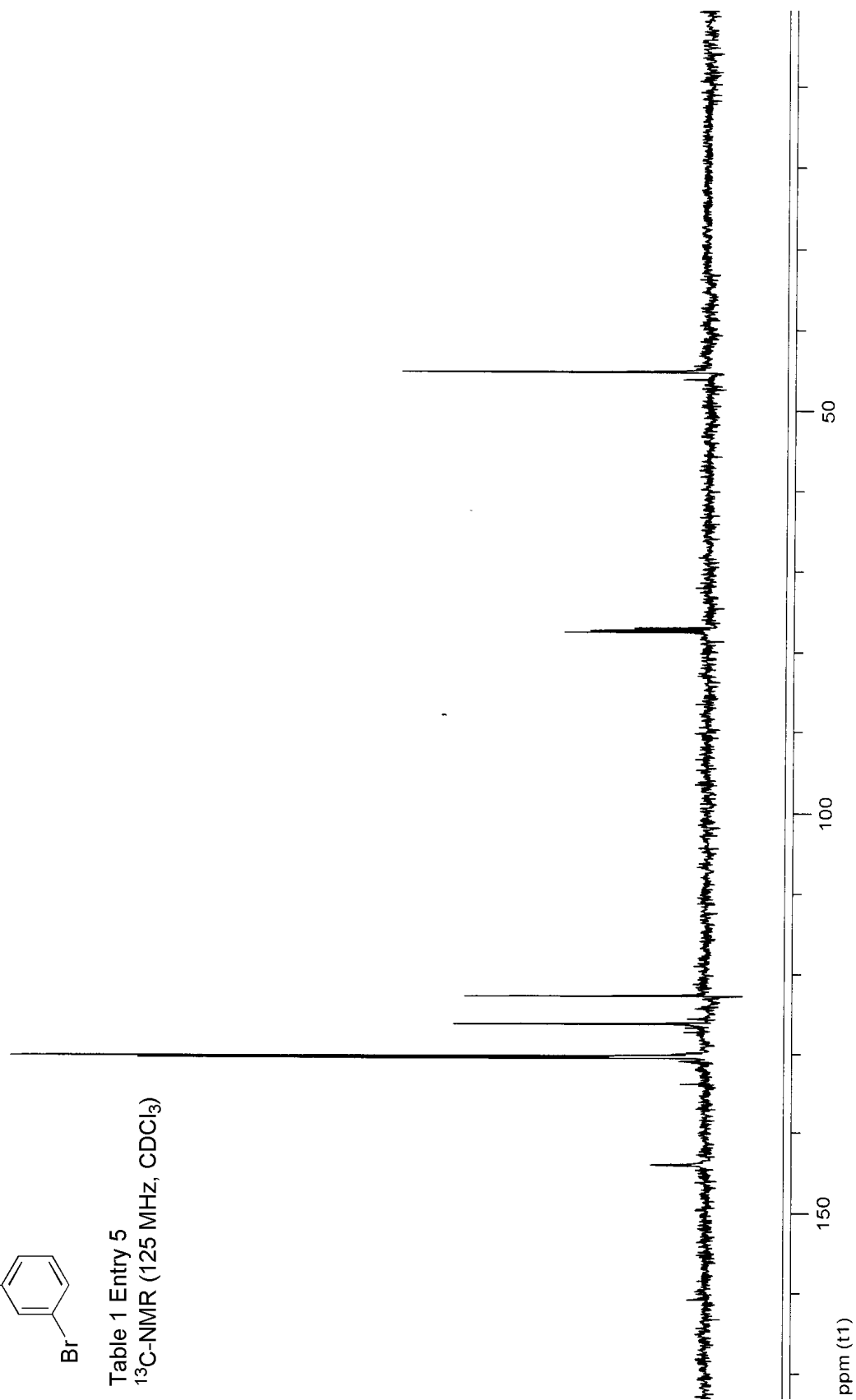
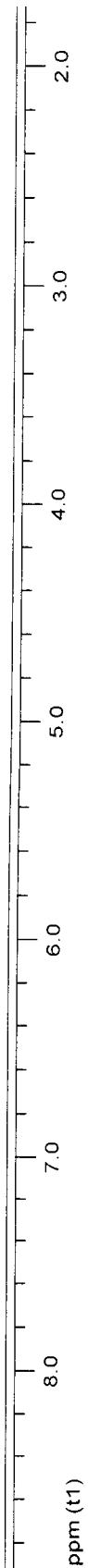


Table 1 Entry 5
¹H-NMR (500 MHz, CDCl₃)



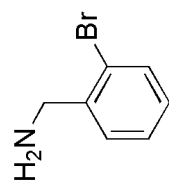
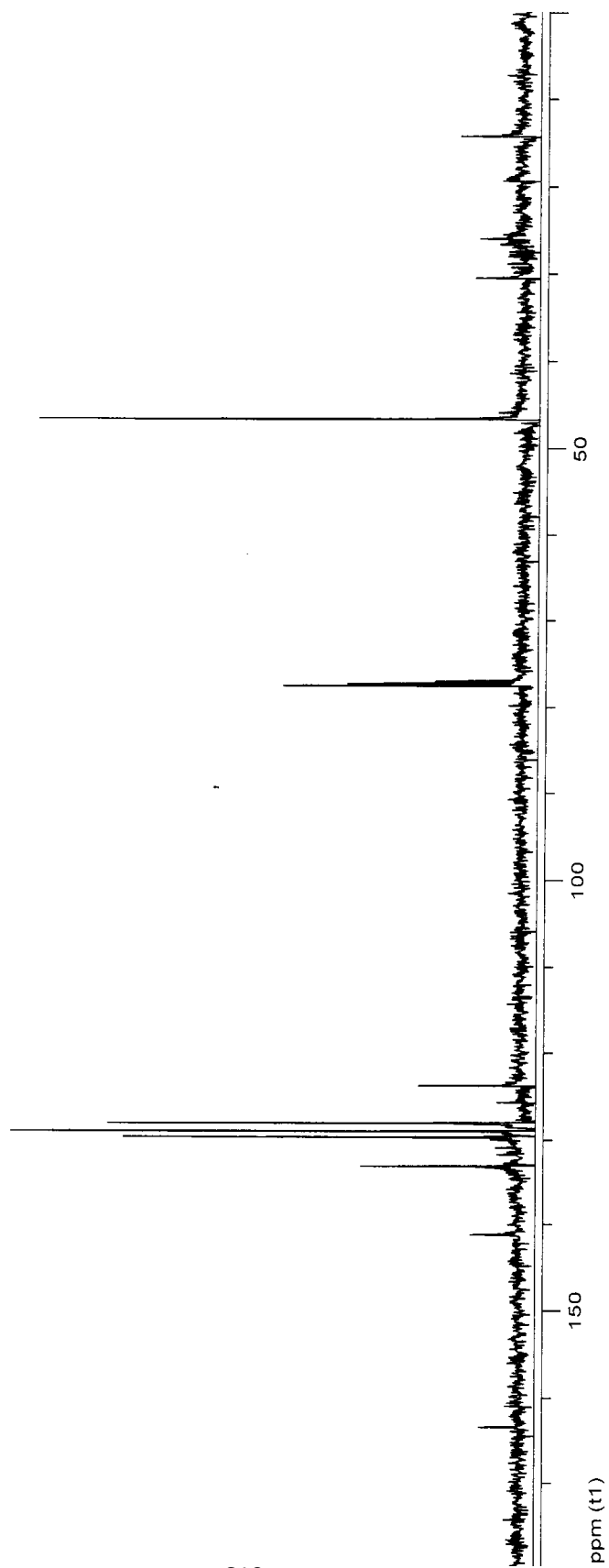


Table 1 Entry 6
 ^{13}C -NMR (125 MHz, CDCl_3)



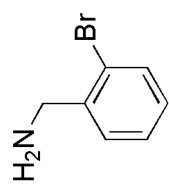
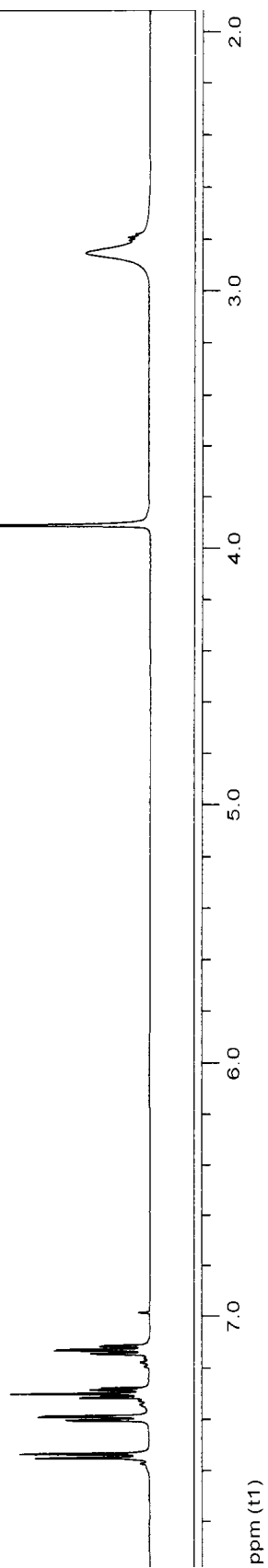


Table 1 Entry 6
 ^1H -NMR (500 MHz, CDCl_3)



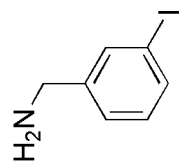
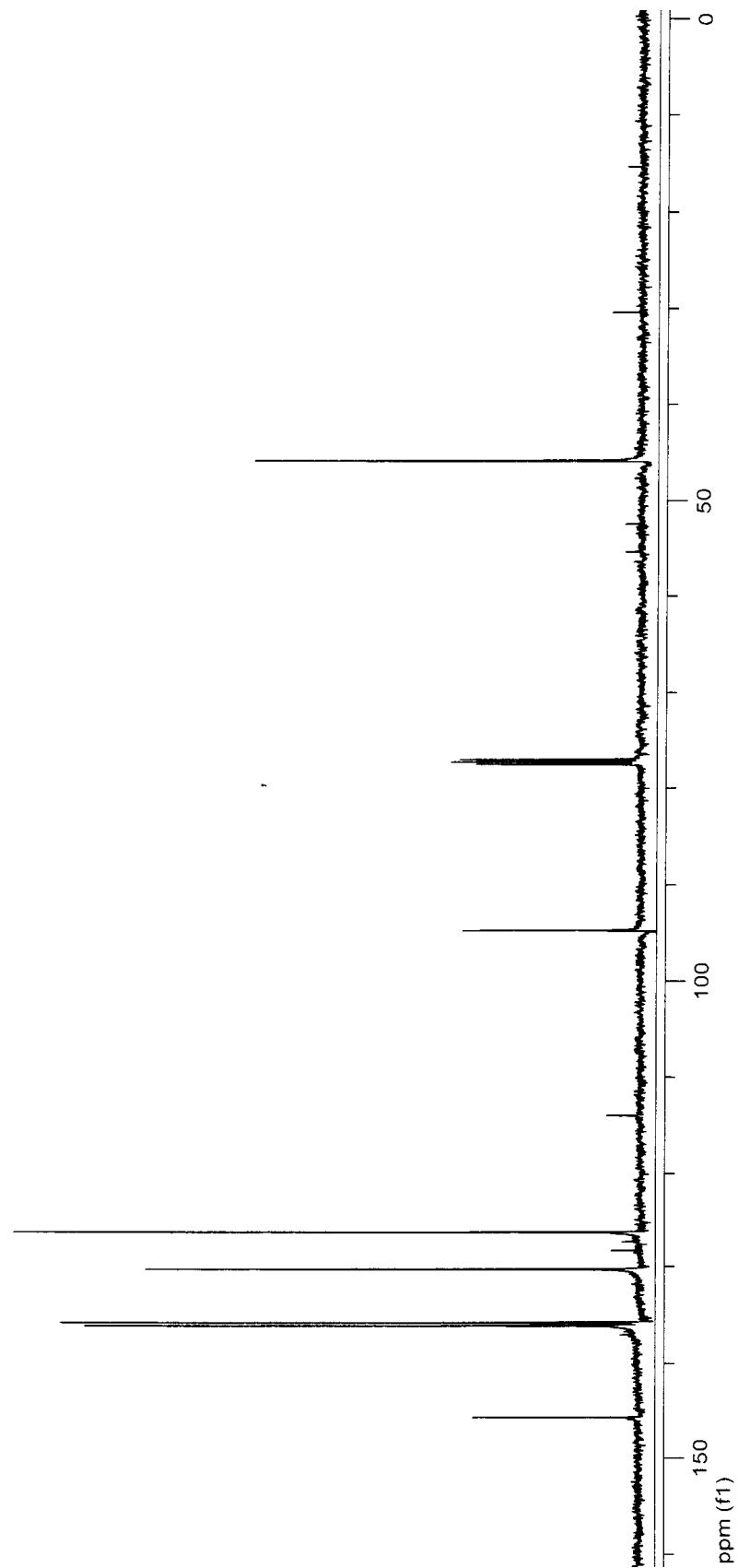


Table 1 Entry 7
 ^{13}C -NMR (125 MHz, CDCl_3)



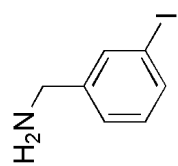
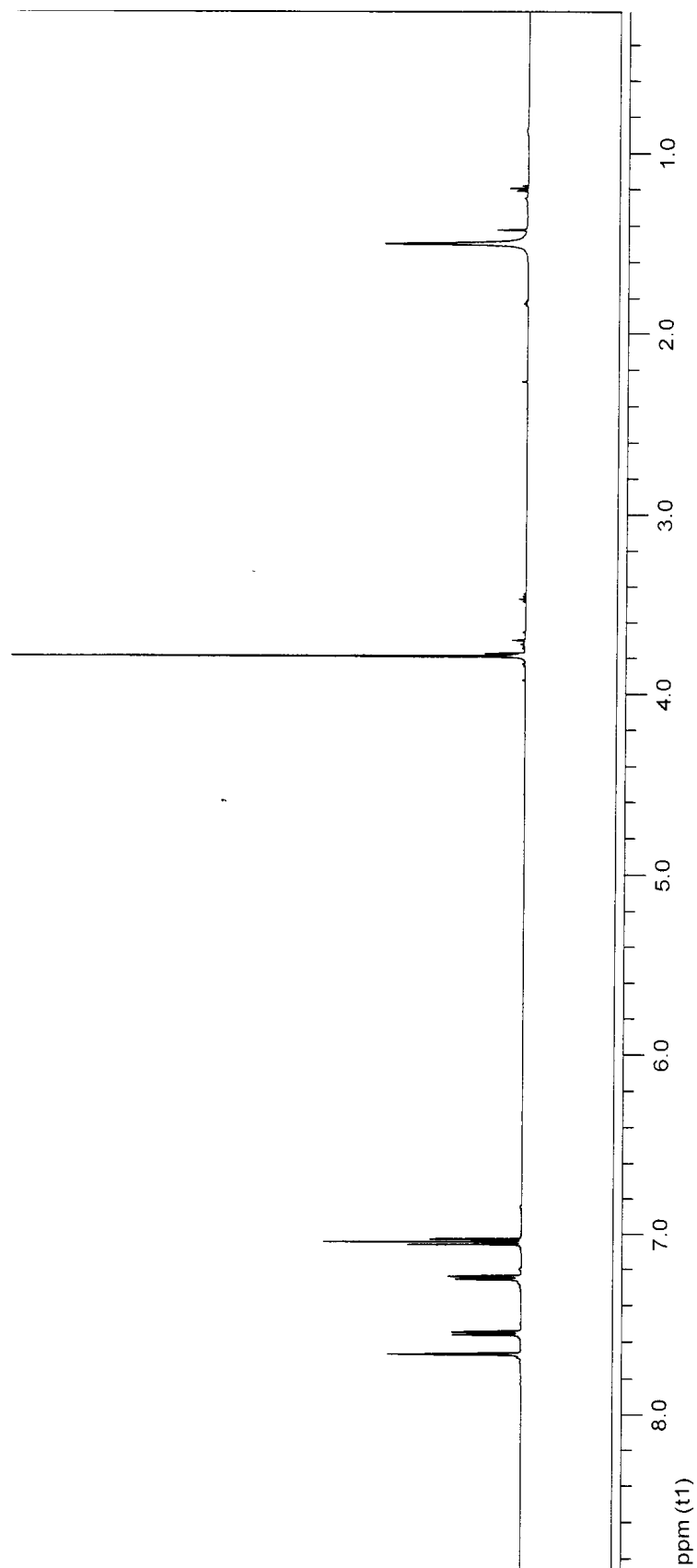


