Supporting Information for

Constructions of Tetrahydro-γ-carboline Skeletons via Intramolecular Oxidative Carbon-Carbon Bond Formation of Enamines

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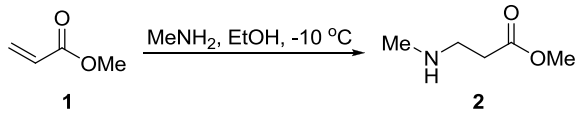
Supplementary Material

General Information

^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer at 25 ^{o}C. Chemical shifts values are given in ppm and referred as the internal standard to TMS: 0.00 ppm. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quadruplet; m, multiplet; br, broad and dd, doublet of doublets. The coupling constants J, are reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained on a Q-TOF micro spectroMeter. Melting points were determined with a national micromelting point apparatus without corrections. TLC plates were visualized by exposure to ultraviolet light. Dichloromethane was dried by CaH_2 before use, other reagents and solvents were purchased as reagent grade and were used without further purification. Flash column chromatography was performed over silica gel 200-300 m and the eluent was a mixture of ethyl acetate (EA) and petroleum ether (PE), or a mixture of methanol (M) and dichloromethane (D).

Experimental section

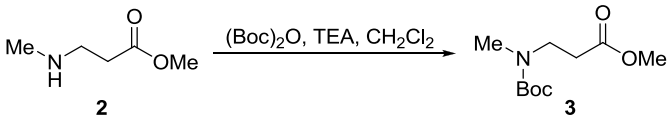
Methyl 3-(methylamino)propanoate (2)



1 2

Compound **2** was obtained according to published procedure.[1] Yield: 86%, colorless oil.

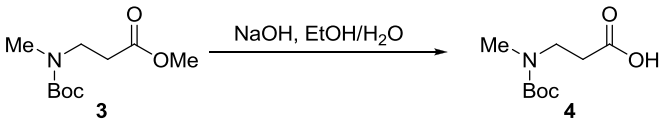
Methyl 3-[(*tert*-butoxycarbonyl)(methyl)amino]propanoate (3)[2]



2 3

To a stirred solution of **2** (9.4 g, 80 mmol) in dry CH_2Cl_2 (200 mL), Boc_2O (19.0 g, 88 mmol) and Et_3N (22 mL, 160 mmol) were added. The resulted mixture was stirred at rt until TLC indicated the total consumption of **2**. To the reaction mixture was added saturated aq. NH_4Cl solution (100 mL) and stirred for another 30 min, CH_2Cl_2 was used to extract the mixture. The organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent, and the residue was purified by chromatography to afford the desired product **3** (17.4 g, 75 mmol). Yield: 94%, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.69 (s, 3H), 3.51 (t, *J* = 6.8 Hz, 2H), 2.87 (s, 3H), 2.55 (t, *J* = 6.8 Hz, 2H), 1.46(s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.9, 155.2, 79.3, 51.4, 44.9, 34.5, 32.9, 28.2.

3-[(*tert*-Butoxycarbonyl)(methyl)amino]propanoic acid (4)[3]

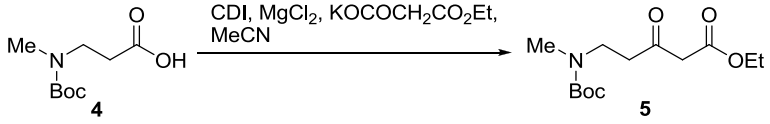


3 4

To a stirred solution of **3** (6.5 g, 30 mmol) in alcohol (150 mL) was added 50 mL aq. NaOH (1.8 g, 45 mmol) solution dropwise, the resulted mixture was stirred at rt until TLC indicated the total consumption of **3**. The mixture was evaporated to remove the alcohol, and the resulted solution was carefully acidified by aq. HCl (2 N) until the pH reached 3. EA was used to extract the mixture. The combined organic phase, after dried with anhydrous Na_2SO_4, was evaporated to

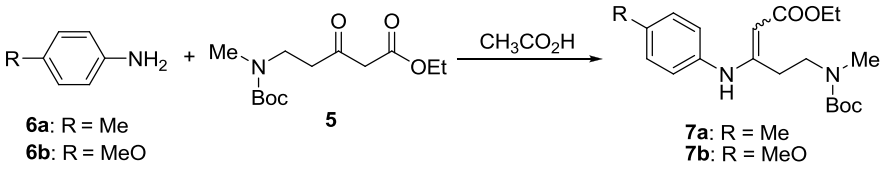
remove the solvent under vacuum and the residue was purified by chromatography to give the desired product **4** (5.8 g, 28 mmol). Yield: 95%, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.53 (t, *J* = 6.9 Hz, 2H), 2.89 (s, 3H), 2.61 (t, *J* = 6.8 Hz, 2H), 1.47 (s, 9H).

Eethyl 5-[methyl(*tert*-butoxycarbonyl)amino]-3-oxopentanoate (5)[4]



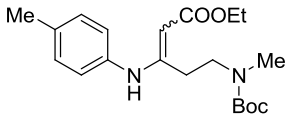
Under N_2, CDI (4.2 g, 26 mmol) was added to a stirred solution of **4** (4.9 g, 24 mmol) in anhydrous MeCN (120 mL) and the mixture was stirred at rt for 2 h. A solid mixture of anhydrous $MgCl_2$ (3.0 g, 31 mmol) and potassium monomethyl malonate (6.2 g, 36 mmol) was added, the mixture was stirred under N_2 at 40 ^{o}C until TLC indicated the total consumption of starting material. To the reaction mixture was added aq. HCl (2 N) dropwise until the pH reached 3. EA was used to extract the mixture. The combined organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent under vacuum and the residue was purified by column chromatography to afford the desired product **5** (6.0 g, 22 mmol). Yield: 92%, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.26-4.11 (m, 2H), 3.46 (m, 4H), 2.85-2.82 (m, 5H), 1.43 (s, 9H), 1.32-1.20 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 166.93, 155.48, 79.62, 61.36, 59.99, 49.45, 43.88, 41.48, 34.86, 28.36, 14.04.

General procedure for the preparation of 7a and 7b[5]



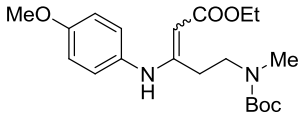
A mixture of compound **5** (23 mmol), substituted aniline **6** (25 mmol) and acetic acid (25 mmol) were stirred at room temperature for 18 h. After the completion of the reaction, EtOH (50 mL) was added. The solution was dried with anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel to give the desired products **7a**-**7b**, which were characterized as follows:

Ethyl 5-[(*tert*-butoxycarbonyl)(methyl)amino]-3-(*p*-tolylamino)pent-2-enoate (7a)



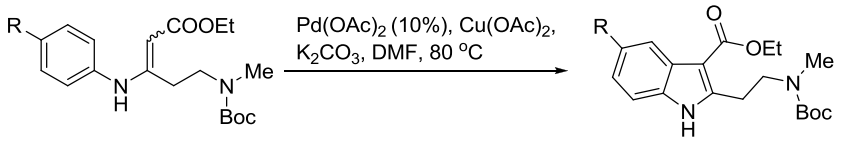
Following the general procedure, **7a** was obtained. Yield: 94%, white solid, mp 94-96 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 10.16 (s, 1H), 7.17-7.11 (d, 2H), 7.07-6.96 (d, 2H), δ 4.14 (q, *J* = 7.0 Hz, 2H), 3.24 (t, *J* = 6.9 Hz, 2H), 2.65 (s, 3H), 2.47 (d, *J* = 6.8 Hz, 2H), 2.33 (s, 3H), 1.39 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 170.50, 160.49, 154.97, 136.33, 135.65, 129.81, 125.63, 85.71, 79.56, 58.79, 48.08, 34.11, 30.76, 28.41, 20.89, 14.56. HRMS (ESI) *m/z* calcd for $C_{20}H_{31}N_2O_4^+$ [M + H^+] 363.2278, found 363.2280.

Ethyl 5-[(*tert*-butoxycarbonyl)(methyl)amino]-3-[(4-methoxyphenyl)amino]pent-2-enoate (7b)



Following the general procedure, **7b** was obtained. Yield: 91%, white solid, mp 78-79 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 10.06 (s, 1H), 7.18-6.97 (m, 2H), 6.86 (d, *J* = 18.3, 8.8 Hz, 2H), 4.14 (q, *J* = 7.0 Hz, 2H), 3.80 (s, 3H), 3.23 (s, 2H), 2.64 (s, 3H), 2.41 (s, 2H), 1.40 (s, 9H), 1.31-1.24 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 170.53, 160.96, 157.86, 155.30, 131.71, 127.57, 114.38, 85.20, 79.55, 58.74, 55.43, 48.07, 34.09, 30.73, 28.35, 14.56. HRMS (ESI) *m/z* calcd for $C_{20}H_{31}N_2O_5^+$ [M + H^+] 379.2227, found 379.2228.

