Manganese-Catalyzed Dehydrogenative Csp^3–Csp^2 Coupling of Imidazo[1,2-a]pyridines with Methyl Ketones

Hua Yao, Xiaoyang Zhong, Bingqing Wang, Sen Lin*, Lichi Liu, and Zhaohua Yan*

College of Chemistry, Nanchang University, Nanchang 330031, PR China

Email: senlin@ncu.edu.cn

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1. General information

^{1}H and ^{13}C NMR spectra were recorded on Bruker Ascend™ 400 (400 MHz) using tetramethylsilane as an internal reference. NMR multiplicities are abbreviated as follows: s = singlet, d = doublet, m = multiplet, br = broad signal. Chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. The rest of chemicals were purchased from the Sinopharm Chemical Reagent Co., Adamas, Aladdin and TCI used as received. Q-TOF were used for the HRMS and GC-MS measurement. HRMS (ESI) data were obtained using electron spray ionization and GC-MS data were obtained using electron impact ionization.

2. General procedure for for the synthesis of products

Without other special notes, all products were synthesized according to the following procedure: 50 mL Schlenk tube was added imidazopyridines **1** (0.2 mmol), ketones **2** (2 mL), $Mn(OAc)_2{\cdot}4H_2O$ (10 mol %) and DCP (3 equiv). The tube was stirred at 120°C for 24 h. After the reaction mixture was cooled to room temperature, the reaction mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was washed with brine (10 mL), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash chromatography on silica gel with a mixture of petroleum ether and ethyl acetate as eluent to afford various target compounds (PE/EA = 20/1--2/1).

3. Characterization data for products

1-(2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3a)** [1]

Purified by using a flash column chromatography; isolated yield = 78%. 36.9 mg; white solid; mp:70-71 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) 1H NMR (400 MHz, Chloroform-d) δ 7.81 (d, J = 6.9 Hz, 1H), 7.66 – 7.59 (m, 3H), 7.43 (t, J = 7.5 Hz, 2H), 7.34 (t, J = 7.3 Hz, 1H), 7.19 – 7.15 (m, 1H), 6.78 (t, J = 6.6 Hz, 1H), 4.10 (s, 2H), 2.14 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.05, 145.05, 144.48, 134.11, 128.72, 128.38, 127.99, 124.61, 123.34, 117.67, 113.35, 112.63, 39.60, 29.23.

1-(2-(4-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)propan-2-one **(3b)**

Purified by using a flash column chromatography; isolated yield = 69%, 38.6 mg; white solid; mp:65-66 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 (dd, *J* = 12.2, 7.6 Hz, 3H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.3 Hz, 1H), 6.92 (d, *J* = 2.0 Hz, 1H), 6.54 (dd, *J* = 7.4, 2.5 Hz, 1H), 4.08 (s, 2H), 3.85 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.41, 157.94, 146.51, 143.78, 134.24, 128.67, 128.15, 127.77, 123.89, 112.08, 107.62, 94.78, 55.53, 39.56, 29.14. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{16}N_2O_2$ 281.1285; Found: 281.1282.

1-(2-(4-fluorophenyl)imidazo[1,2-a]pyridin-3-yl)propan-2-one **(3c)**

Purified by using a flash column chromatography; isolated yield = 59%, 31.6 mg; yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (s, 1H), 7.68 – 7.60 (m, 3H), 7.25 (s, 1H), 7.17 (s, 2H), 6.83 (d, *J* = 6.8 Hz, 1H), 4.11 (s, 2H), 2.19 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.82, 162.65 (d, *J*=247 Hz), 145.07, 143.71, 130.35, 130.06 (d, *J*=8 Hz), 127.80, 123.99(d, *J*=139 Hz), 117.67, 115.73 (d, *J*=22 Hz), 113.19, 112.68, 39.52, 29.27. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{13}FN_2O$ 269.1085; Found: 269.1084.