Table 1 Entry 7
 ^1H -NMR (500 MHz, CDCl_3)



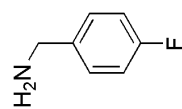
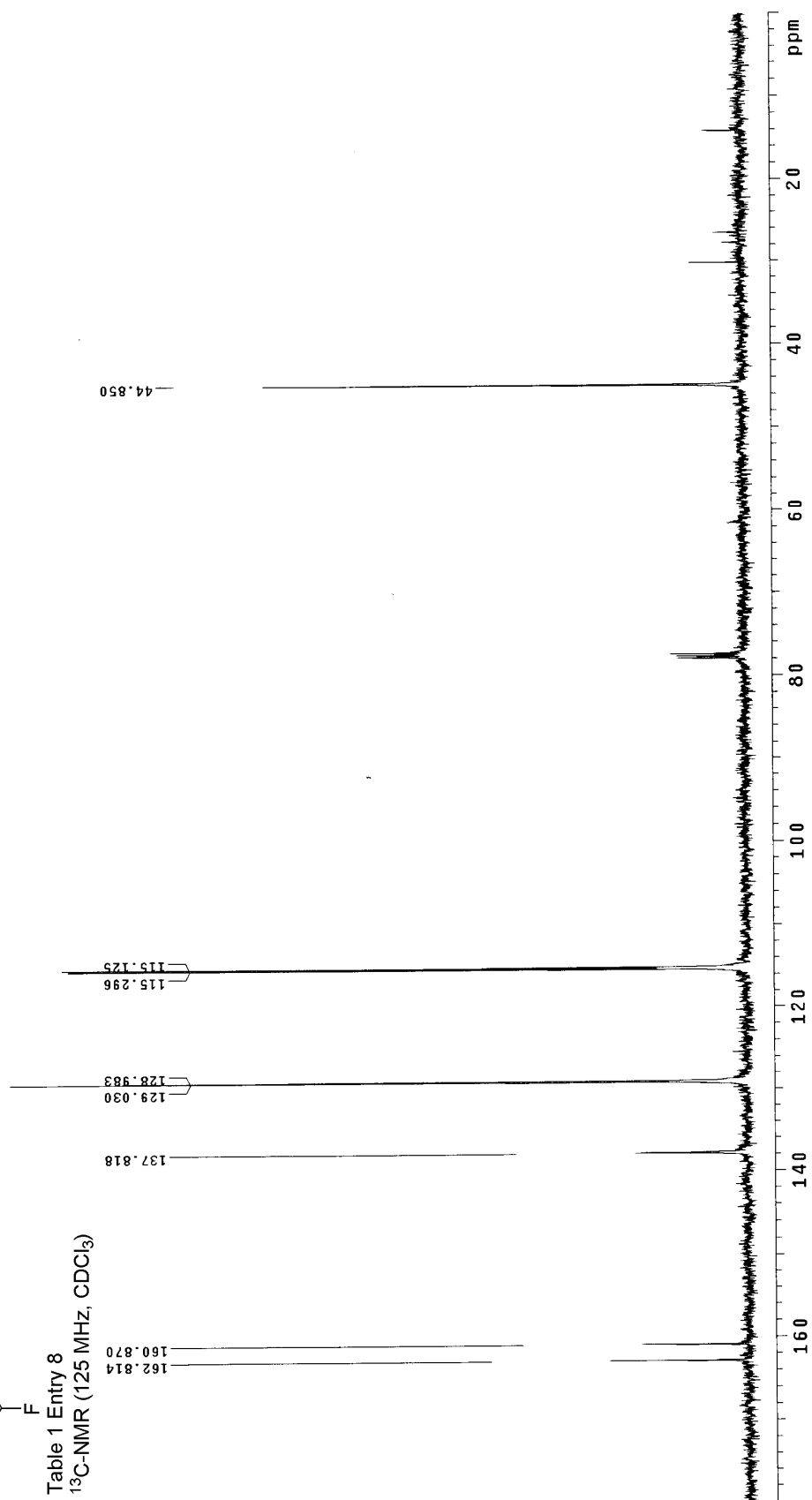


Table 1 Entry 8
¹³C-NMR (125 MHz, CDCl₃)



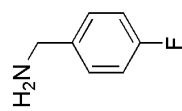
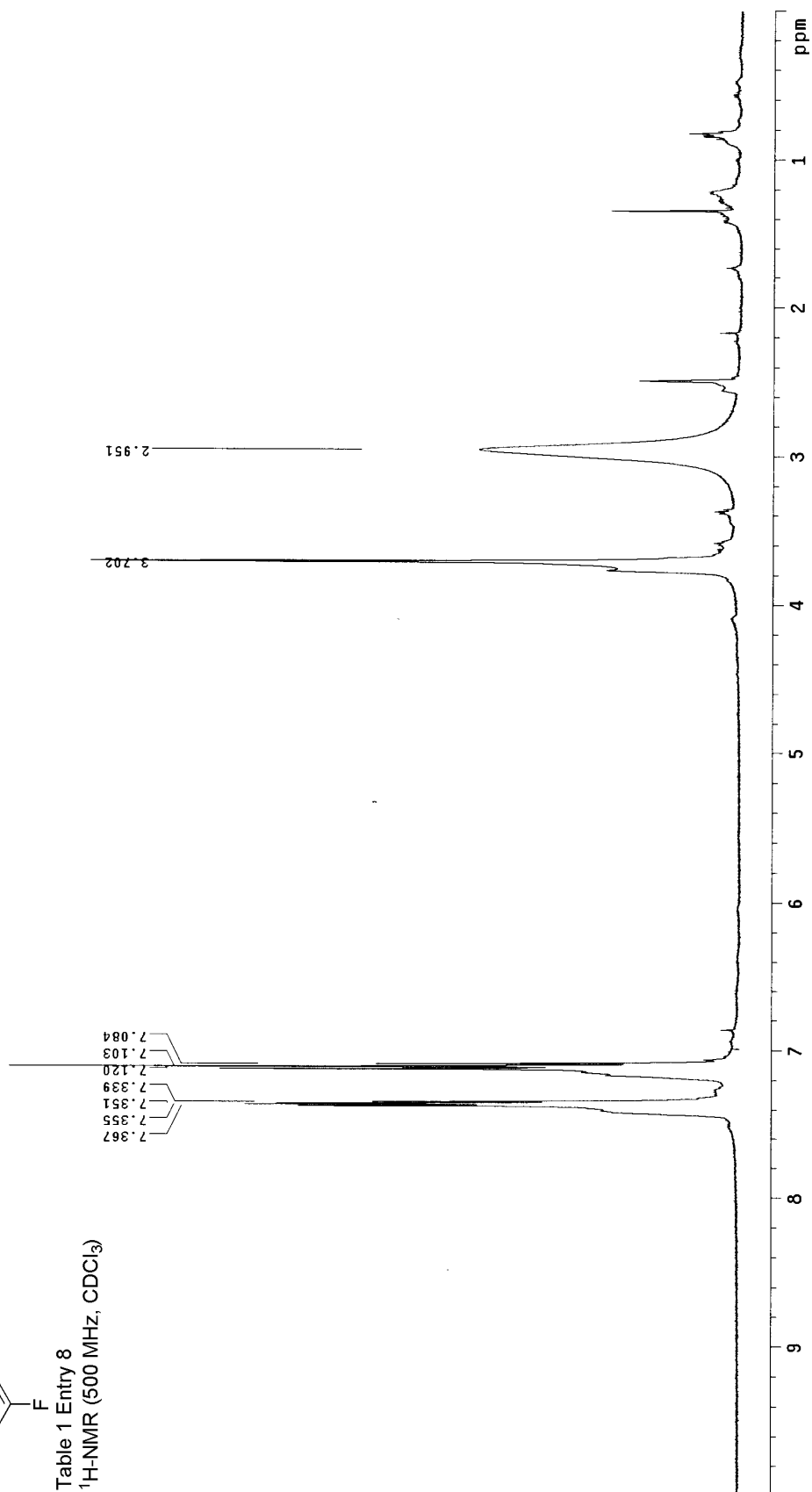


Table 1 Entry 8
¹H-NMR (500 MHz, CDCl₃)



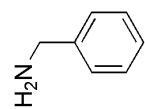
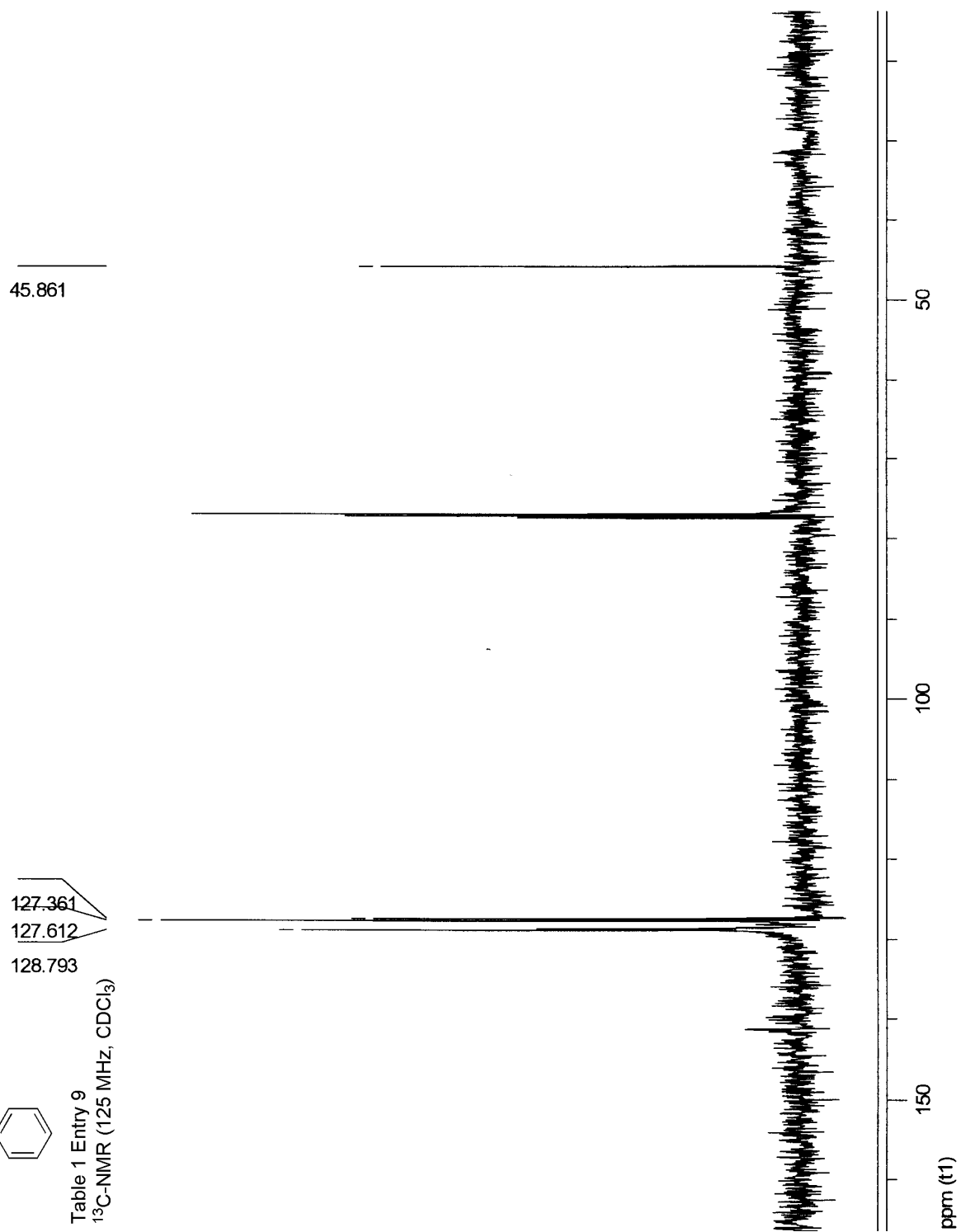


Table 1 Entry 9
¹³C-NMR (125 MHz, CDCl₃)