General procedure for the preparation of 8a and 8b[6]

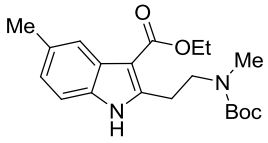


7a: R = Me
7b: R = MeO

8a: R = Me
8b: R = MeO

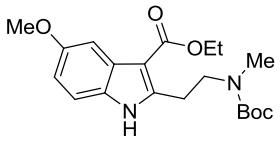
7 (5 mmol) was stirred with $Pd(OAc)_2$ (0.5 mmol), $Cu(OAc)_2$ (15 mmol), K_2CO_3 (15 mmol) and DMF (50 mL) under N_2 in a preheated oil bath at 80 °C for 4 h until TLC indicated the total consumption of starting material. The reaction mixture was diluted with EA (150 mL) and filtered through a short pad of silica. The solid was washed with EA (50 mL), and the combined filtrates were evaporated to dryness. The residue was purified by flash column chromatography on silica gel to give the desired products **8a-8b**, which were characterized as follows:

Ethyl 2-{2-[(*tert*-butoxycarbonyl)(methyl)amino]ethyl}-5-methyl-1*H*-indole-3-carboxylate (8a)



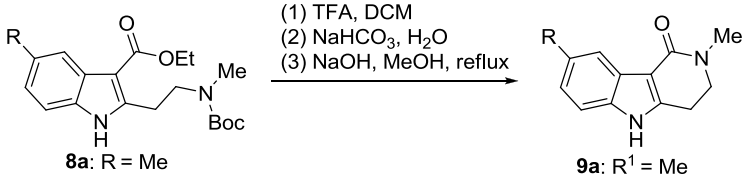
Following the general procedure, **8a** was obtained. Yield: 89%, light brown solid, mp 99-102 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 10.48 (s, 1H), 7.87 (s, 1H), 7.21 (d, *J* = 6.4 Hz, 1H), 7.00 (d, *J* = 9.2 Hz, 1H), 4.44-4.37 (q, *J* = 7.0 Hz, 2H), 3.74 (m, 2H), 3.47 (m, 2H), 2.86 (s, 3H), 2.46 (s, 3H), 1.56-1.32 (m, 12H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 166.24, 157.06, 145.17, 133.28, 130.88, 127.40, 123.67, 120.91, 110.84, 103.60, 80.58, 59.32, 45.91, 34.05, 28.38, 26.97, 21.73, 14.72. HRMS (ESI) *m/z* calcd for $C_{20}H_{28}N_2NaO_4^+$ [M + Na^+] 383.1941, found 383.1942.

Ethyl 2-{2-[(*tert*-butoxycarbonyl)(methyl)amino]ethyl}-5-methoxy-1*H*-indole-3-carboxylate (8b)



Following the general procedure, **8b** was obtained. Yield: 90%, light brown solid, mp 82-86 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.54 (s, 1H), 7.63 (s, 1H), 7.24 (d, *J* = 8.6 Hz, 1H), 6.84 (d, *J* = 8.7 Hz, 1H), 4.42 (q, *J* = 7.1 Hz, 2H), 3.89 (s, 3H), 3.75 (m, 2H), 3.49 (m, 2H), 2.88 (s, 3H), 1.59-1.28 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 166.06, 157.07, 155.52, 145.45, 129.99, 128.03, 112.01, 111.81, 103.90, 103.49, 80.45, 59.29, 55.71, 46.01, 34.08, 28.32, 27.04, 14.65. RMS (ESI) *m/z* calcd for $C_{20}H_{28}N_2NaO_5^+$ [M + Na^+] 399.1890, found 399.1894.

General procedure for the preparation of 9a and 9b



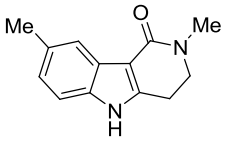
8a: R = Me
8b: R = MeO

9a: R^1 = Me
9b: R^1 = MeO

To a stirred solution of **8** (5 mmol) in CH_2Cl_2 (100 mL) was added TFA (25 mL) dropwise at 0 °C under N_2. The mixture was stirred at rt until TLC indicated the total consumption of starting material. The mixture was evaporated to remove the solvent and excess TFA. Then saturated $NaHCO_3$ (60 mL) was added and CH_2Cl_2 was used to extract the mixture. The combined organic

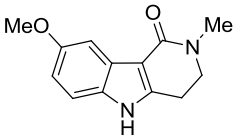
phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent. The residue was redissolved in MeOH (100 mL), NaOH (6 mmol) was added, the resulted mixture was refluxed for 5 h. After the completion of the reaction, the mixture was evaporated to remove the solvent. The residue was purified by flash column chromatography on silica gel to give the desired products **9a-9b**, which were characterized as follows:

2,8-Dimethyl-2,3,4,5-tetrahydro-1*H*-pyrido[4,3-*b*]indol-1-one (9a)



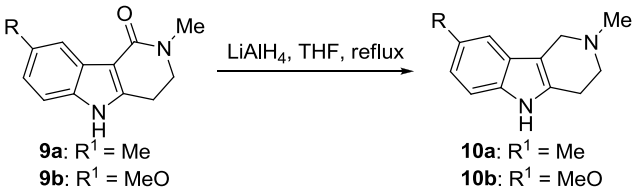
Following the general procedure, **9a** was obtained. Yield: 91%, white solid, mp > 300 °C. ^{1}H NMR (600 MHz, DMSO): ^{1}H NMR (600 MHz, DMSO): δ 11.44 (s, 1H), 7.70 (s, 1H), 7.25 (s, 1H), 6.93 (s, 1H), 3.58 (t, 2H), 3.03 (t, 2H), 2.93 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (151 MHz, DMSO): δ 164.83, 143.40, 134.28, 129.10, 125.61, 122.81, 119.38, 111.07, 104.87, 48.07, 33.17, 22.29, 21.24. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}N_2O^+$ [M + H^+] 215.1179, found 215.1184.

8-Methoxy-2-methyl-2,3,4,5-tetrahydro-1*H*-pyrido[4,3-*b*]indol-1-one (9b)



Following the general procedure, **9b** was obtained. Yield: 92%, white solid, mp 294-296 °C. ^{1}H NMR (600 MHz, DMSO): δ 7.37 (s, 1H), 7.29 (d, *J* = 6.3 Hz, 1H), 6.75-6.70 (m, 1H), 3.75 (s, 3H), 3.56 (t, *J* = 6.9 Hz, 2H), 3.04 (t, *J* = 6.7 Hz, 2H), 2.93 (s, 3H). ^{13}C NMR (151 MHz, DMSO): δ 164.91, 154.42, 143.80, 130.86, 126.01, 112.26, 110.88, 105.09, 101.63, 55.16, 48.07, 33.12, 22.41. HRMS (ESI) *m/z* calcd for $C_{13}H_{15}N_2O_2^+$ [M + H^+] 231.1128, found 231.1130.

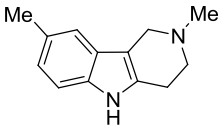
General procedure for the preparation of 10a and 10b[7]



To a suspension of $LiAlH_4$ (6 mmol) in anhydrous THF (20 mL) was added **9** (2 mmol) at 0°C. The resulted mixture was stirred at relux temperature for 4 h. The reaction mixture was cooled to 0 °C before water (5 mL) was added dropwise. CH_2Cl_2 was used to extract the mixture. The organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent under vacuum.

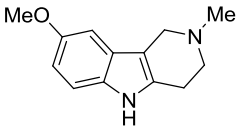
The residue was purified by flash column chromatography on silica gel to give the desired products **10a-10b**, which were characterized as follows:

2,8-Dimethyl-2,3,4,5-tetrahydro-1*H*-pyrido[4,3-*b*]indole (10a)



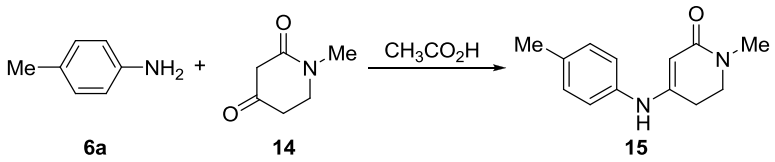
Following the general procedure, **10a** was obtained. Yield: 85%, white solid, mp 97-99 ^{o}C. ^{1}H NMR (400 MHz, DMSO): δ 10.75 (s, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.11 (s, 1H), 6.84 (d, *J* = 8.4 Hz, 2H), 3.72 (s, 2H), 2.91-2.93 (m, 2H), 2.85-2.86 (m, 2H), 2.51 (s, 3H), 2.35 (s, 3 H). HRMS (ESI) m/z calcd for $C_{13}H_{17}N_2^+$ [M + H^+] 201.1386, found 201.1388.

8-Methoxy-2-methyl-2,3,4,5-tetrahydro-1*H*-pyrido[4,3-*b*]indole (10b)



Following the general procedure, **10b** was obtained. Yield: 88%, white solid, mp 149-151 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29 (s, 1H), 7.13 (d, *J* = 8.7 Hz, 1H), 6.87 (s, 1H), 6.77 (d, *J* = 8.7, 1H), 3.86 (s, 3H), 3.70 (s, 2H), 2.84 (t, *J* = 5.6 Hz, 2H), 2.80 (t, *J* = 5.5 Hz, 2H), 2.60 (s, 3H). HRMS (ESI) m/z calcd for $C_{13}H_{17}N_2O^+$ [M + H^+] 217.1335, found 217.1338.

1-Methyl-4-(*p*-tolylamino)-5,6-dihydropyridin-2(1*H*)-one (15)



1-Methylpiperidine-2,4-dione **14** was prepared by the known method.[8] **14** (2.9 g, 23 mmol), *p*-toluidine (2.7 g, 25 mmol) and acetic acid (150 mg, 25 mmol) were stirred at rt for 18 h. After the completion of the reaction, EtOH (50 mL) was added, the solution was dried with anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by column chromatography to afford the pure product **15** (4.5 g, 21 mmol). Yield: 92%, white solid, mp 179-180 ^{o}C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.06 (d, *J* = 12 Hz, 2H), 7.01 (d, *J* = 6 Hz, 2H), 5.25 (s, 1H), 3.34 (t, *J* = 6 Hz, 2H), 2.92(s, 3H), 2.54(t, *J* = 6 Hz, 2H), 2.29(s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 169.18, 152.56, 136.83, 133.83, 129.71, 122.44, 90.74, 46.82, 34.21, 28.70, 20.85. HRMS (ESI) m/z calcd for $C_{13}H_{17}N_2O^+$ [M + H^+] 217.1335, found 217.1335.