1-(2-(2,4-dimethylphenyl)imidazo[1,2-a]pyridin-3-yl)propan-2-one **(3d)**

Purified by using a flash column chromatography; isolated yield = 56%, 31.1mg; yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 6.8 Hz, 1H), 7.63 (d, *J* = 9.1 Hz, 1H), 7.22 (d, *J* = 6.8 Hz, 1H), 7.14 (s, 1H), 7.12 (s, 1H), 7.06 (s, 1H), 6.84 (s, 1H), 3.90 (s, 2H), 2.37 (s, 3H), 2.24 (s, 3H), 2.07 (s, 3H). ^{13}C

NMR (101 MHz, Chloroform-*d*) δ 204.12,145.18, 144.81, 138.03, 137.40, 131.22, 130.37, 130.29, 126.28, 124.17, 123.50, 117.63, 114.22, 112.31, 39.32, 29.02, 21.23, 19.93. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{18}N_2O$ 279.1492; Found:279.1497.

1-(7-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3e)**

Purified by using a flash column chromatography; isolated yield = 82%, 46.7 mg; white solid; mp: 86-87 ℃. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.79 (s, 1H), 7.67 (s, 3H), 7.50 (s, 2H), 7.41 (d, *J* = 6.1 Hz, 1H), 6.84 – 6.81 (m, 1H), 4.14 (s, 2H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.57, 145.43, 144.88, 133.82, 131.09, 128.81, 128.38, 128.23, 123.83, 116.51, 114.13, 113.71, 39.44, 29.34. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{13}ClN_2O$ 285.0789; Found: 285.0784.

1-(7-methoxy-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3f)**

Purified by using a flash column chromatography; isolated yield = 75%, 42.2 mg; white solid; mp: 52-53 ℃. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.64 (s, 3H), 7.47 – 7.42 (m, 2H), 7.36 (s, 1H), 6.92 (d, *J* = 2.5 Hz, 1H), 6.54 (dd, *J* = 7.5, 2.5 Hz, 1H), 4.08 (s, 2H), 3.85 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.41, 157.94, 146.51, 143.78, 134.24, 128.67, 128.15, 127.77, 123.89, 112.08, 107.62, 94.78, 55.53, 39.56, 29.14. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{16}N_2O_2$ 281.1285; Found: 281.1285.

1-(7-methyl-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3g)**

Purified by using a flash column chromatography; isolated yield = 76%, 40.1 mg; colorless oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 7.4 Hz, 1H), 7.69 (s, 2H), 7.44 (s, 2H), 7.41 (s, 1H), 7.37 (t, *J* = 7.4 Hz, 1H), 6.67 (dd, *J* = 7.0, 1.7 Hz, 1H), 4.12 (s, 2H), 2.41 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.28, 145.52, 144.16, 135.58, 134.32, 128.67, 128.32, 127.83, 122.56, 116.06, 115.23, 112.68, 39.65, 29.12, 21.30. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{16}N_2O$ 265.1335; Found: 265.1345.

1-(8-methyl-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3h)**

Purified by using a flash column chromatography； isolated yield = 42%, 22.2 mg; white solid; mp: 70-71 ℃. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 6.9 Hz, 1H), 7.69 (d, *J* = 7.1 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.39 (s, 1H), 7.01 (d, *J* = 6.9 Hz, 1H), 6.76 (t, *J* = 6.8 Hz, 1H), 4.11 (s, 2H), 2.66 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 202.92, 152.26, 145.46, 130.93, 129.99, 129.71, 124.34, 123.82, 117.34, 116.13, 112.96, 104.22, 53.44, 48.15, 30.20. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{16}N_2O$ 265.1335; Found:265.1328.

1-(6-nitro-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3i)**

Purified by using a flash column chromatography; isolated yield = 45%, 23.9 mg; yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.98 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 9.5 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.39 (t, *J* = 7.3 Hz, 1H), 7.27 (dd, *J* = 9.5, 1.8 Hz, 1H), 4.14 (s, 2H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.40, 155.64, 133.75, 128.97, 128.78, 128.64, 128.36, 128.22, 127.96, 123.51, 118.32, 107.29, 39.41, 29.43. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{13}N_3O_3$ 296.1030; Found:296.1032.