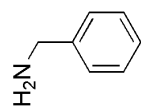
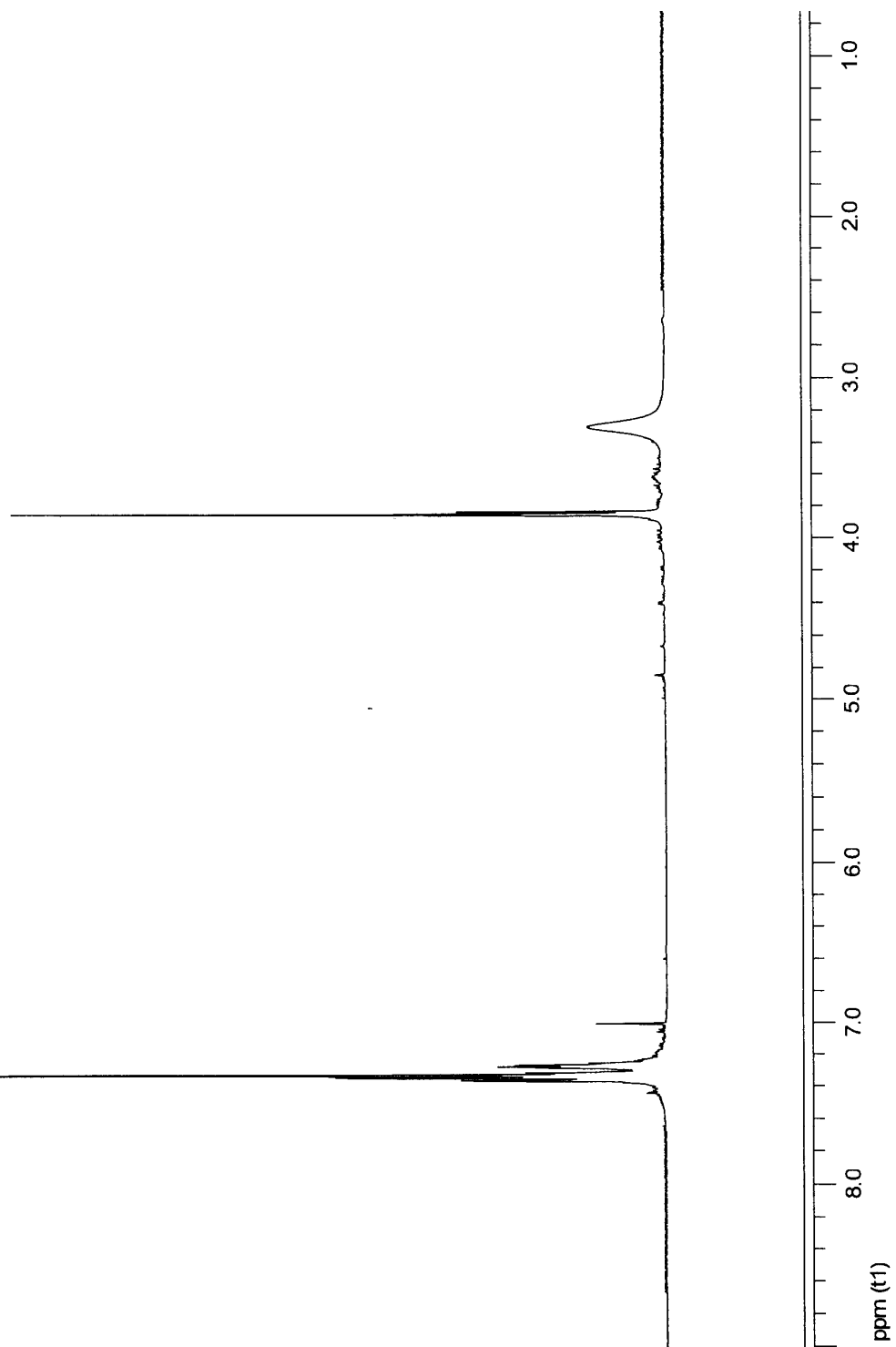


Table 1 Entry 9
 $^1\text{H-NMR}$ (500 MHz, CDCl_3)



45.920

64.386

127.254

128.417

140.480

141.543

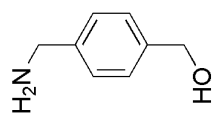


Table 1 Entry 10
¹³C-NMR (125 MHz, CDCl₃)

50

100

150

ppm (t1)

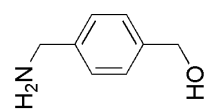
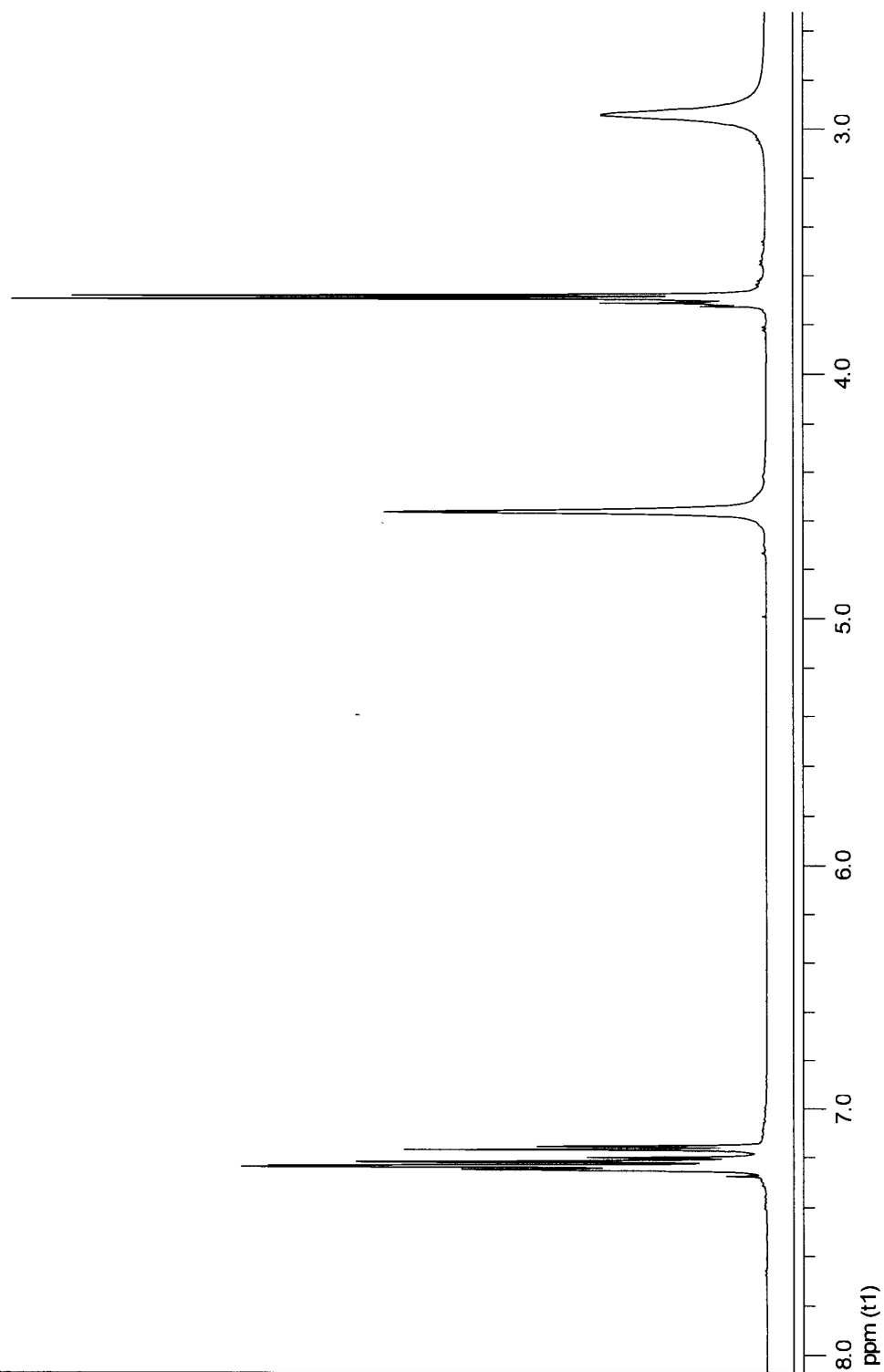


Table 1 Entry 10
 $^1\text{H-NMR}$ (500 MHz, CDCl_3)



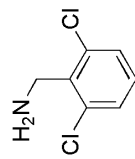
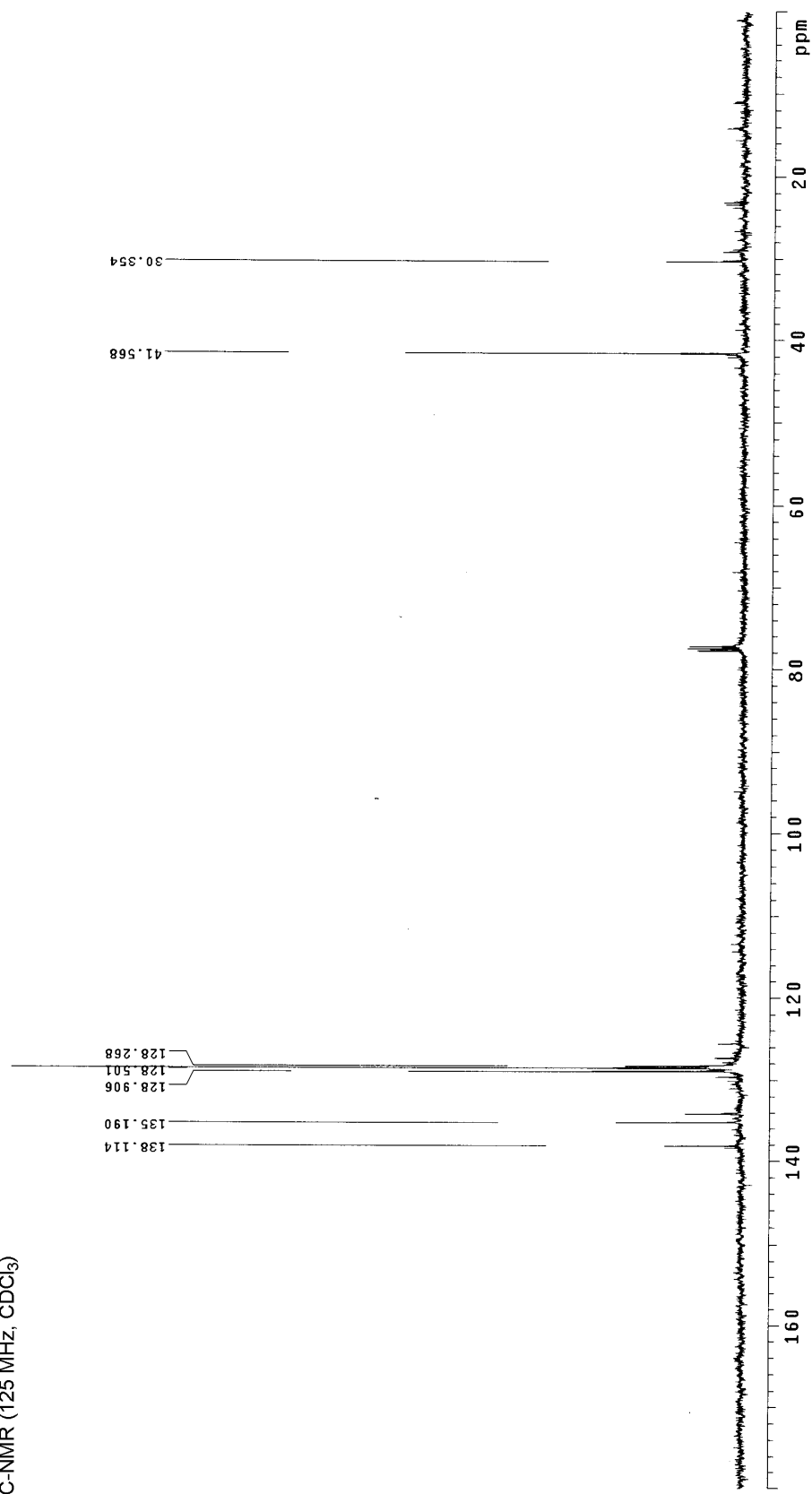


Table 2 Entry 1
¹³C-NMR (125 MHz, CDCl₃)



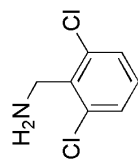
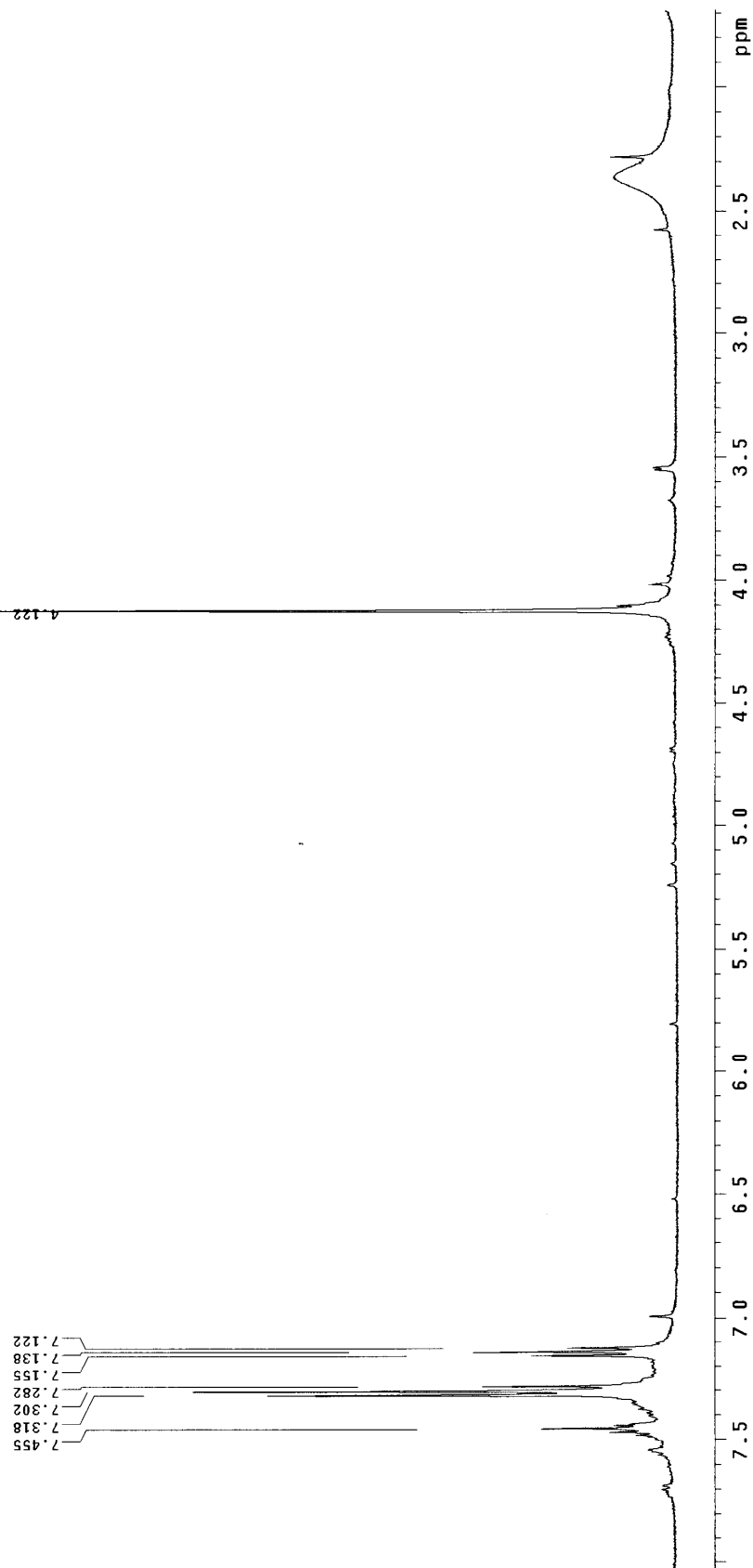


Table 2 Entry 1
¹H-NMR (500 MHz, CDCl₃)