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Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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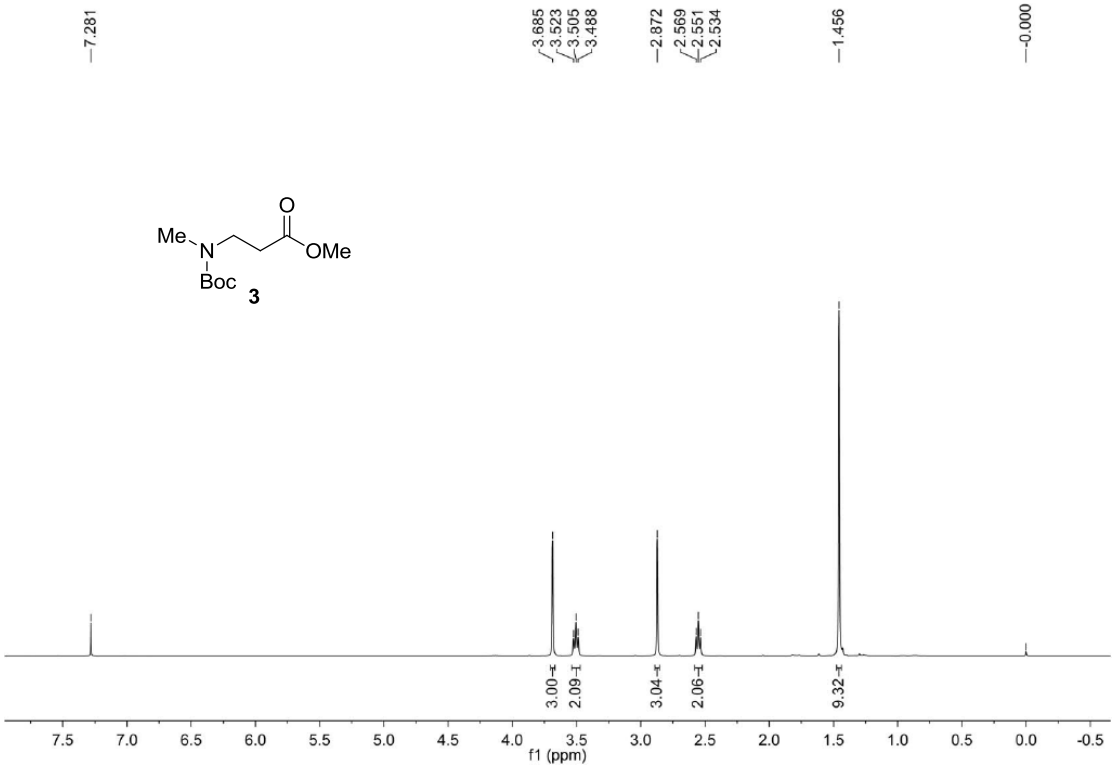
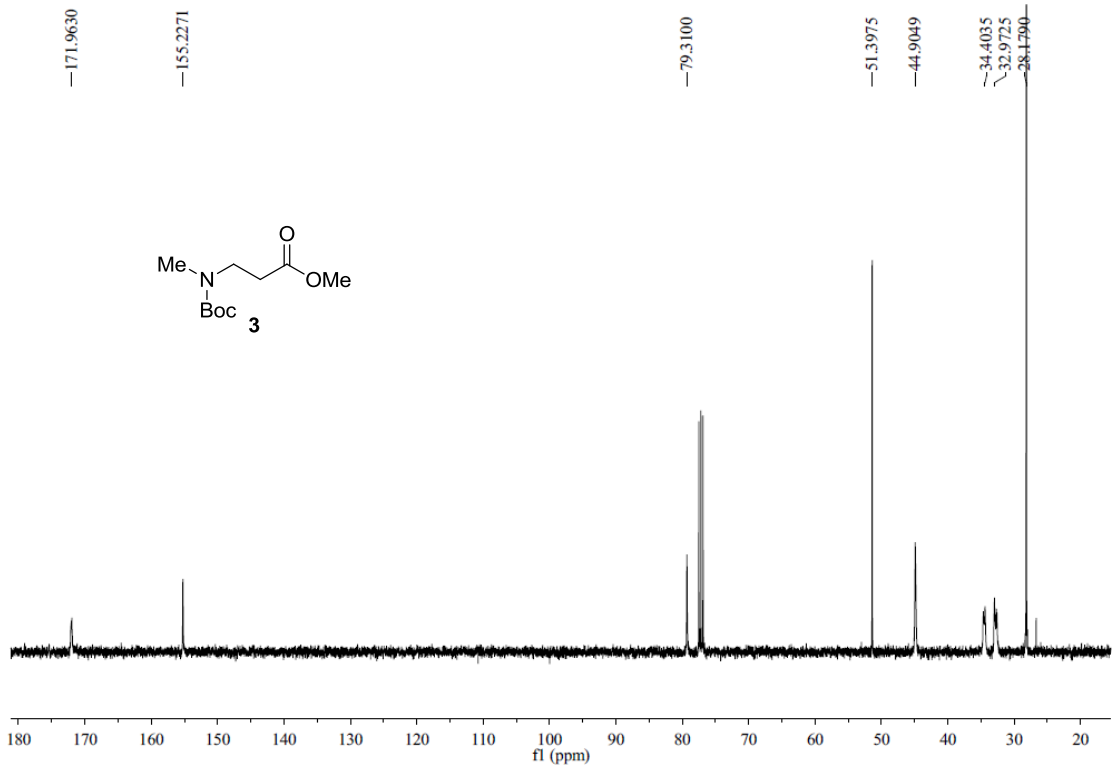
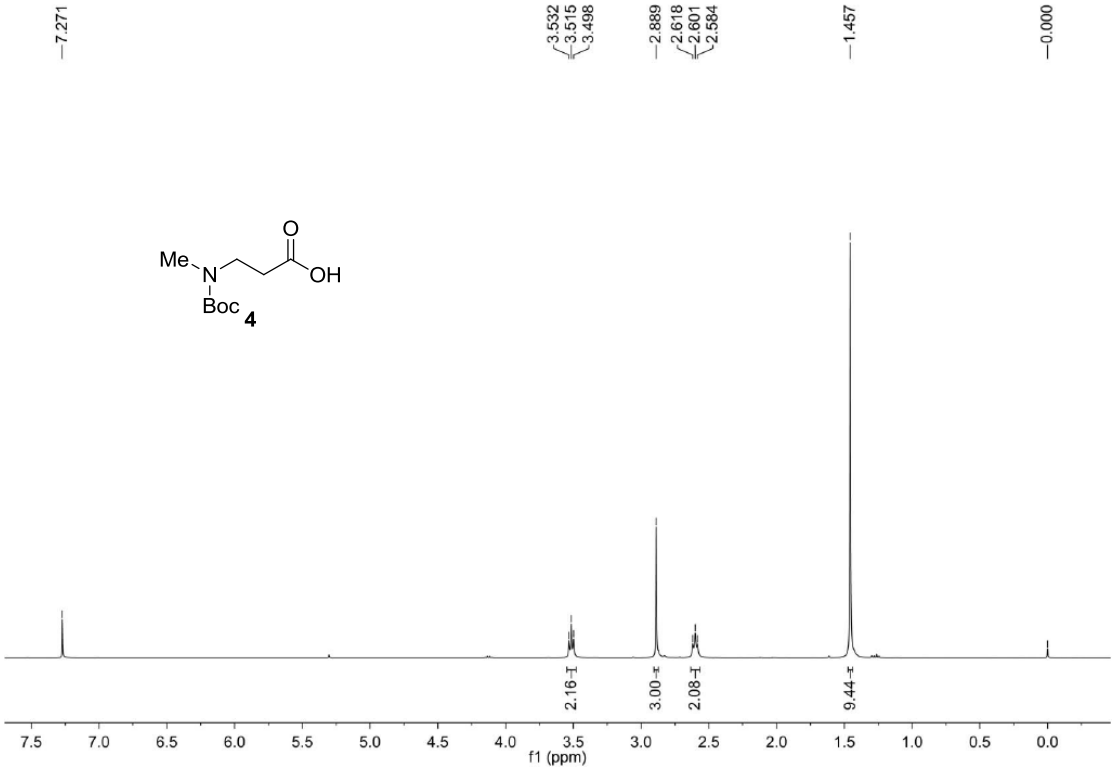
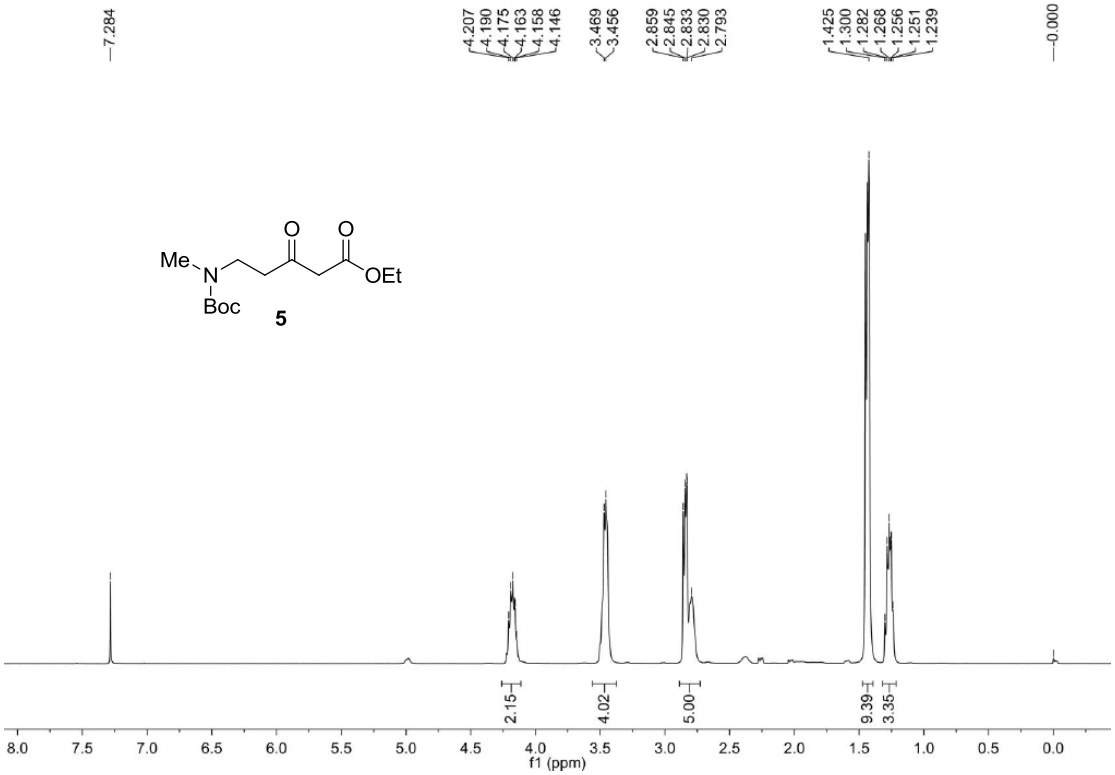
^{1}H and ^{13}C NMR Spectra