1-(6-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)propan-2-one **(3j)**

Purified by using a flash column chromatography; isolated yield = 56%, 31.9 mg; white solid; mp: 55-56 ℃. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 1.0 Hz, 1H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 9.5 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.19 (dd, *J* = 9.5, 1.8 Hz, 1H), 4.15 (s, 2H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.43, 140.35, 130.90, 128.96, 128.81, 128.79, 128.64, 128.35, 128.22, 125.92, 121.32, 118.04, 39.43, 29.44. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{13}ClN_2O$ 285.0789, Found: 285.0784.

1-(2-(tert-butyl)imidazo[1,2-a]pyridin-3-yl)propan-2-one (**3l)**

Purified by using a flash column chromatography; isolated yield = 37 %, 17.1 mg; yellow solid; mp: 76-77 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.9 Hz, 1H), 7.16 – 7.10 (m, 2H), 6.66 (d, *J* = 6.8 Hz, 1H), 3.95 (s, 2H), 2.20 (s, 3H), 1.39 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 202.92, 130.93, 129.99, 129.71, 123.82, 116.13, 112.96, 104.22, 53.39, 48.15, 30.20, 29.10. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{14}H_{18}N_2O$ 231.1492; Found: 231.1501.

1-(2-(thiophen-2-yl)imidazo[1,2-a]pyridin-3-yl)propan-2-one **(3m)**

Purified by using a flash column chromatography; isolated yield = 58 %, 29.7 mg; yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 6.8 Hz, 1H), 7.64 (d, *J* = 9.0 Hz, 1H), 7.44 (d, *J* = 3.6 Hz, 1H), 7.38 (d, *J* = 5.1 Hz, 1H), 7.24 – 7.19 (m, 1H), 7.16 – 7.13 (m, 1H), 6.84 (td, *J* = 6.9, 1.2 Hz, 1H), 4.21 (s, 2H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.68, 145.09, 138.75, 136.90, 128.81, 127.80, 125.79, 124.98, 124.92, 123.23, 117.52, 112.79, 39.73, 29.23. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{14}H_{12}N_2OS$ 257.0743; Found: 257.0739.

3-(7-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)butan-2-one **(3o)**

Purified by using a flash column chromatography; isolated yield = 57 %, 34.2 mg; colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 7.3 Hz, 1H), 7.69 – 7.64 (m, 3H), 7.50 (t, *J* = 7.3 Hz, 2H), 7.45 – 7.40 (m, 1H), 6.79 (dd, *J* = 7.4, 2.1 Hz, 1H), 4.48 (t, *J* = 7.2 Hz, 1H), 1.97 (s, 3H), 1.55 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.56, 158.84, 144.95, 133.78, 131.07, 128.82, 128.66, 128.49 128.36, 124.52, 116.81, 114.30, 43.92, 28.48, 12.70. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{17}H_{15}ClN_2O$ 299.0946; Found: 299.0947.

1-(7-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)butan-2-one **(3p)**

Purified by using a flash column chromatography; isolated yield = 7 %, 4.2 mg; colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.92 (s, 1H), 7.64 (d, *J* = 7.4 Hz, 2H), 7.60 (d, *J* = 9.6 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.18 (dd, *J* = 9.6, 1.7 Hz, 1H), 4.13 (s, 2H), 2.54 (q, *J* = 7.2 Hz, 2H), 1.08 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.45, 145.24, 144.79, 133.81, 131.02, 128.77, 128.67, 128.36, 128.17, 123.93, 116.41, 114.06, 38.24, 35.36, 7.63. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{17}H_{15}ClN_2O$ 299.0946; Found: 299.0941.