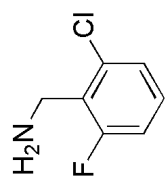
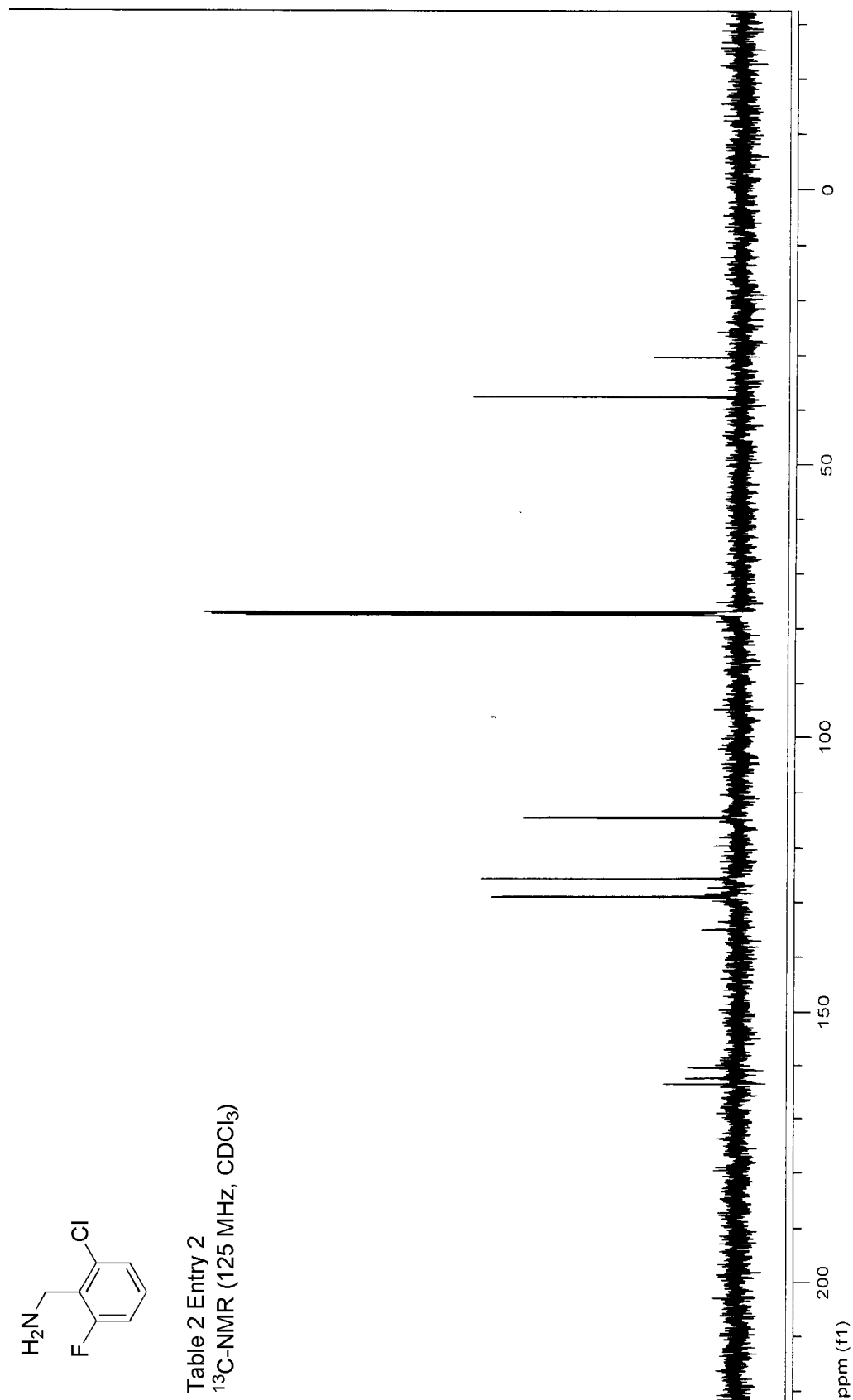


Table 2 Entry 2
¹³C-NMR (125 MHz, CDCl₃)



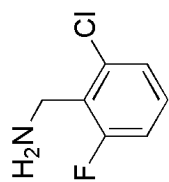
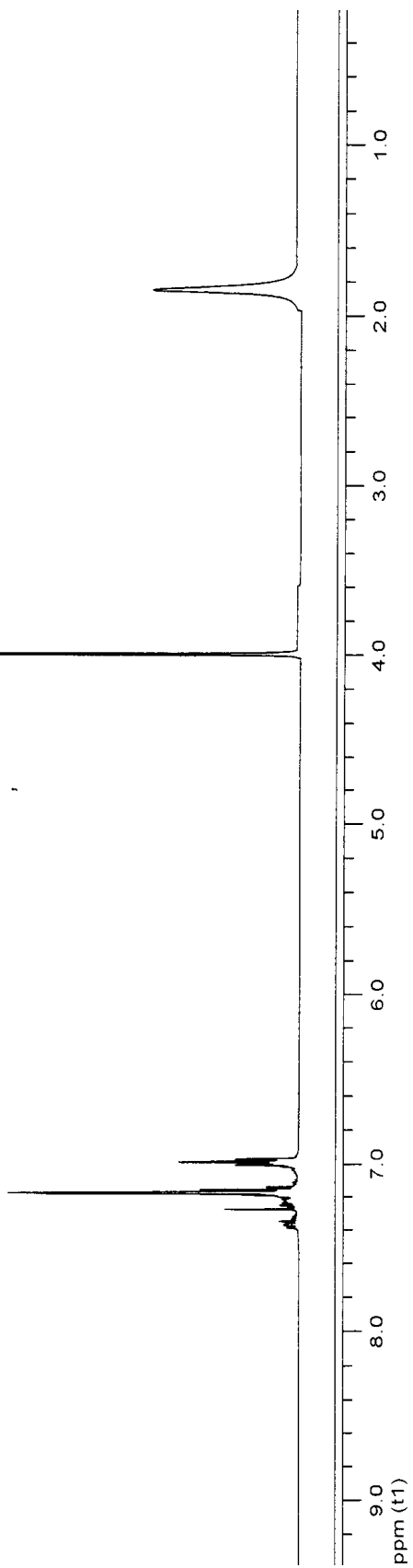


Table 2 Entry 2
¹H-NMR (500 MHz, CDCl₃)



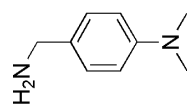
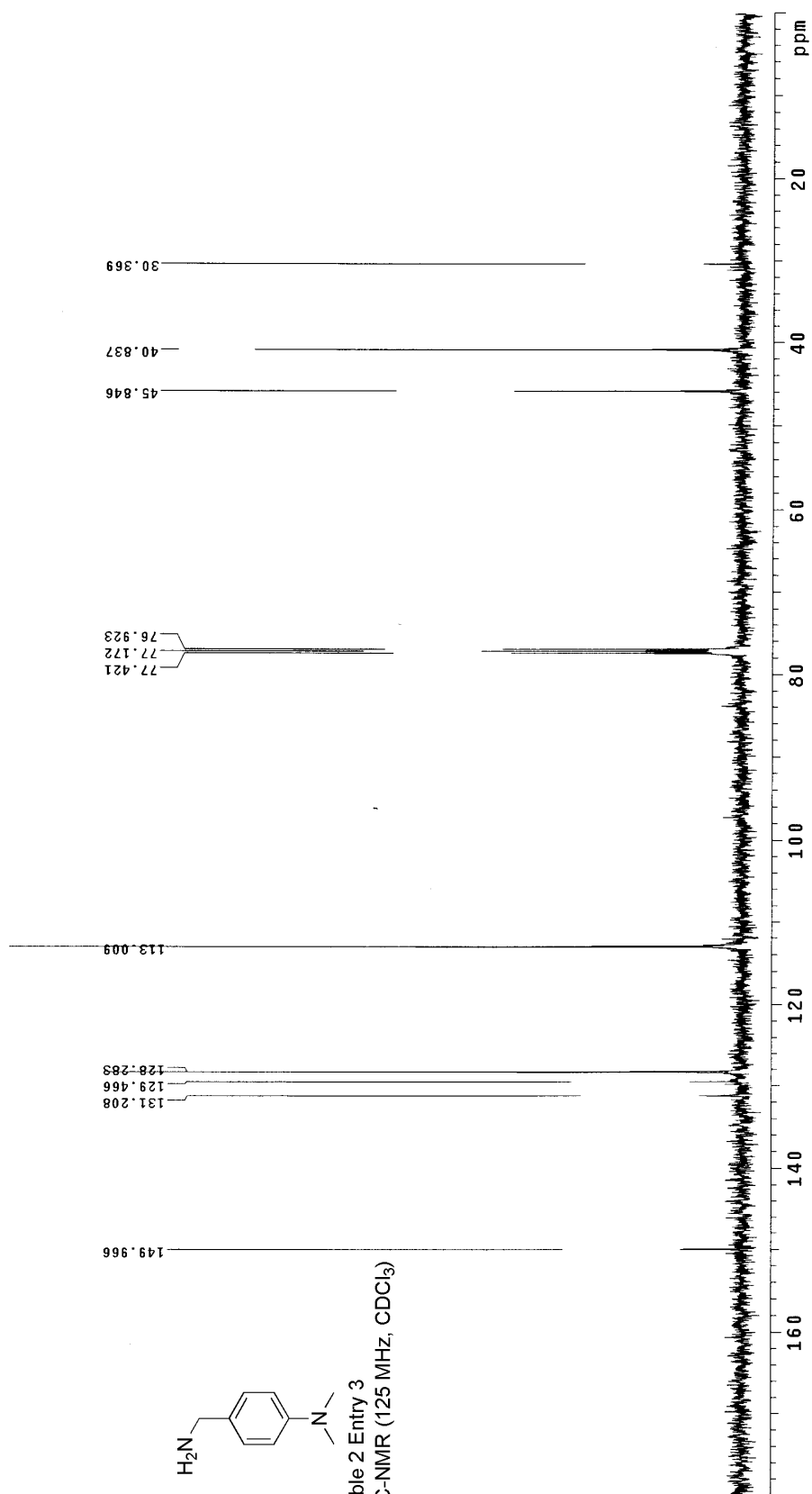


Table 2 Entry 3
¹³C-NMR (125 MHz, CDCl₃)



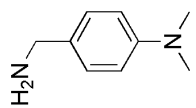
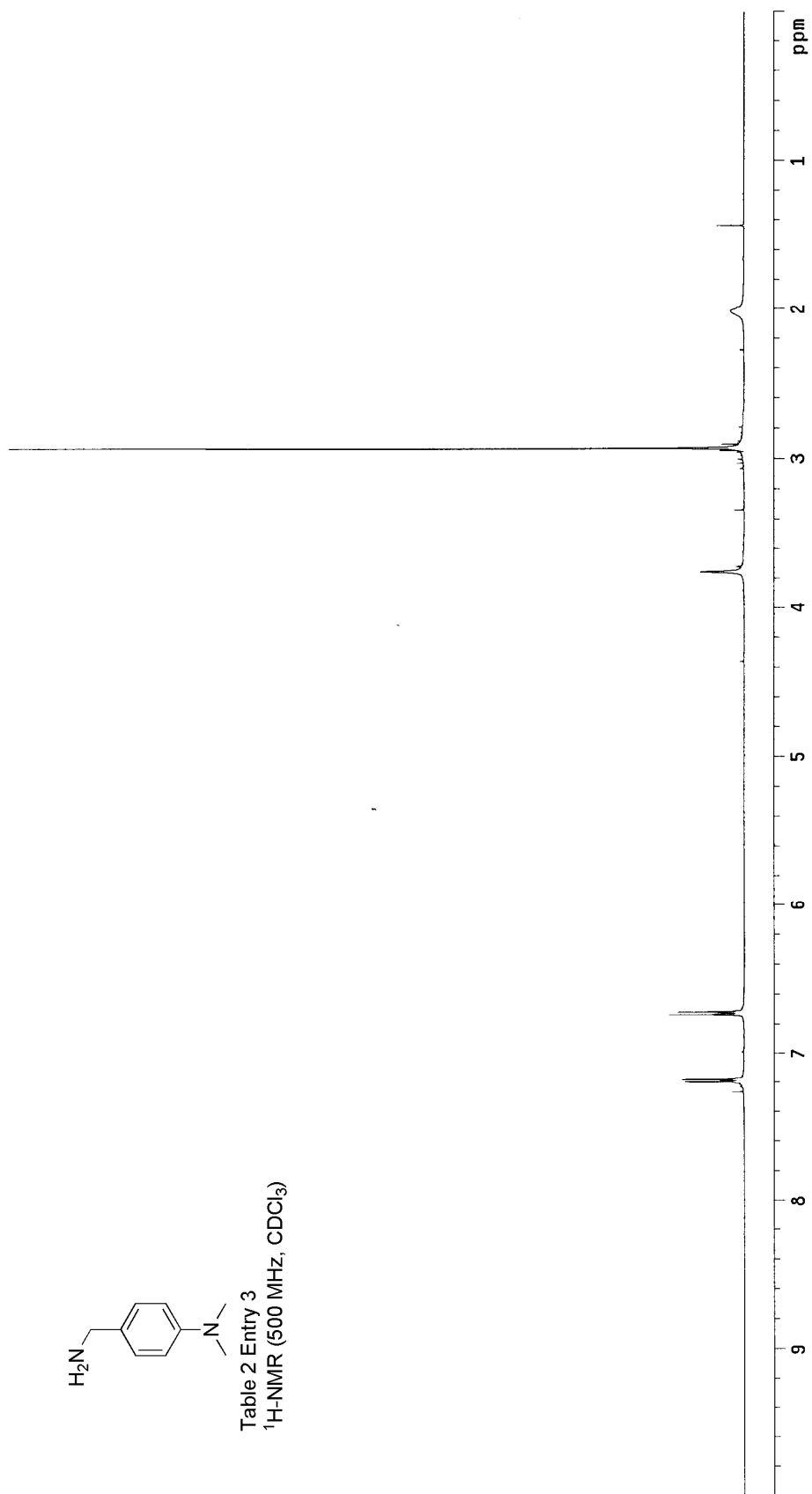


Table 2 Entry 3
¹H-NMR (500 MHz, CDCl₃)