7.271
3.532
3.515
3.498
2.889
2.618
2.601
2.584
1.457
0.000
Me
N
Boc
O
OH
4
2.16
3.00
2.08
9.44
f1 (ppm)


7.284
4.207
4.190
4.175
4.163
4.158
4.146
3.469
3.456
2.859
2.845
2.833
2.830
2.793
1.425
1.300
1.282
1.268
1.256
1.251
1.239
0.000
Me
N
Boc
O
O
OEt
5
2.15
4.02
5.00
9.39
3.35
f1 (ppm)

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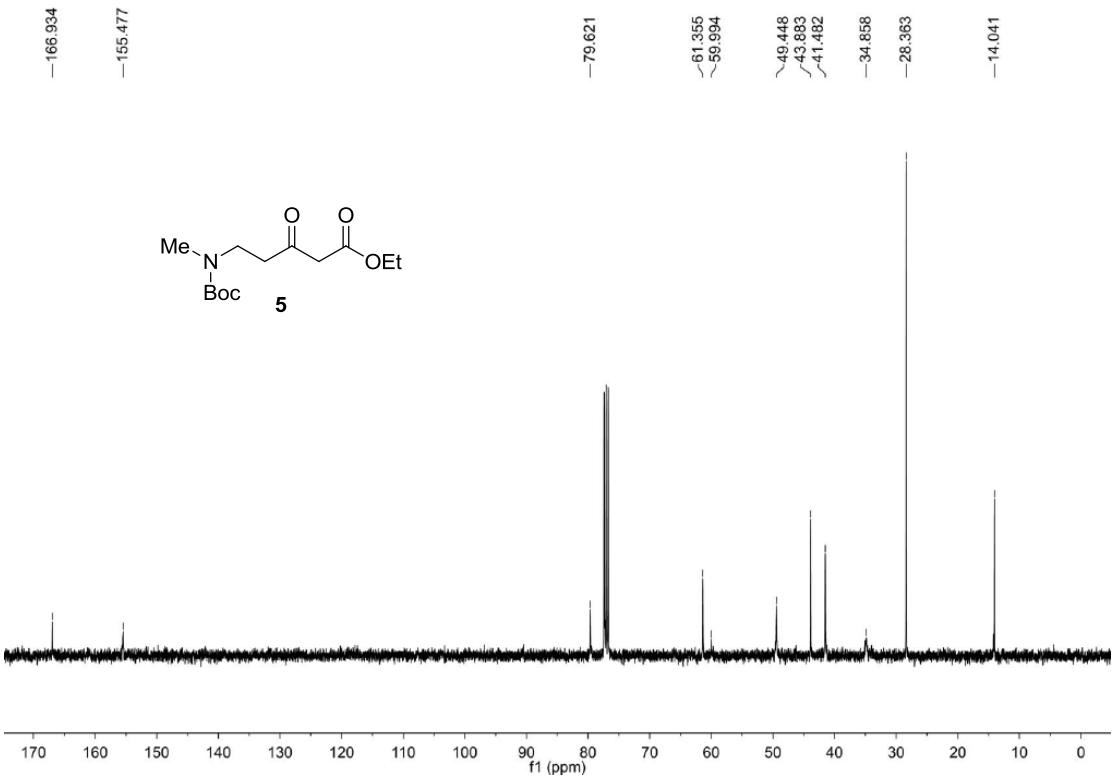
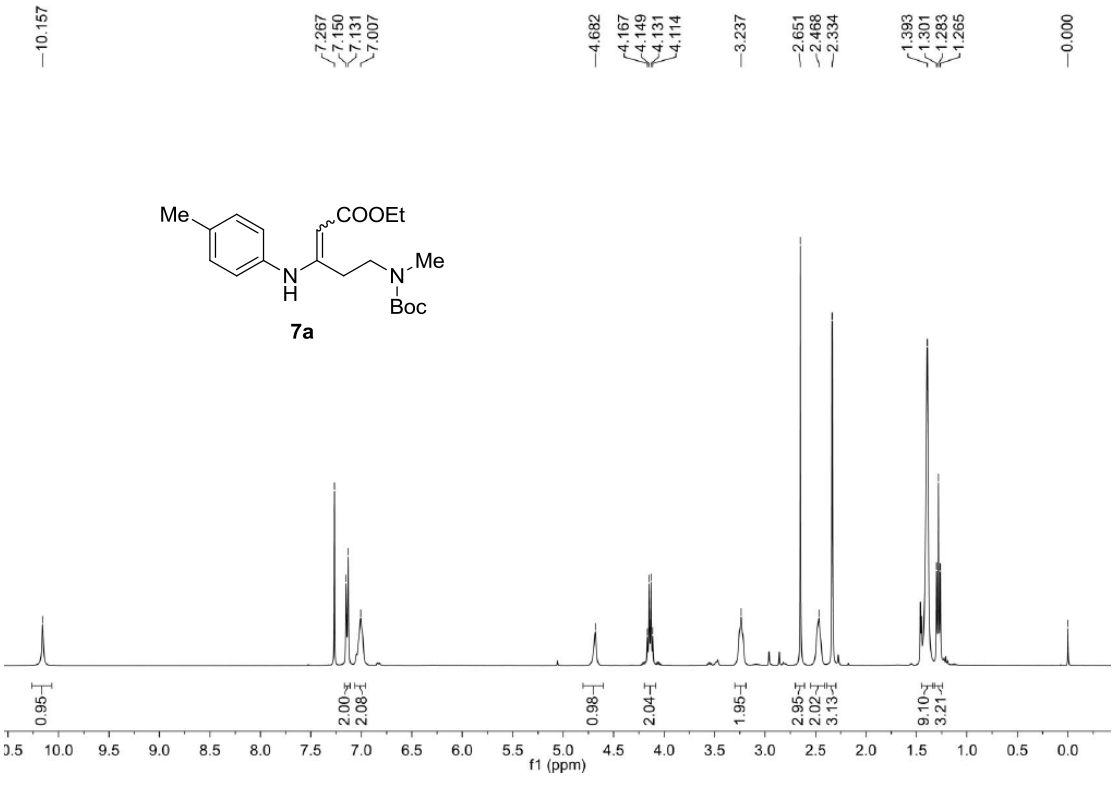
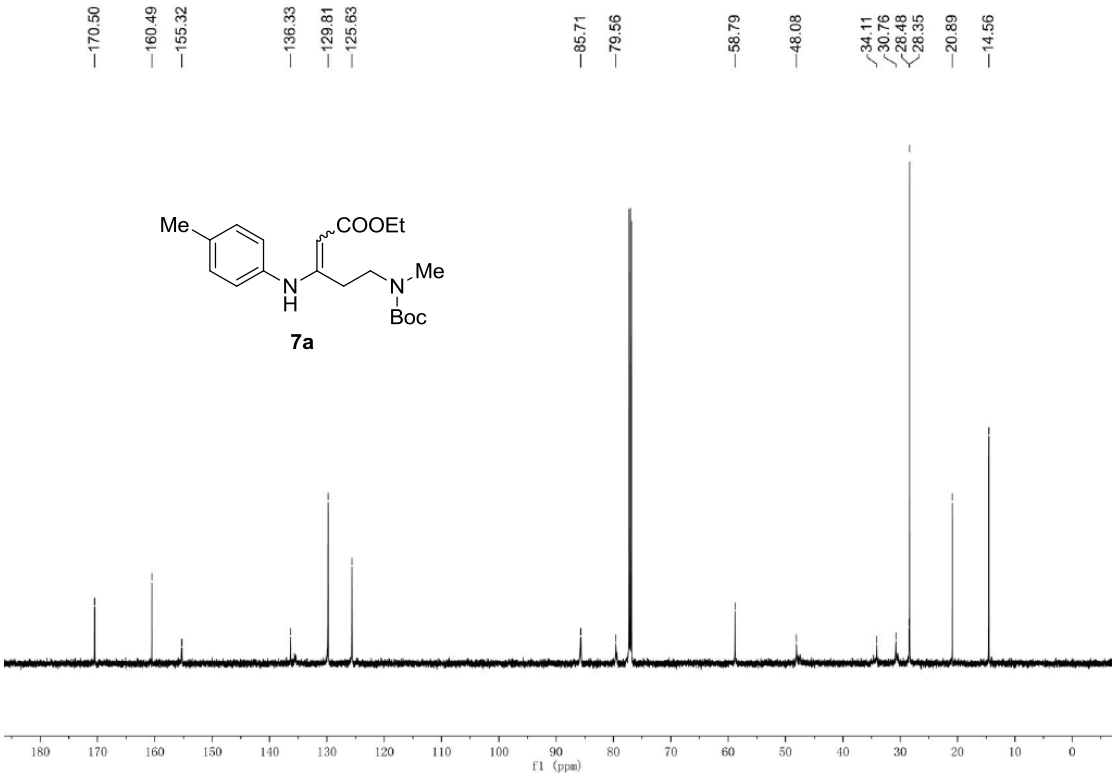
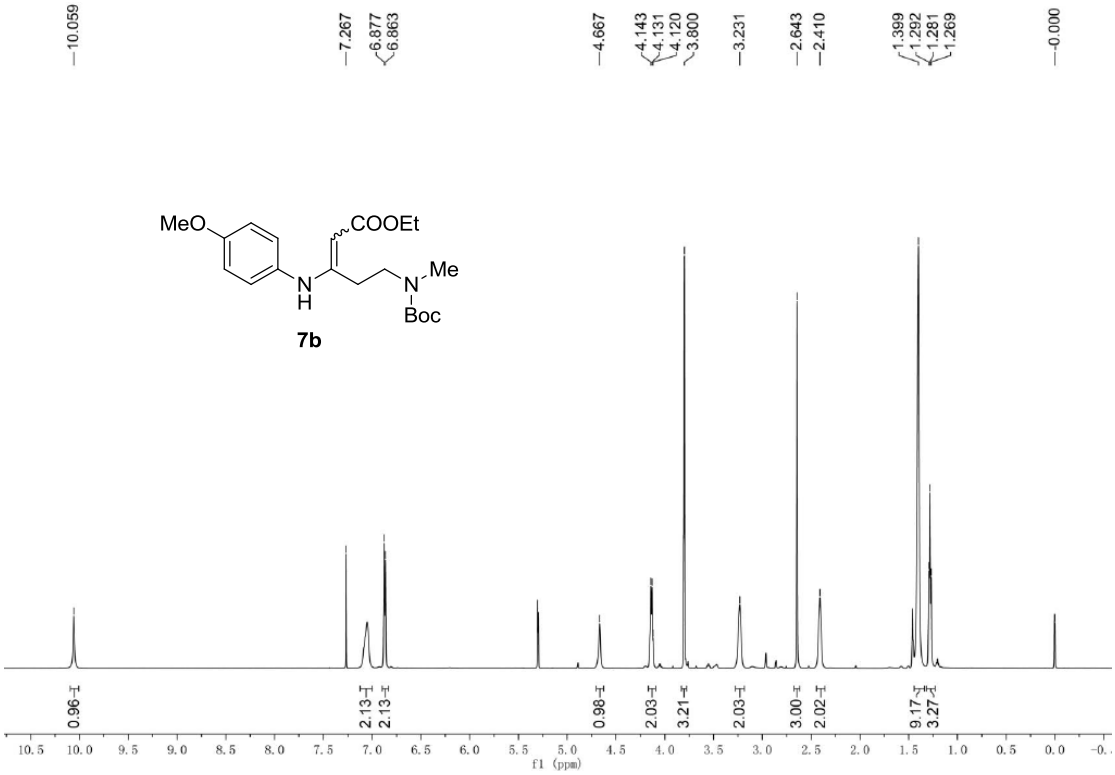