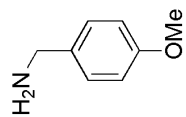
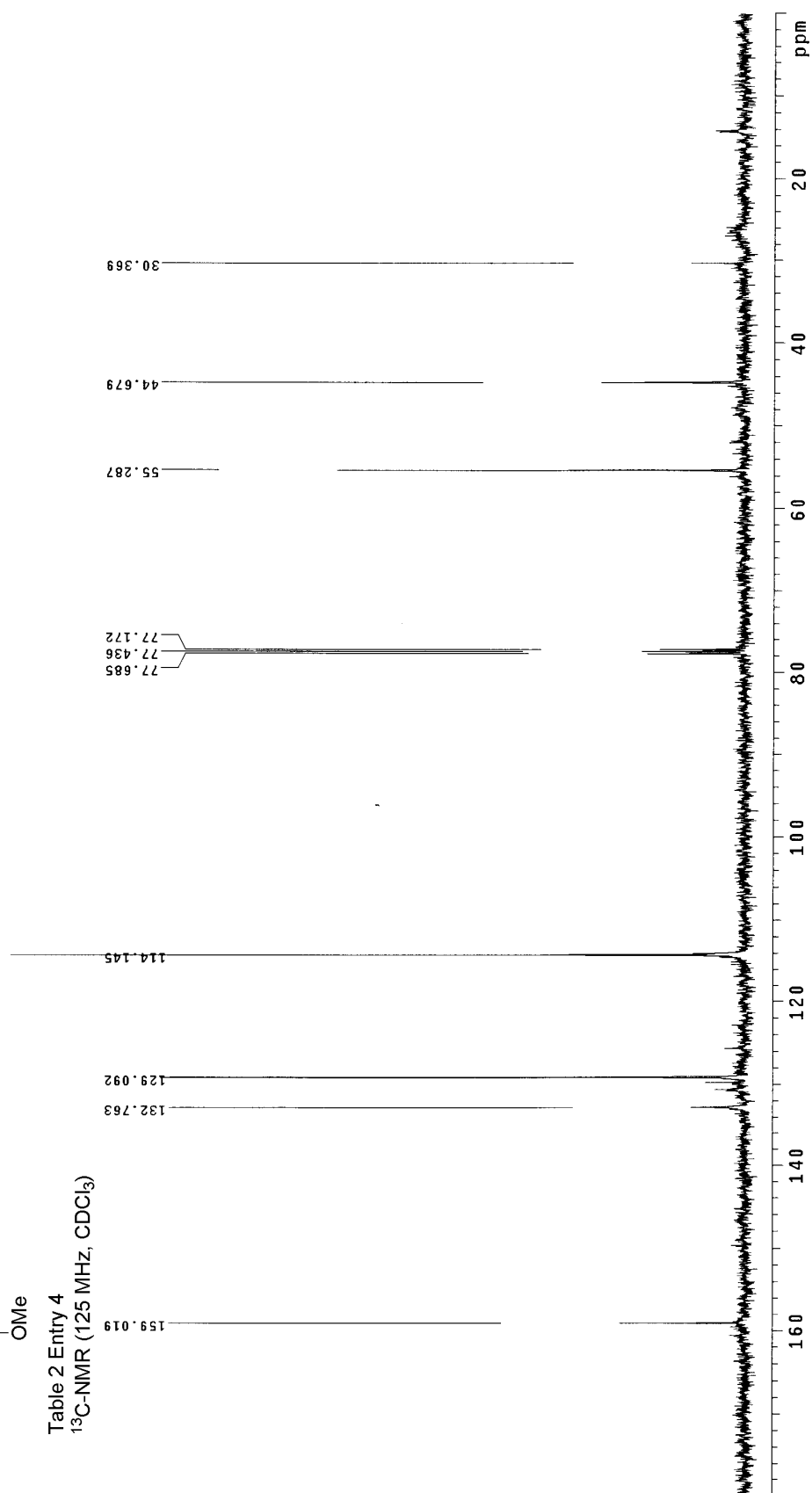


Table 2 Entry 4
¹³C-NMR (125 MHz, CDCl₃)



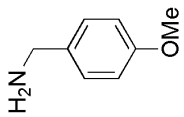
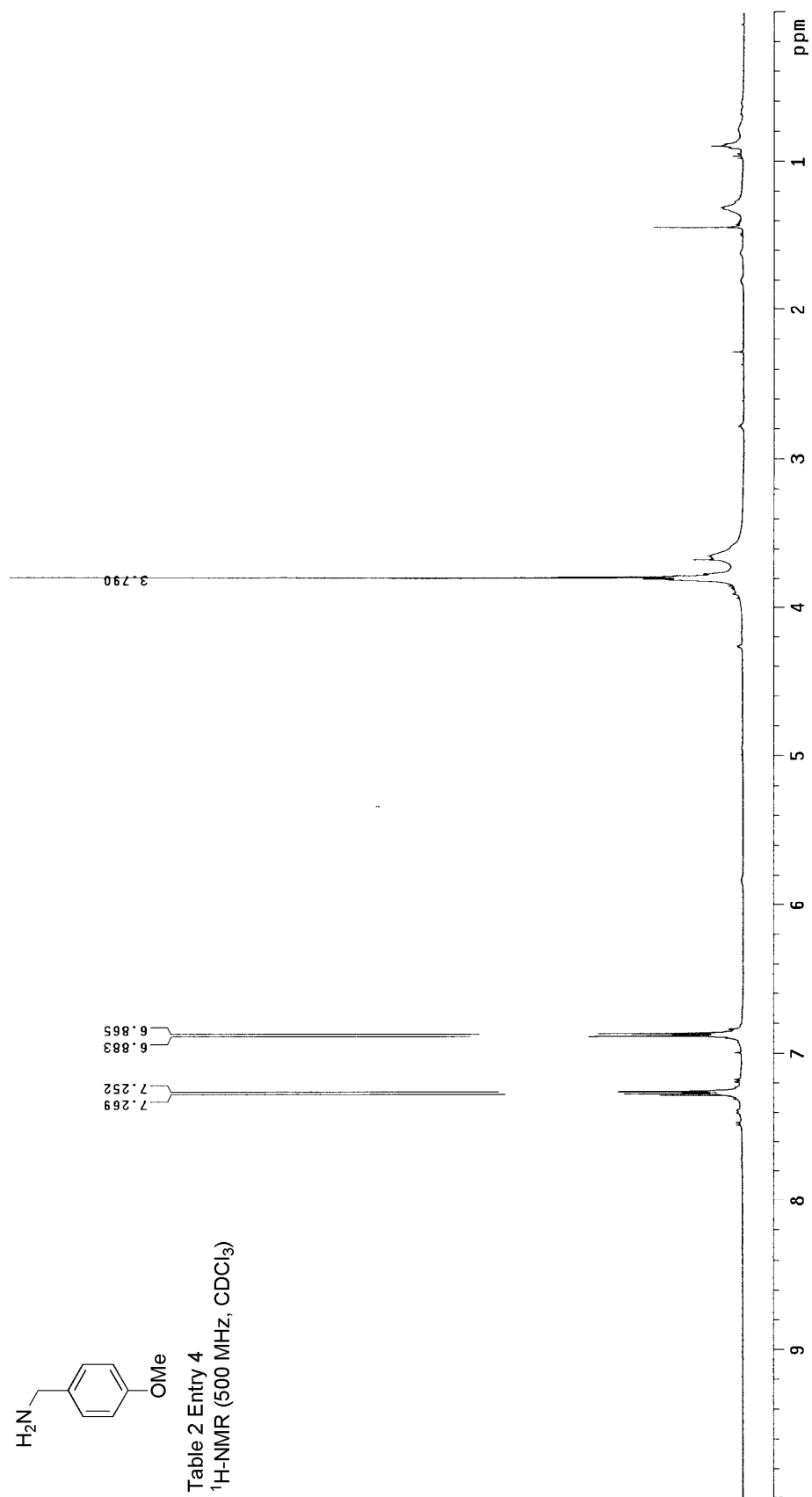
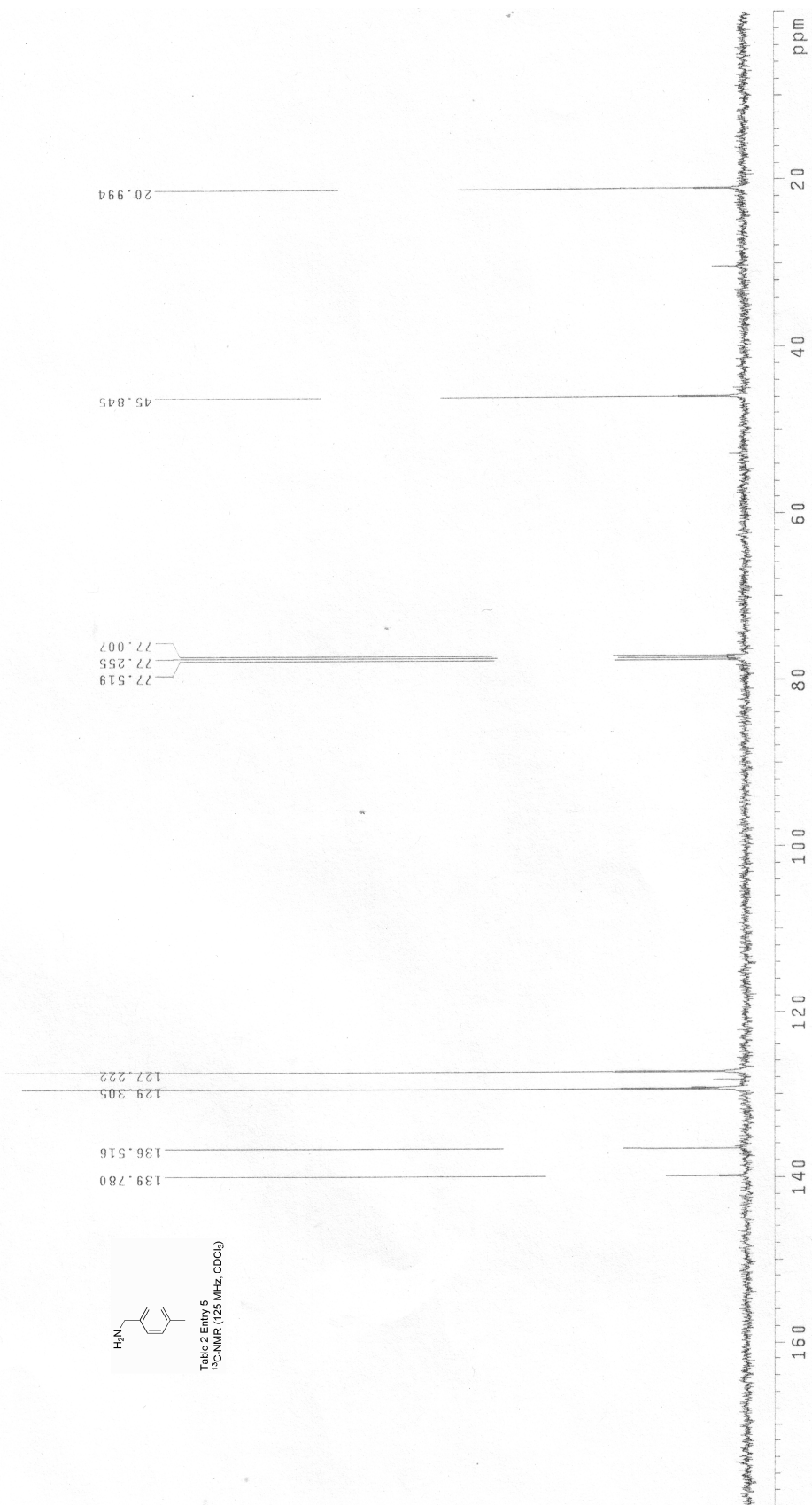
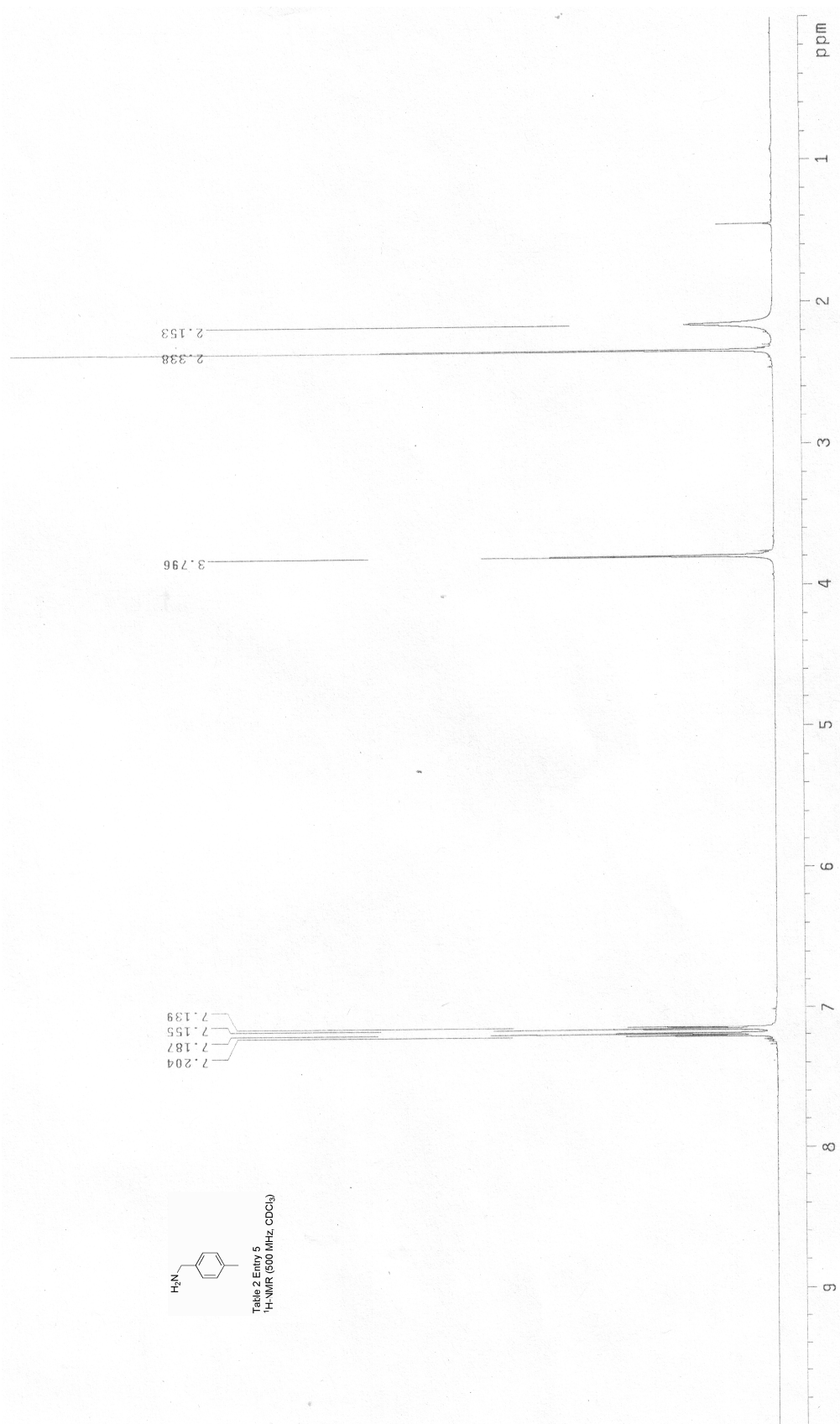


Table 2 Entry 4
¹H-NMR (500 MHz, CDCl₃)







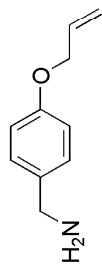
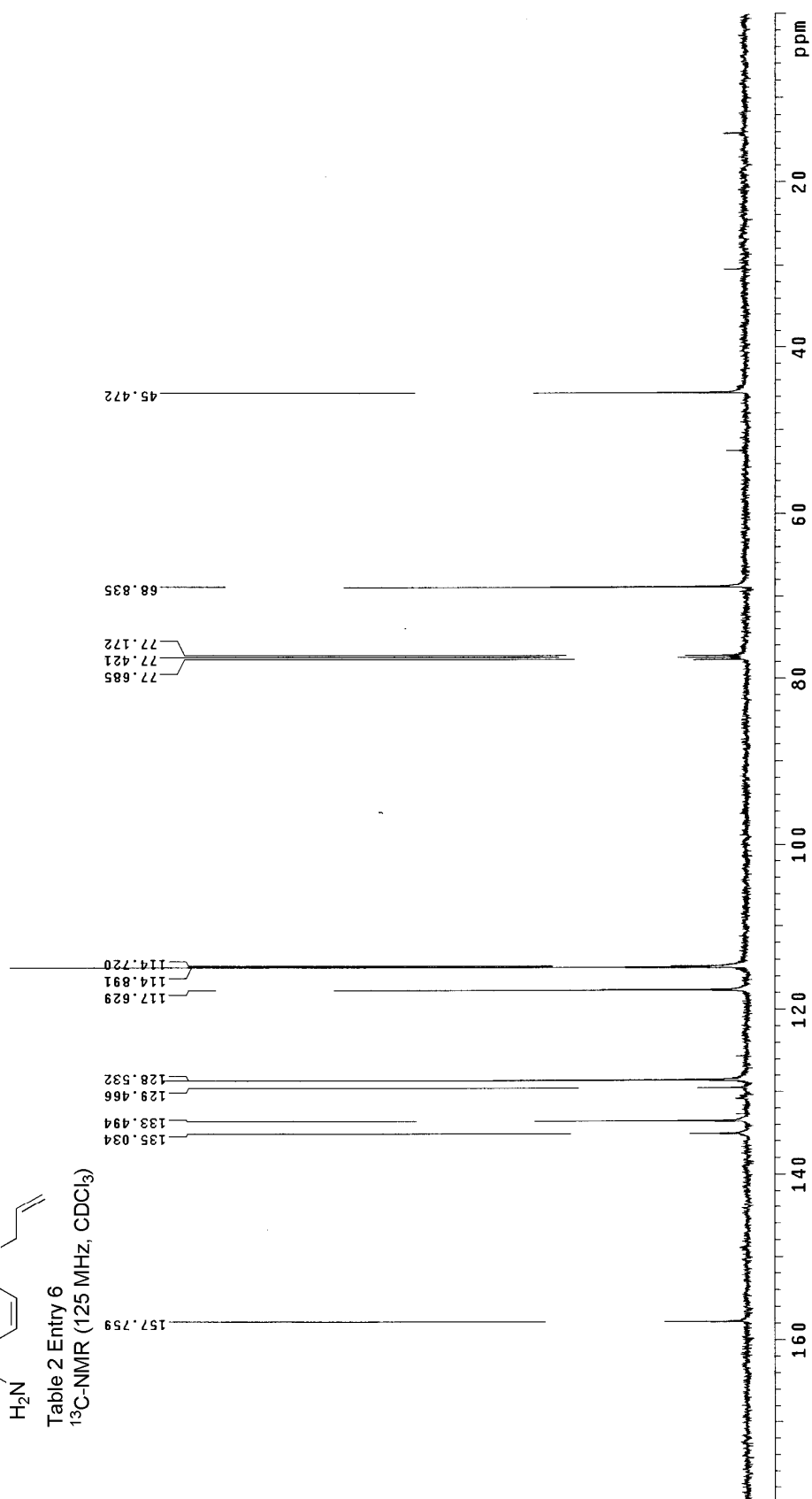


Table 2 Entry 6
¹³C-NMR (125 MHz, CDCl₃)



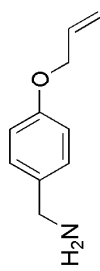
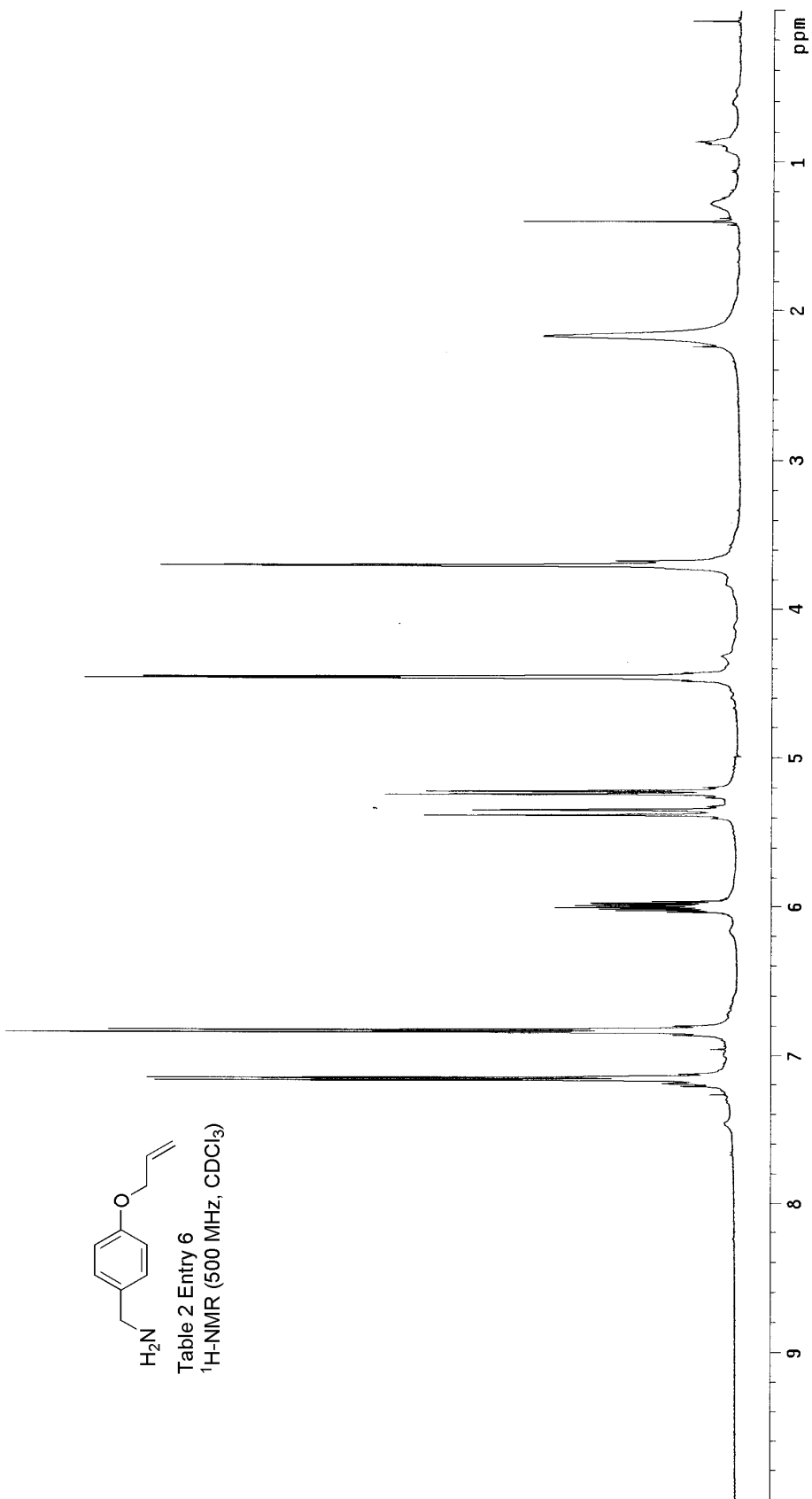
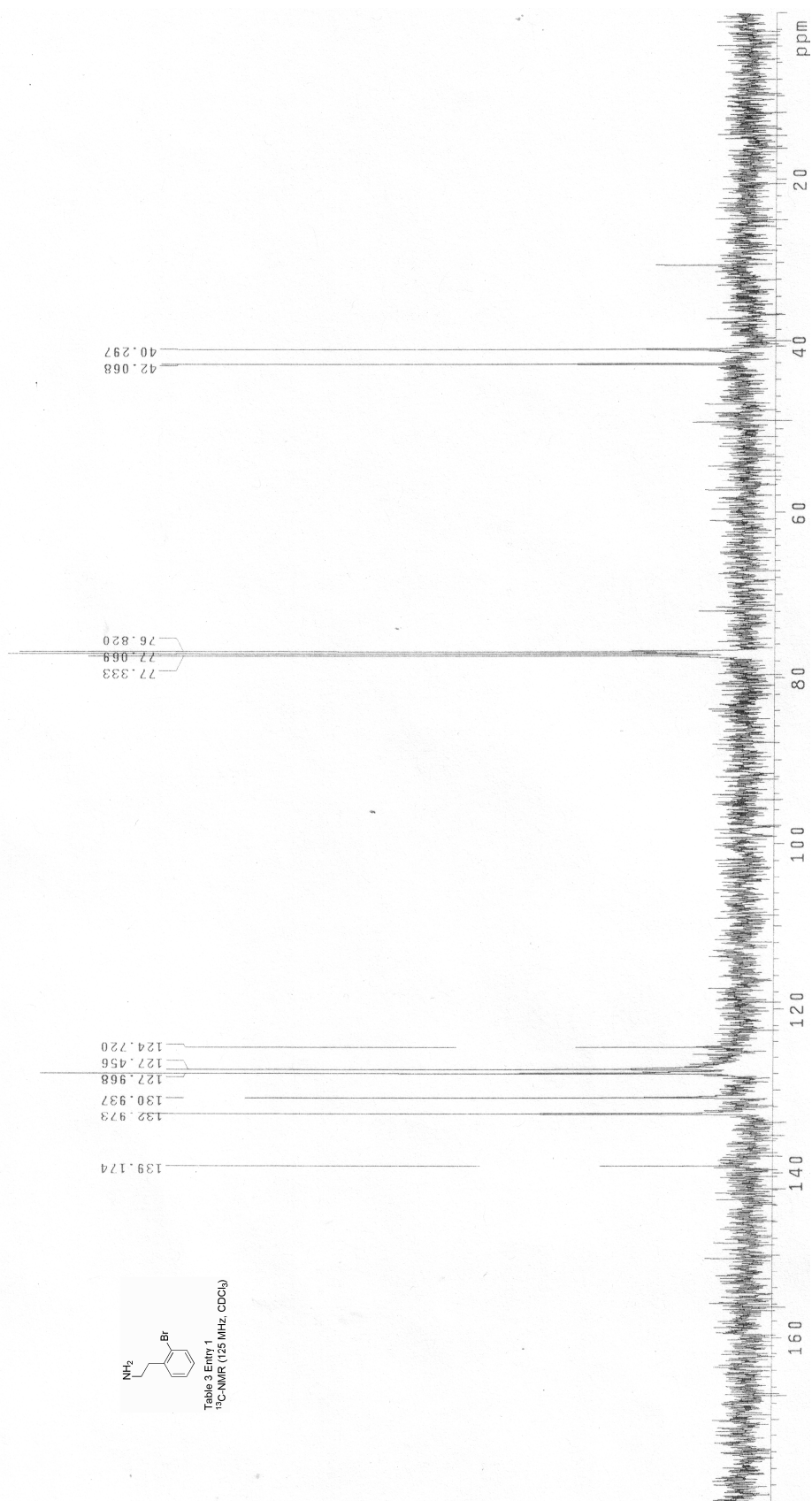
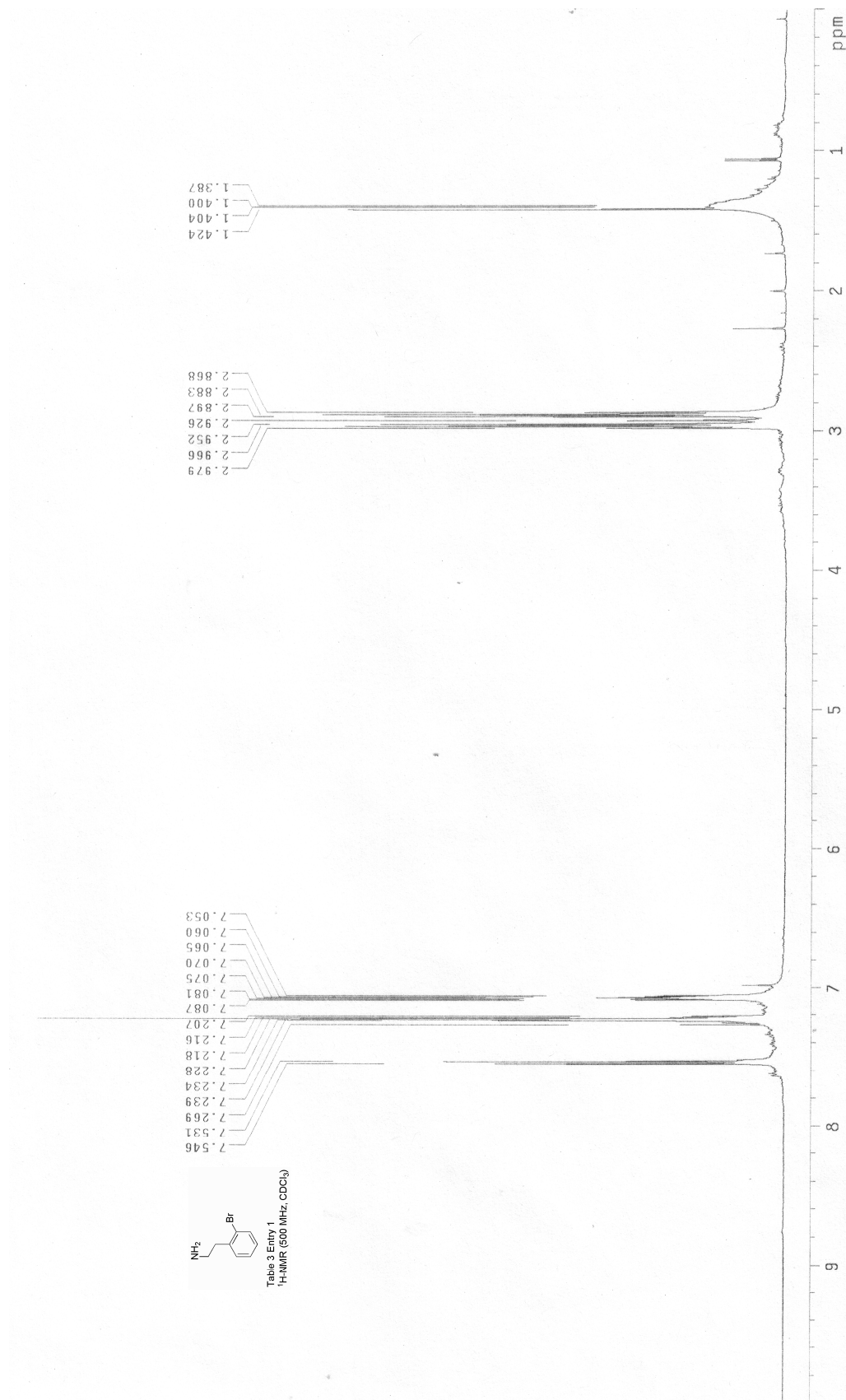
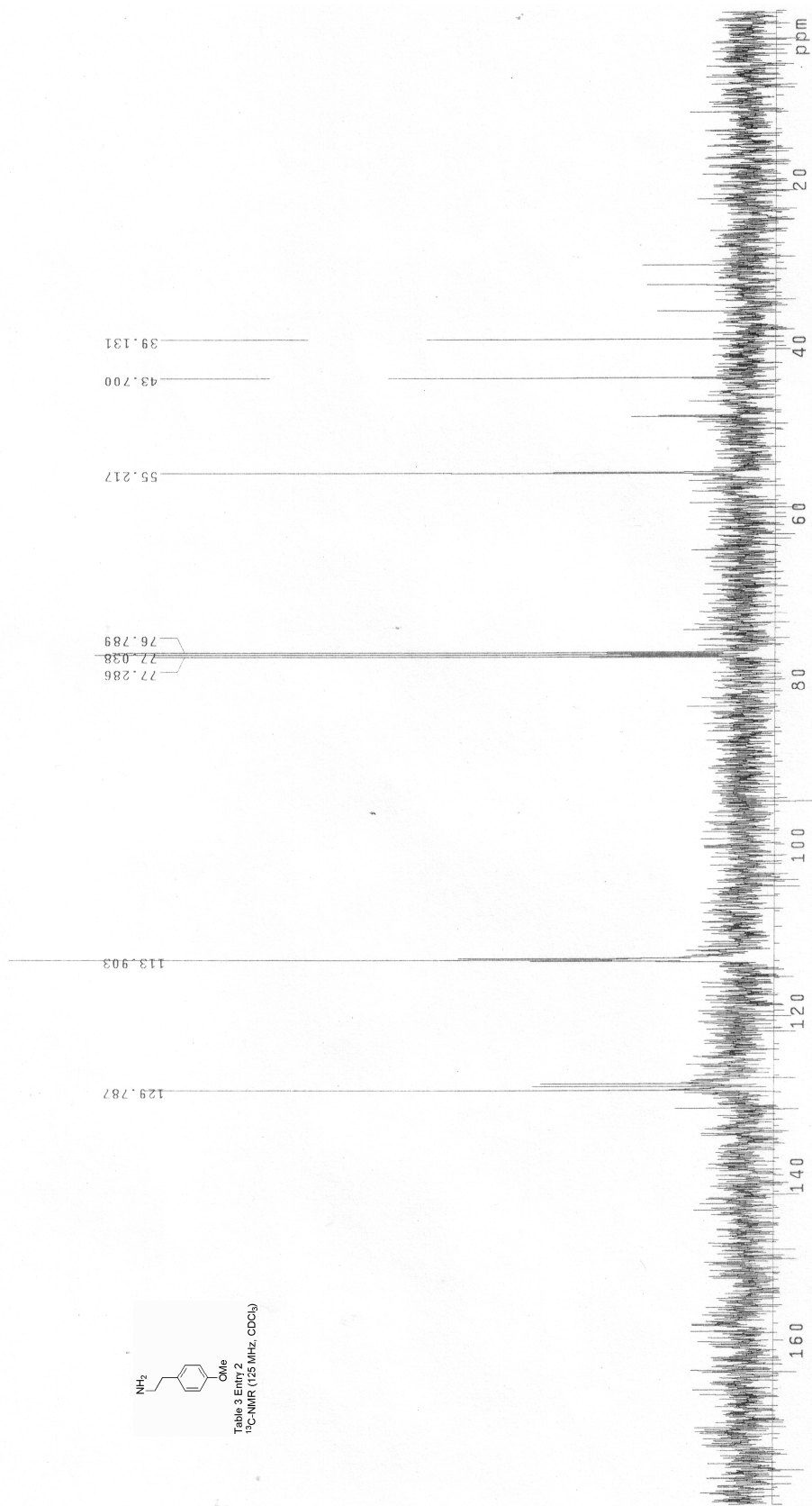