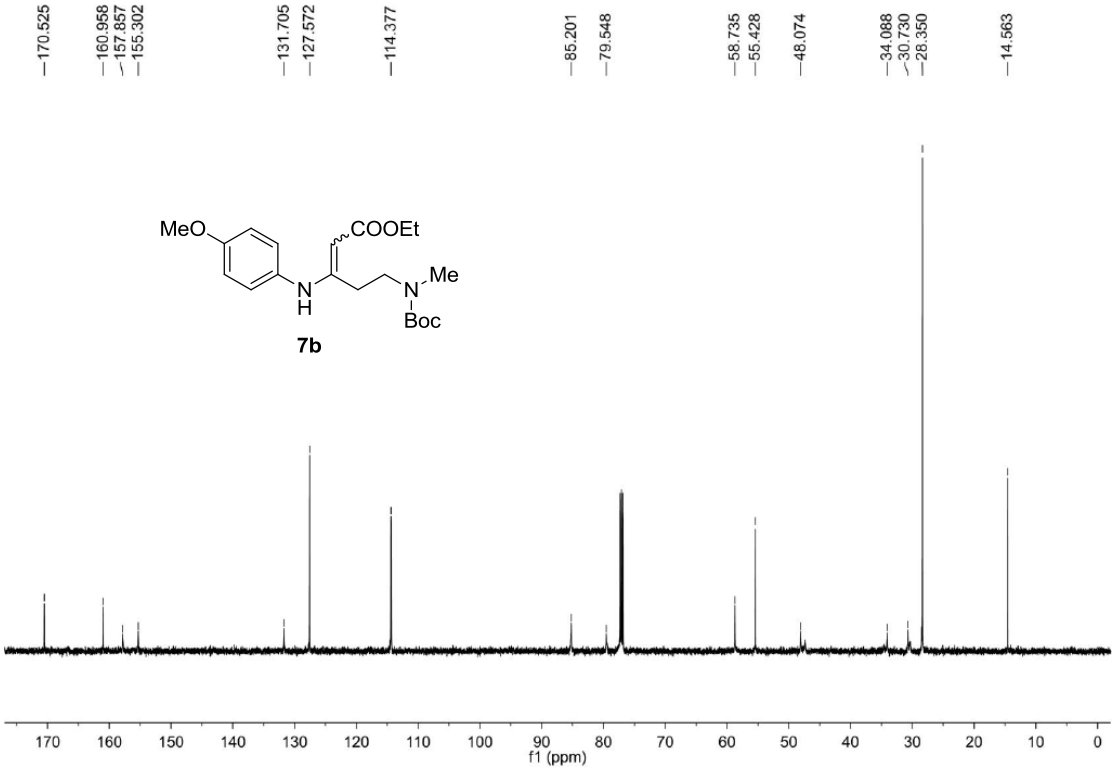
5

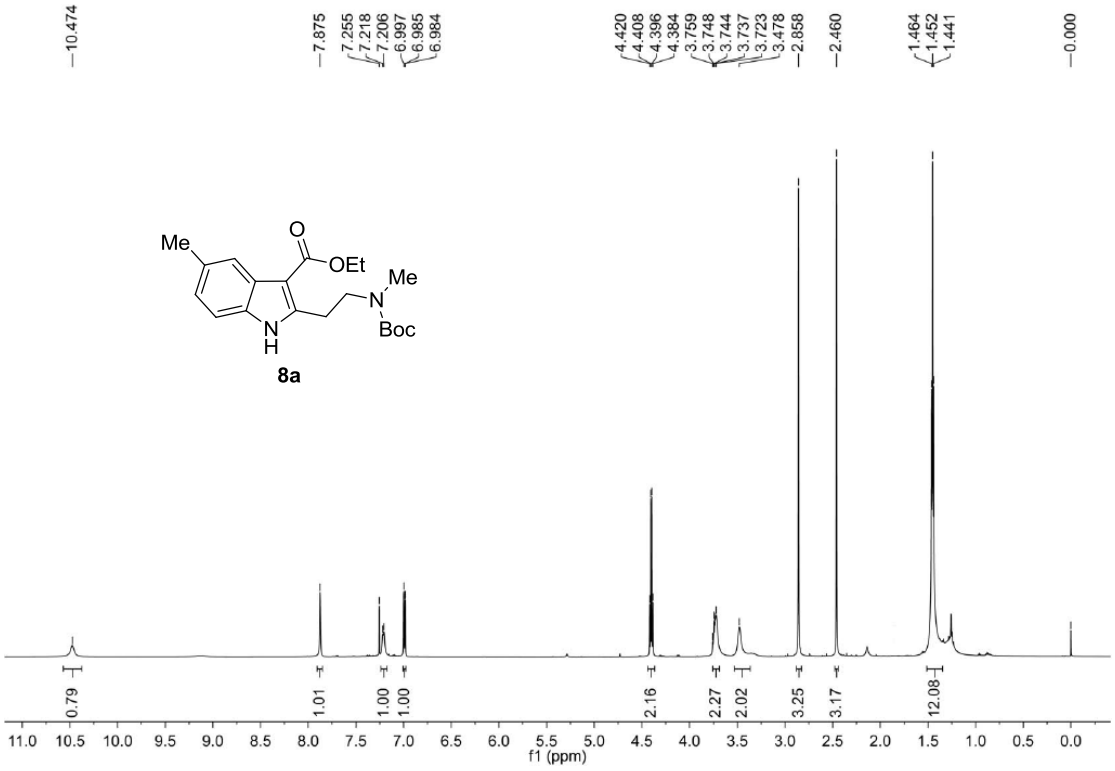


7a


170.50
160.49
155.32
136.33
129.81
125.63
85.71
79.56
58.79
48.08
34.11
30.76
28.48
28.35
20.89
14.56
Me
COOEt
N
H
N
Me
Boc
7a
f1 (ppm)


10.059
7.267
6.877
6.863
4.667
4.143
4.131
4.120
3.800
3.231
2.643
2.410
1.399
1.292
1.281
1.269
0.000
MeO
COOEt
N
H
N
Me
Boc
7b
0.96
2.13
2.13
0.98
2.03
3.21
2.03
3.00
2.02
9.17
3.27
f1 (ppm)


170.525
160.958
157.857
155.302
131.705
127.572
114.377
85.201
79.548
58.735
55.428
48.074
34.088
30.730
28.350
14.563
MeO
COOEt
N
H
N
Me
Boc
7b
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