Table 2 Entry 6
¹H-NMR (500 MHz, CDCl₃)









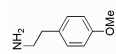
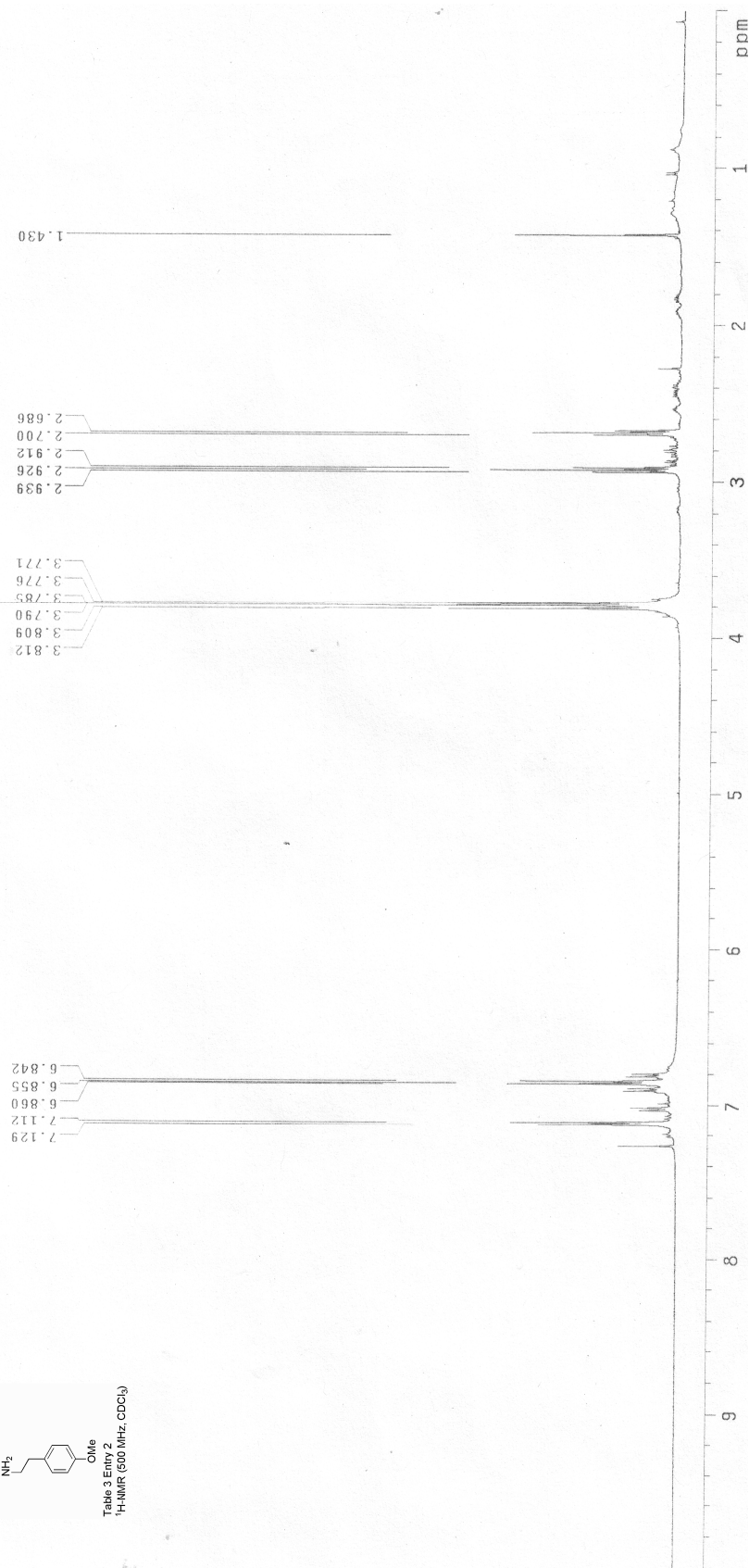


Table 3 Entry 2
¹H-NMR (500 MHz, CDCl₃)



phenethylamine oxalate
500 MHz MeOH

Pulse Sequence: s2pu1

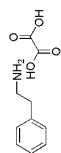
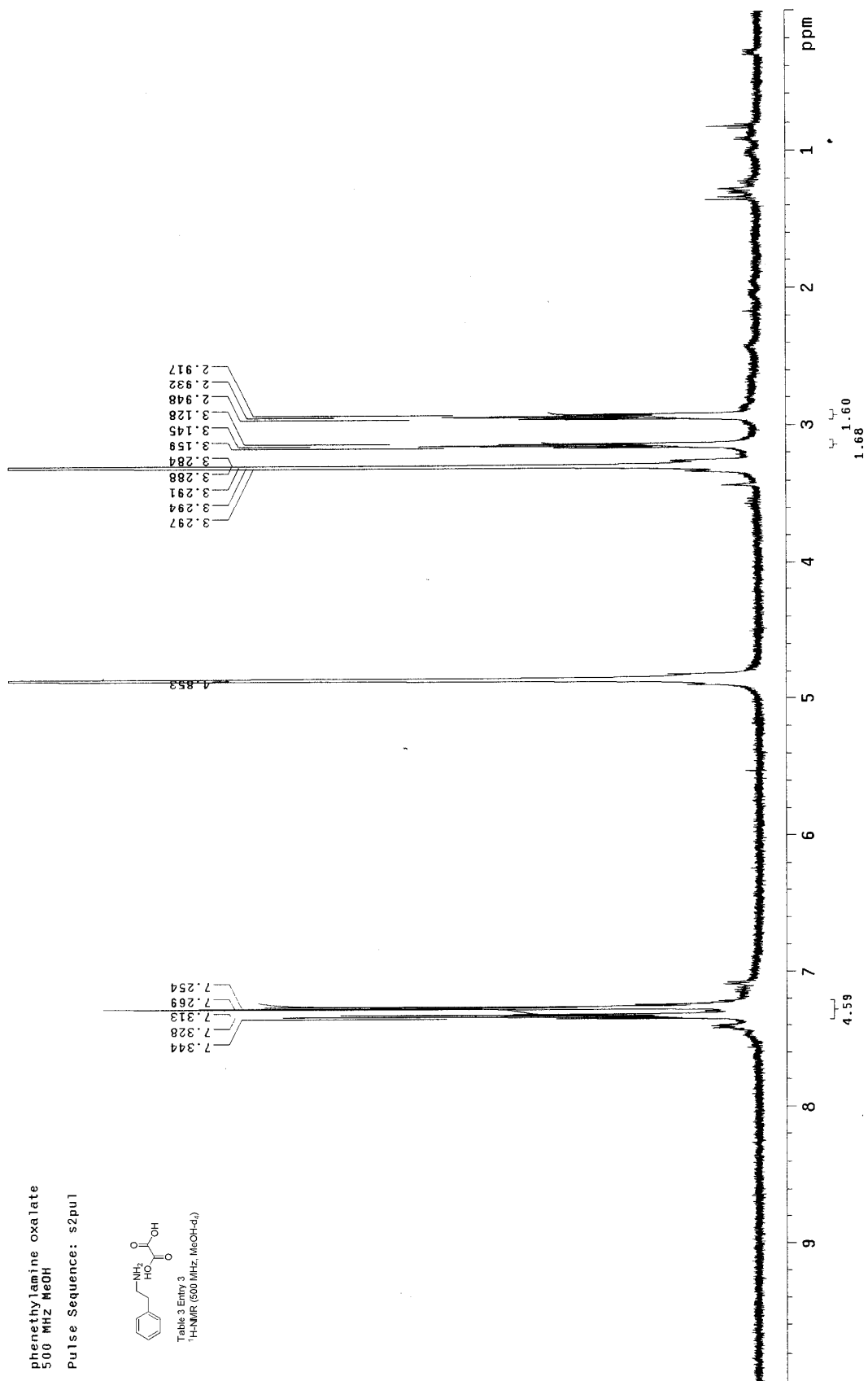
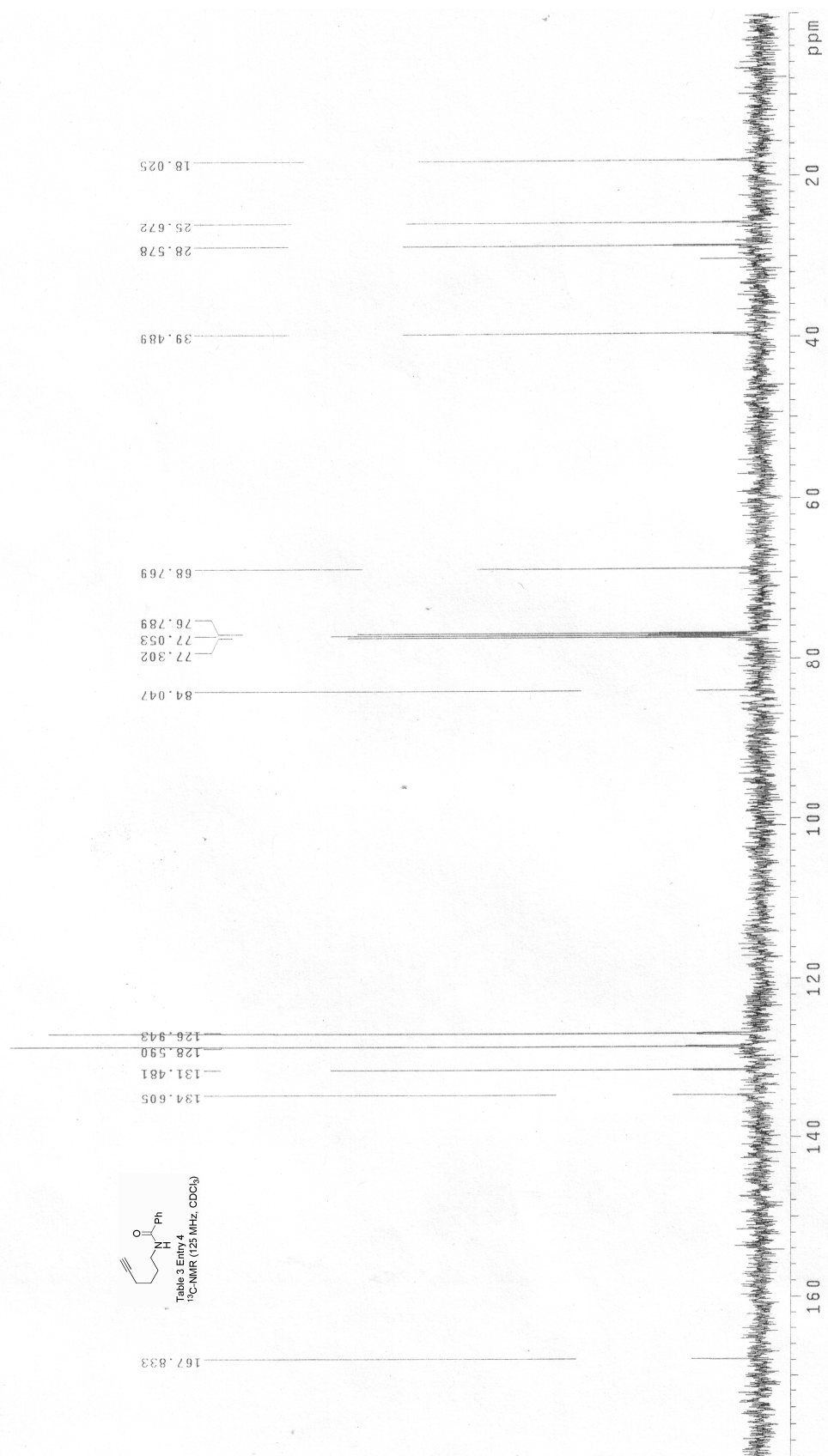
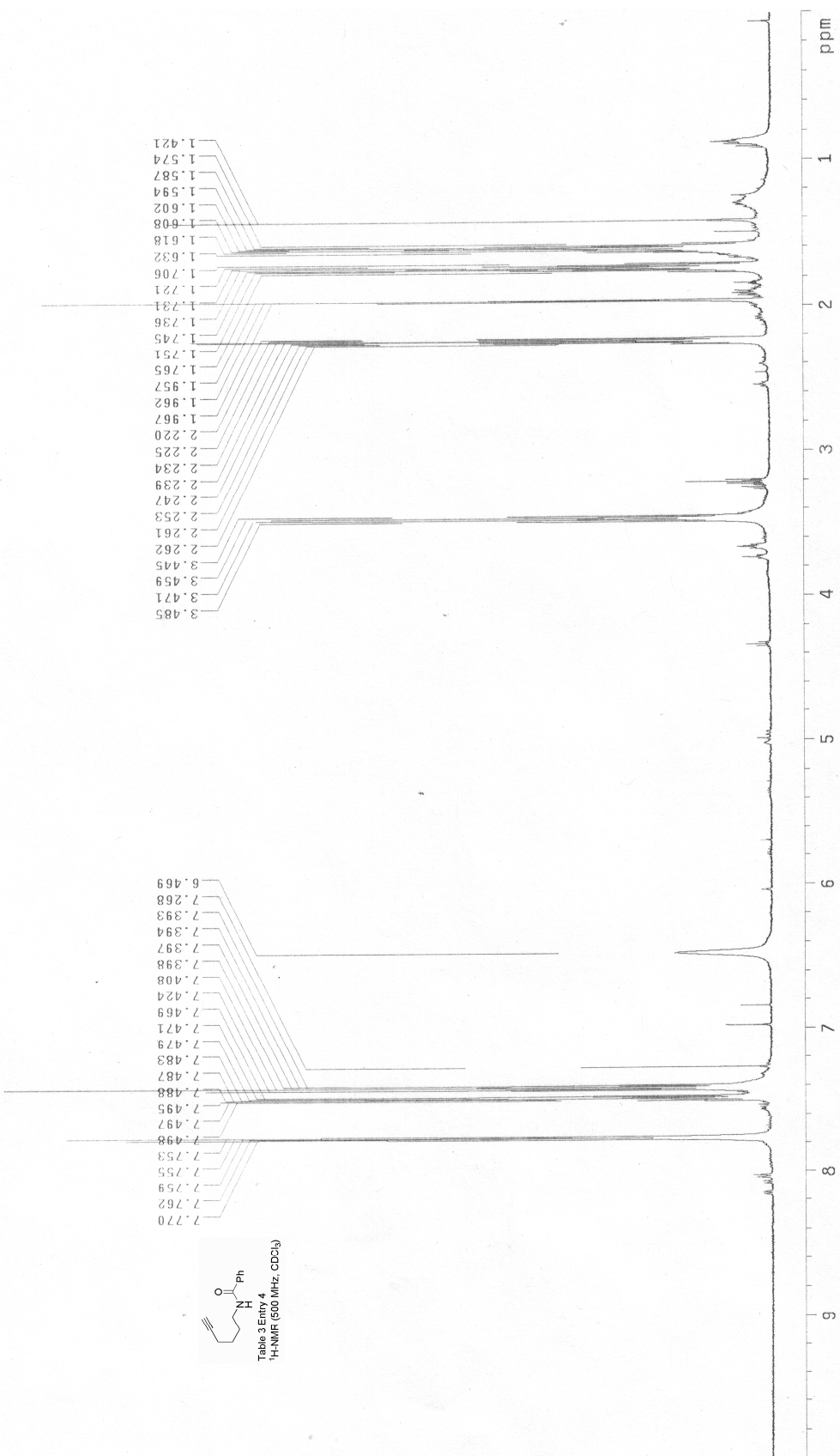