10.474
7.875
7.255
7.218
7.206
6.997
6.985
6.984
4.420
4.408
4.396
4.384
3.759
3.748
3.744
3.737
3.723
3.478
2.858
2.460
1.464
1.452
1.441
0.000
Me
O
OEt
N
Me
Boc
N
H
8a
0.79
1.01
1.00
1.00
2.16
2.27
2.02
3.25
3.17
12.08
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

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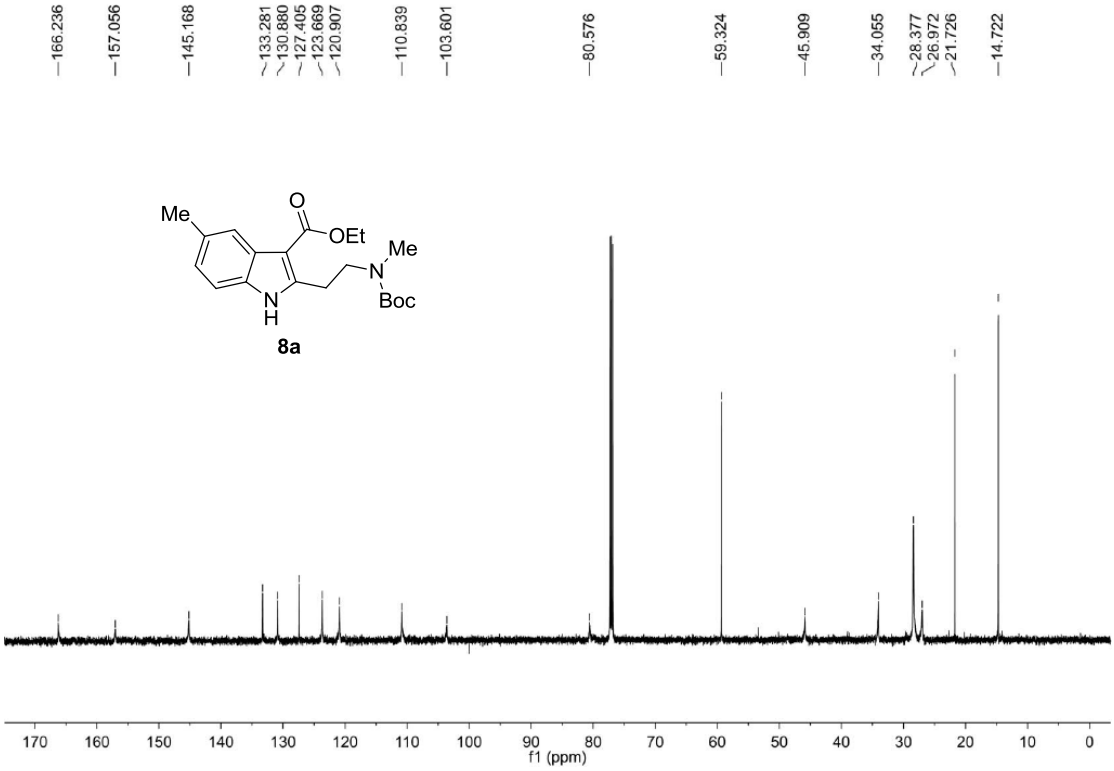