Table 3 Entry 3
¹H-NMR (500 MHz, MeOH-d₄)







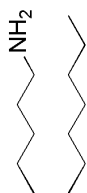
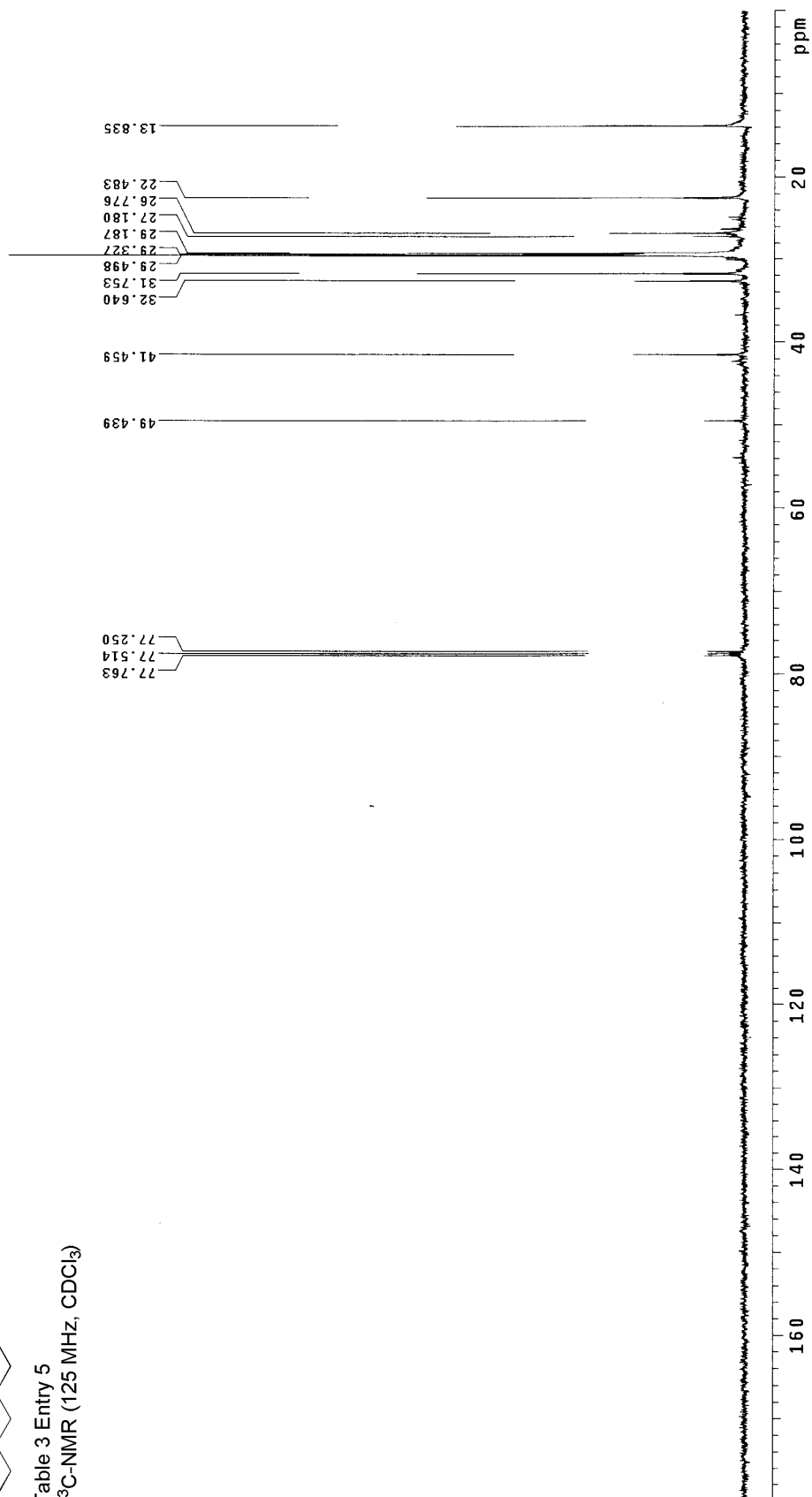


Table 3 Entry 5
³C-NMR (125 MHz, CDCl₃)



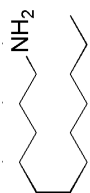
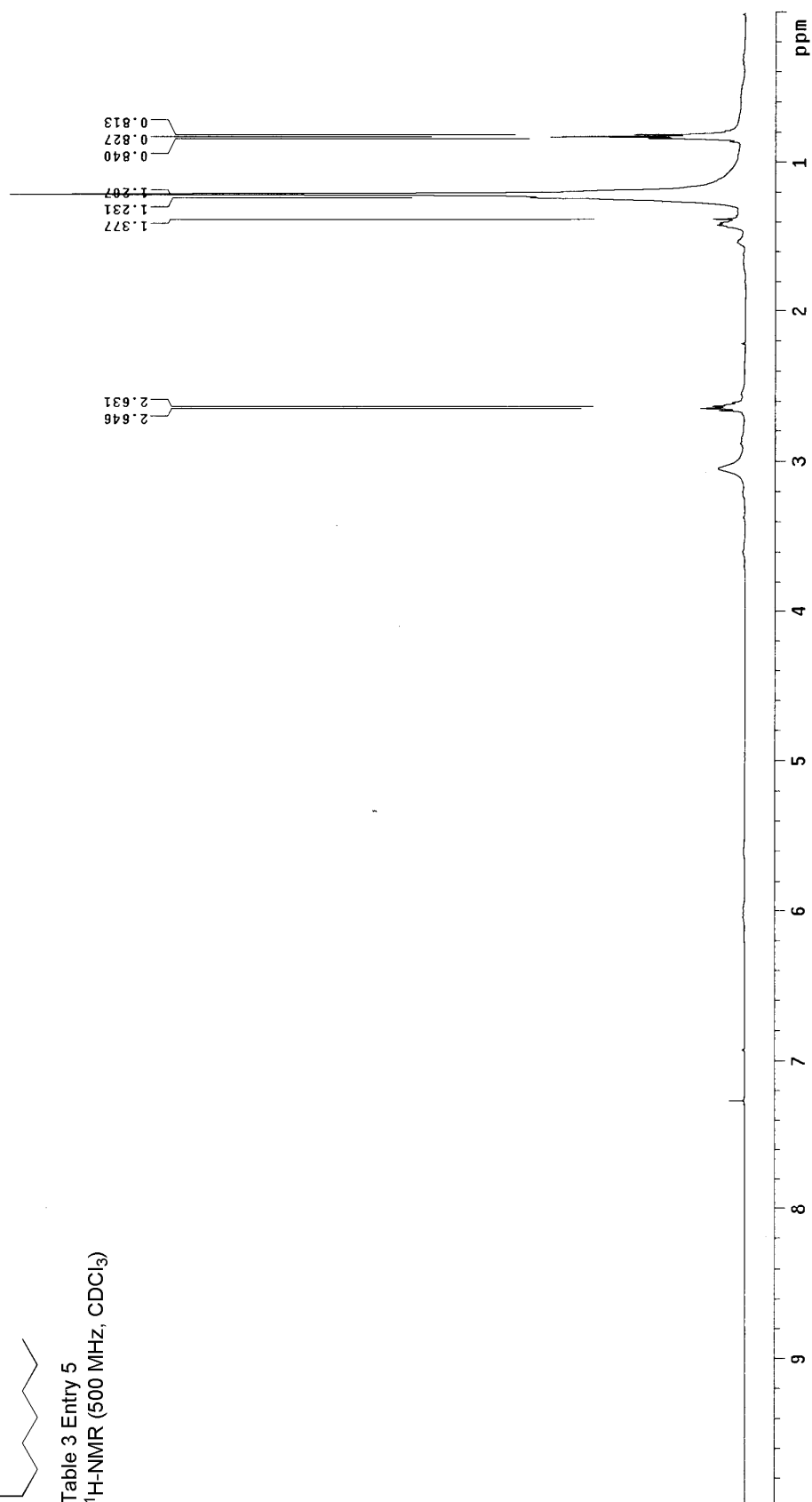


Table 3 Entry 5
¹H-NMR (500 MHz, CDCl₃)



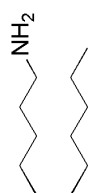


Table 3 Entry 6
 ^{13}C -NMR (125 MHz, CDCl_3)

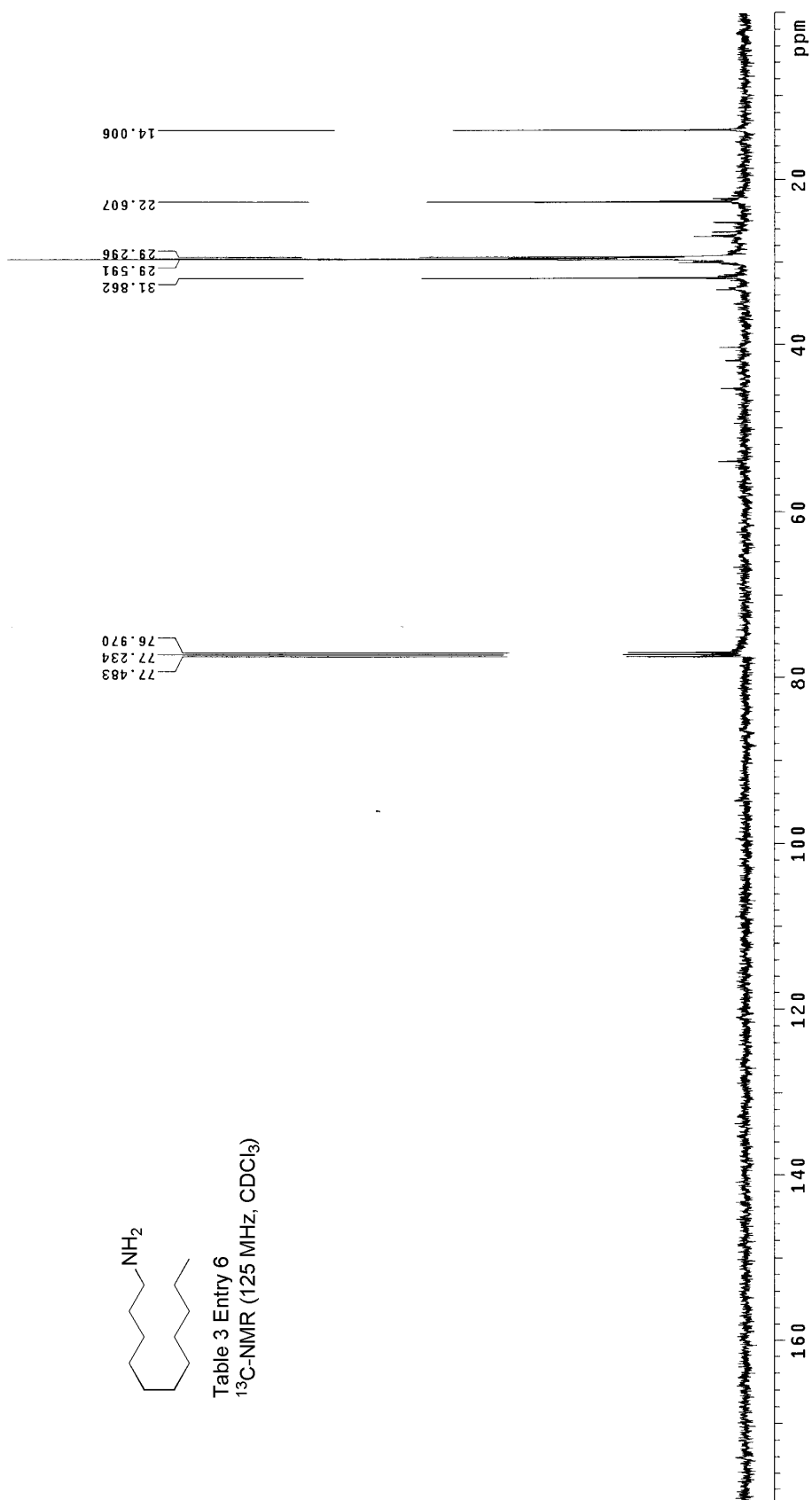




Table 3 Entry 6
¹H-NMR (500 MHz, CDCl₃)