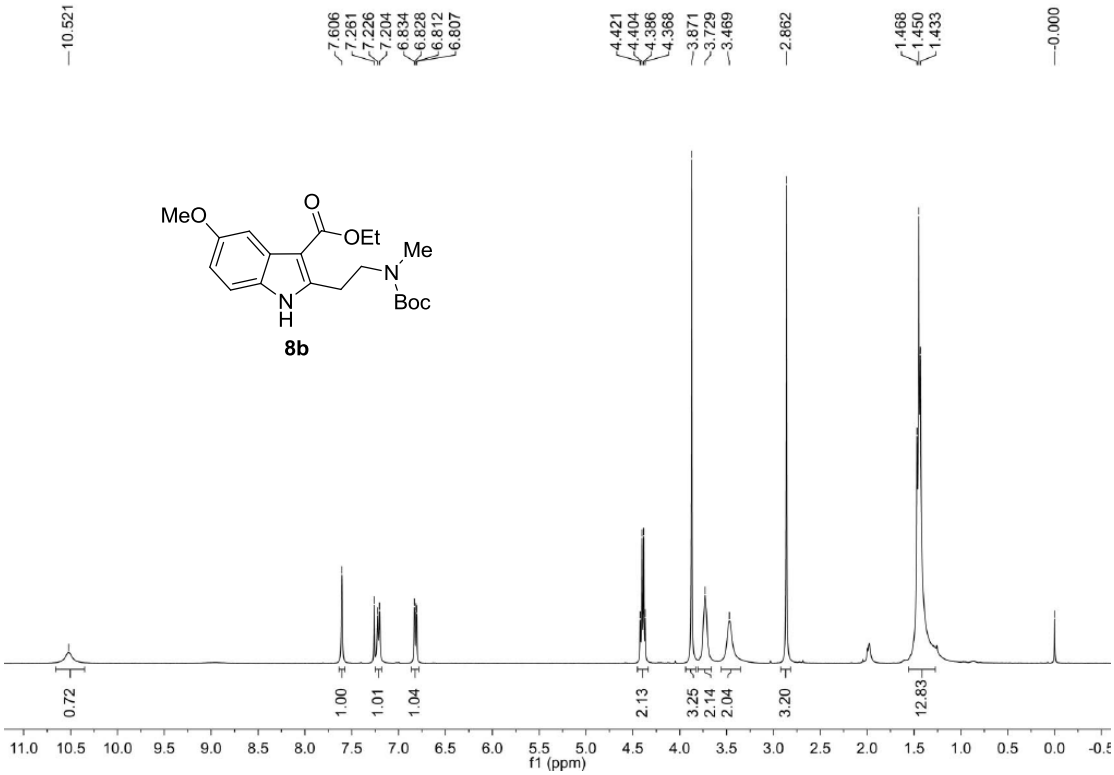

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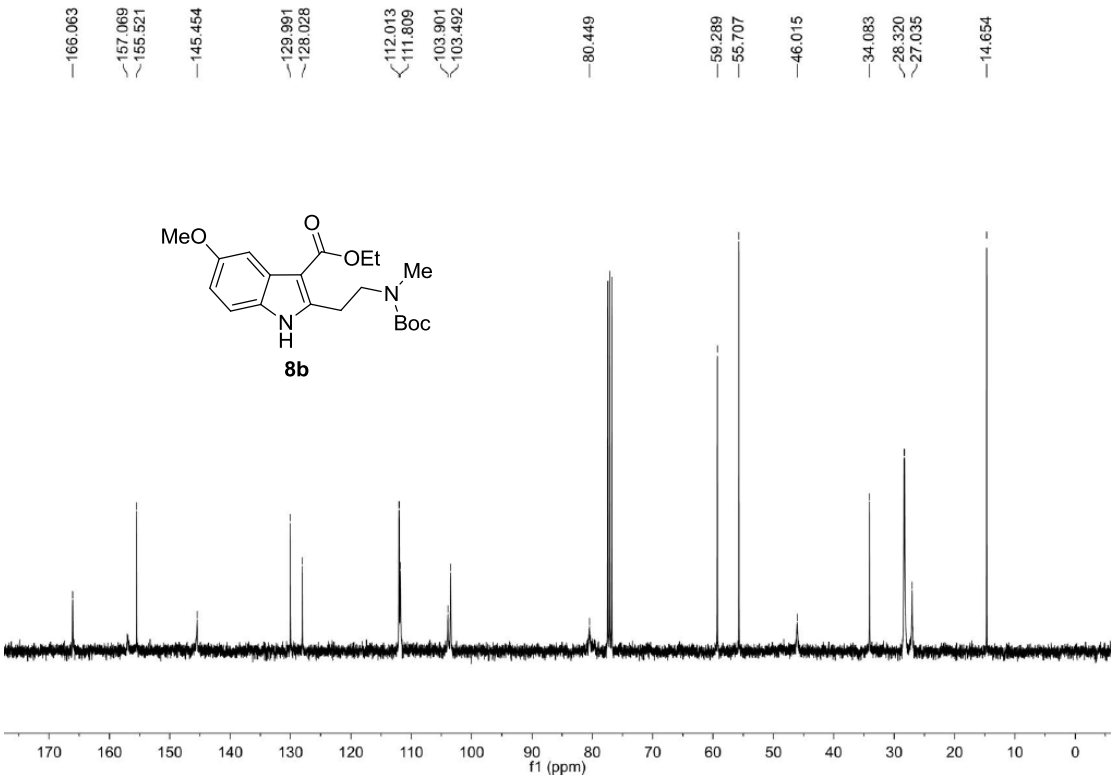
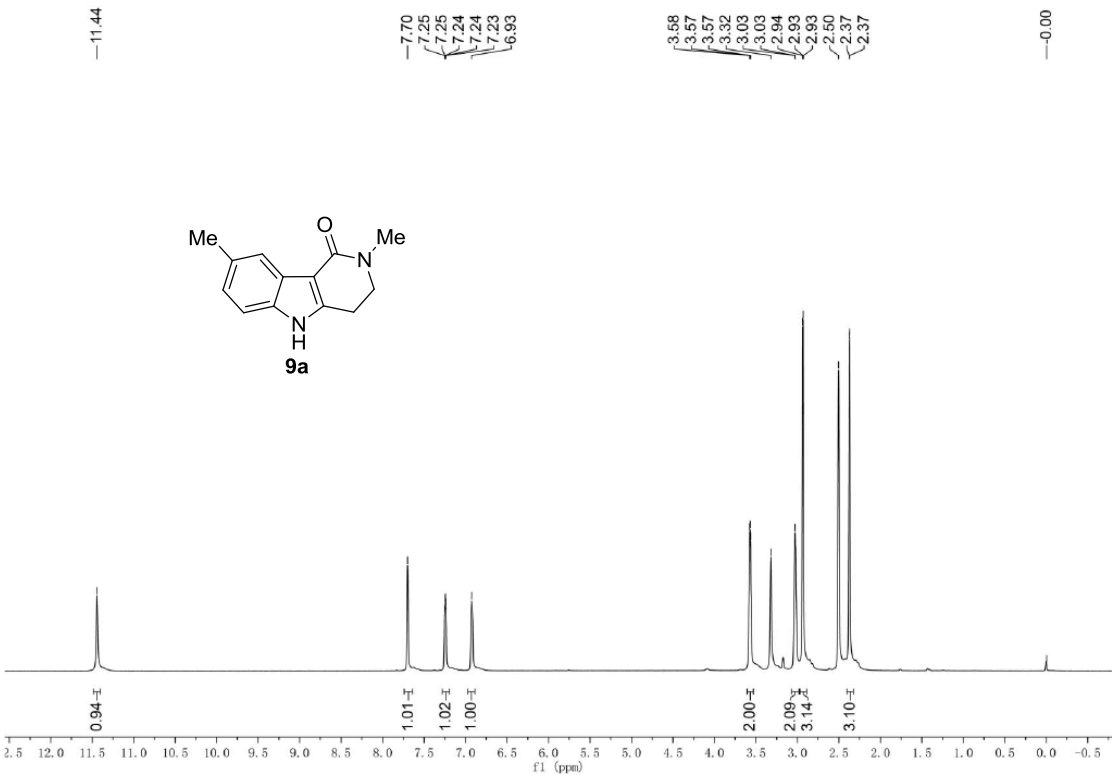
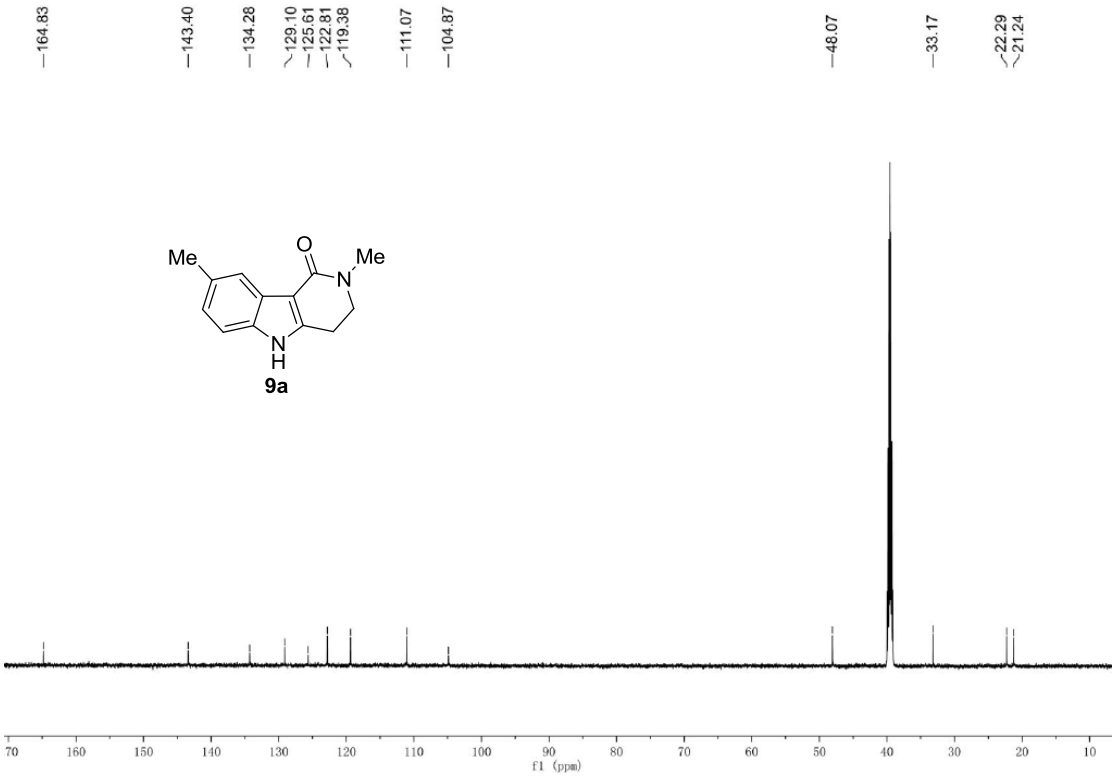
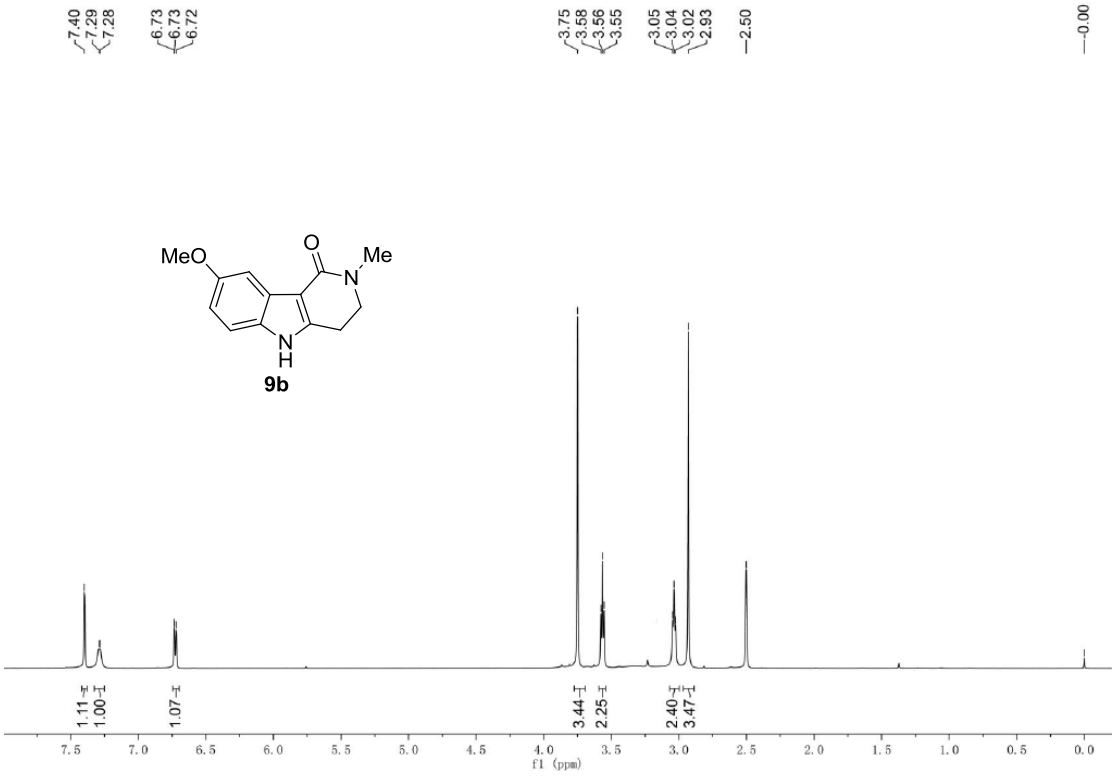


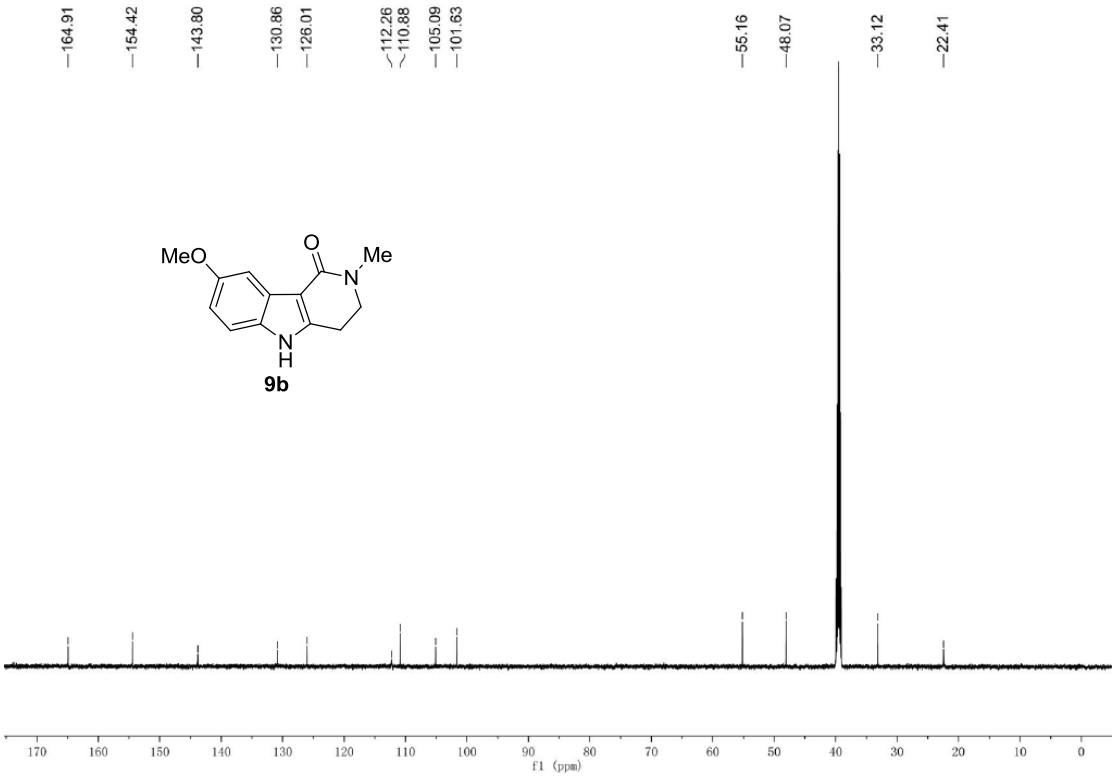


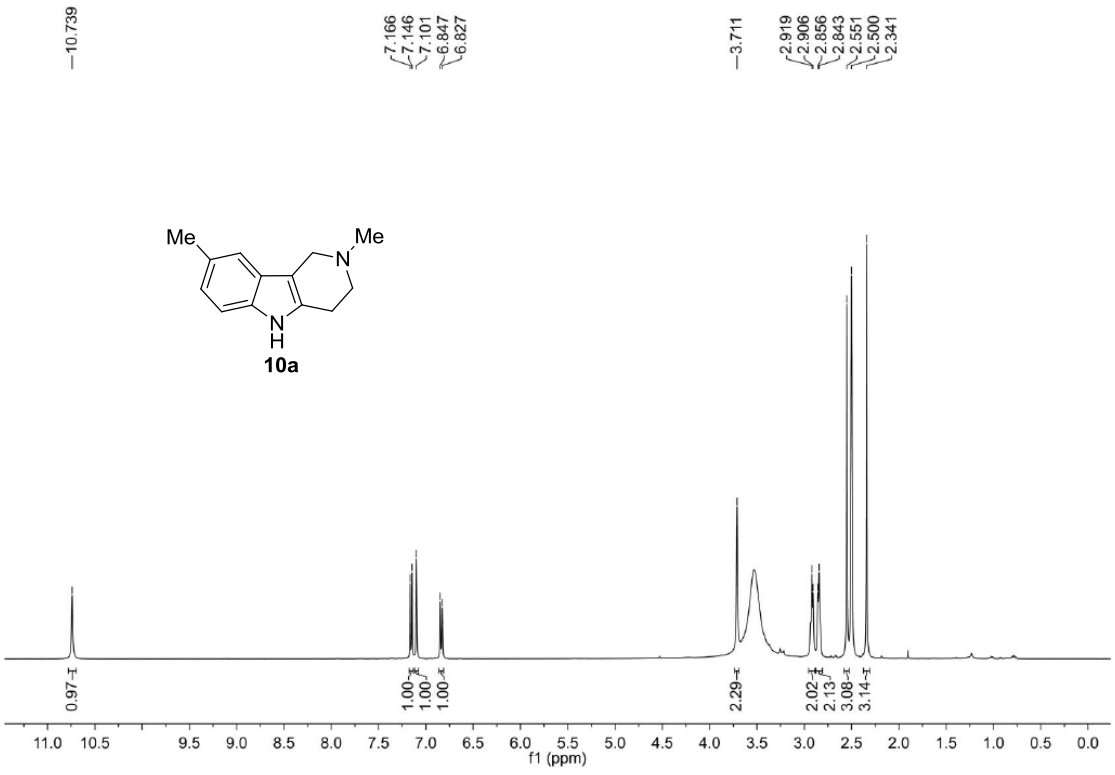


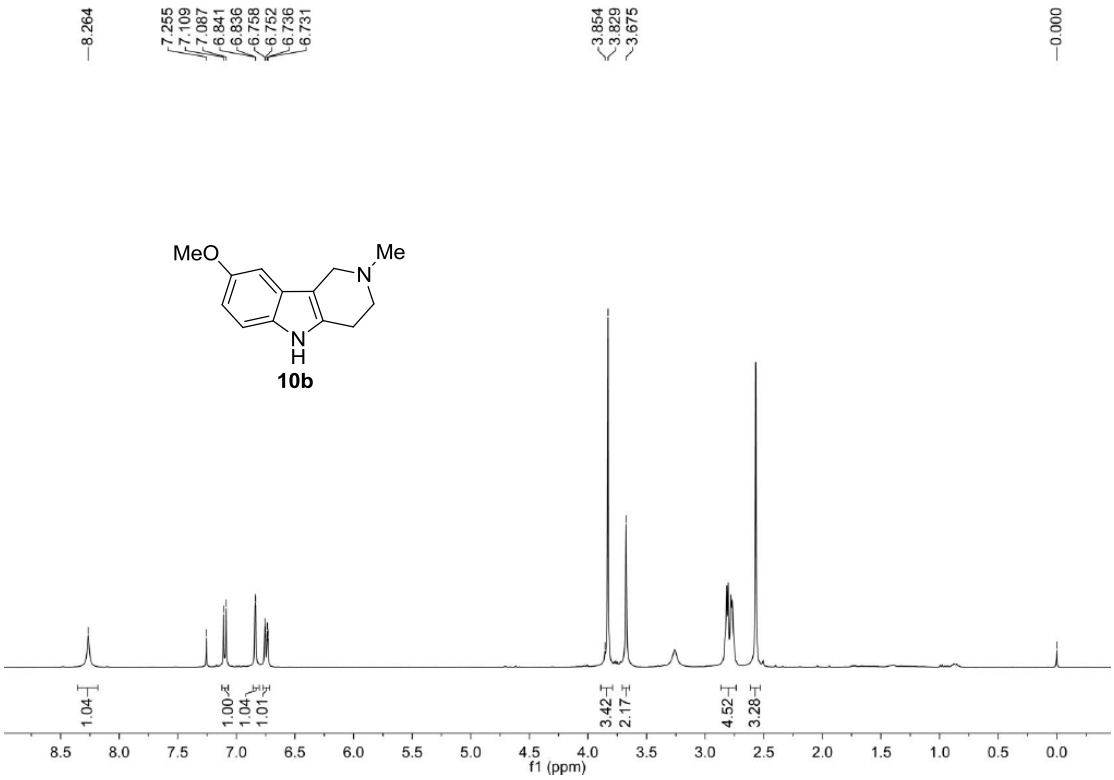
9a

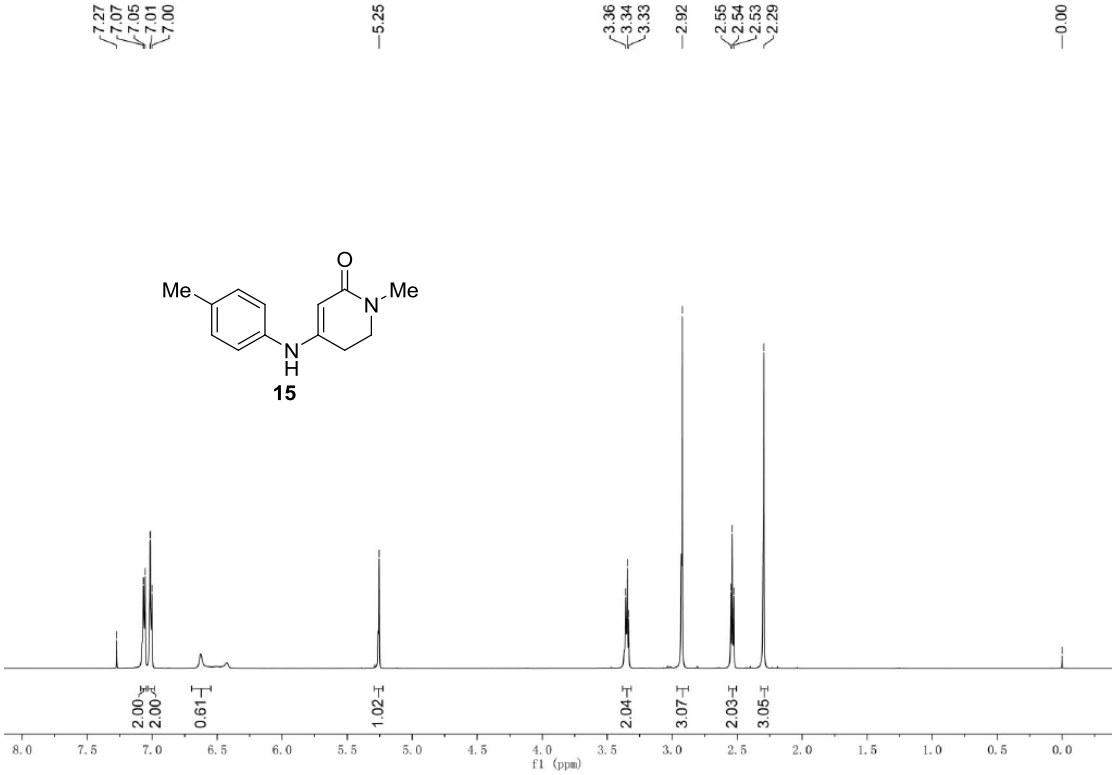


9b


164.91
154.42
143.80
130.86
126.01
112.26
110.88
105.09
101.63
55.16
48.07
33.12
22.41
MeO
O
N
Me
N
H
9b
f1 (ppm)


10.739
7.166
7.146
7.101
6.847
6.827
3.711
2.919
2.906
2.856
2.843
2.551
2.500
2.341
Me
N
Me
N
H
10a
0.97
1.00
1.00
1.00
2.29
2.02
2.13
3.08
3.14
f1 (ppm)


8.264
7.255
7.109
7.087
6.841
6.836
6.758
6.752
6.736
6.731
3.854
3.829
3.675
0.000
MeO
Me
N
N
H
10b
1.04
1.00
1.04
1.01
3.42
2.17
4.52
3.28
f1 (ppm)


7.27
7.07
7.05
7.01
7.00
5.25
3.36
3.34
3.33
2.92
2.55
2.54
2.53
2.29
0.00
O
Me
N
Me
N
H
15
2.00
2.00
0.61
1.02
2.04
3.07
2.03
3.05
f1 (ppm)

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