3-(2-phenylimidazo[1,2-a]pyridin-3-yl)butan-2-one **(3q)**

Purified by using a flash column chromatography; isolated yield = 53 %, 27.9 mg; colorless oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 6.9 Hz, 1H), 7.69 (dd, *J* = 12.5, 8.2 Hz, 3H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.24 – 7.18 (m, 1H), 6.80 (t, *J* = 6.8 Hz, 1H), 4.49 (q, *J* = 7.2 Hz, 1H), 1.95 (s, 3H), 1.57 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.88, 145.21, 144.51, 134.25, 128.74, 128.13, 124.49, 124.05, 118.13, 118.05, 112.76, 44.09, 28.36, 12.60. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.88, 145.21, 144.51, 134.25, 129.80, 128.74, 128.13, 124.49, 124.05, 118.13, 118.05, 112.76, 44.09, 28.36, 12.60. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{17}H_{16}N_2O$ 265.1335; Found:265.1336.

2-(7-chloro-2-phenylimidazo[1,2-a]pyridin-3-yl)-1-phenylethan-1-one **(3s)**

Purified by using a flash column chromatography; isolated yield = 62 %, 42.9 mg; white solid; mp: 48-49 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.5 Hz, 2H), 7.85 (d, *J* = 7.2 Hz, 1H), 7.65 – 7.58 (m, 4H), 7.44 (t, *J* = 7.9 Hz, 4H), 7.40 – 7.36 (m, 1H), 6.80 (dd, *J* = 7.2, 1.9 Hz, 1H), 4.71 (s, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.52, 145.63, 144.98, 135.45, 133.98, 130.92, 128.88, 128.77, 128.54, 128.52, 128.14, 124.21, 116.42, 113.98, 113.90, 109.99, 34.61. HRMS (ESI-TOF) m/z: [M+H] $^{+}$ Calcd for $C_{21}H_{15}ClN_2O$ 347.0946; Found:347.0940.

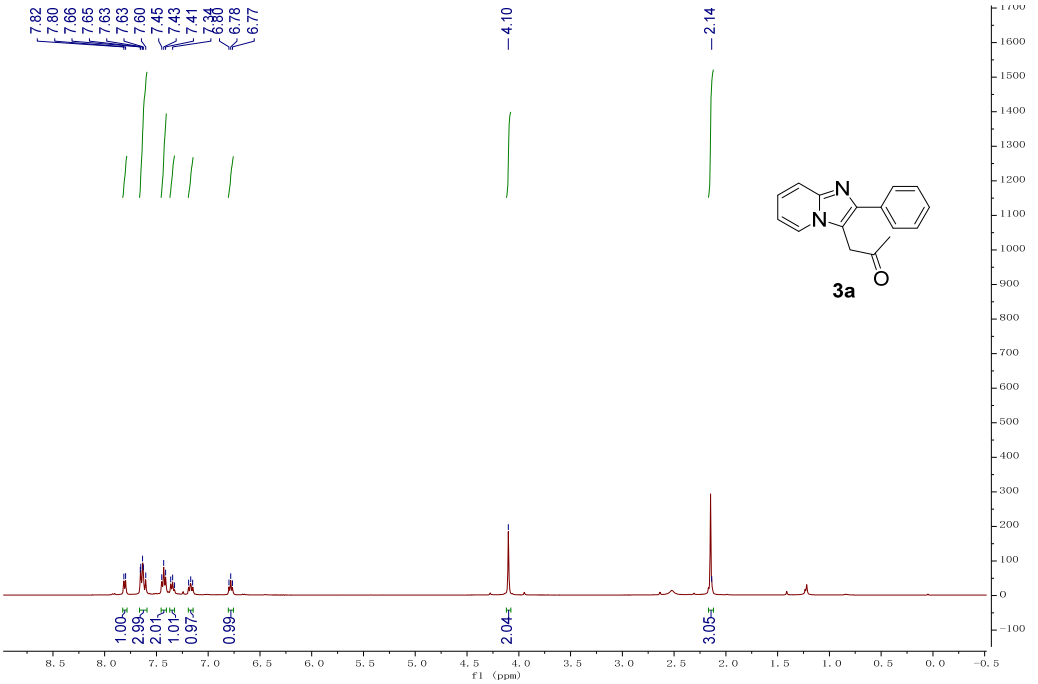
2-(2-phenylimidazo[1,2-a]pyridin-3-yl)cyclohexan-1-one **(3t)**

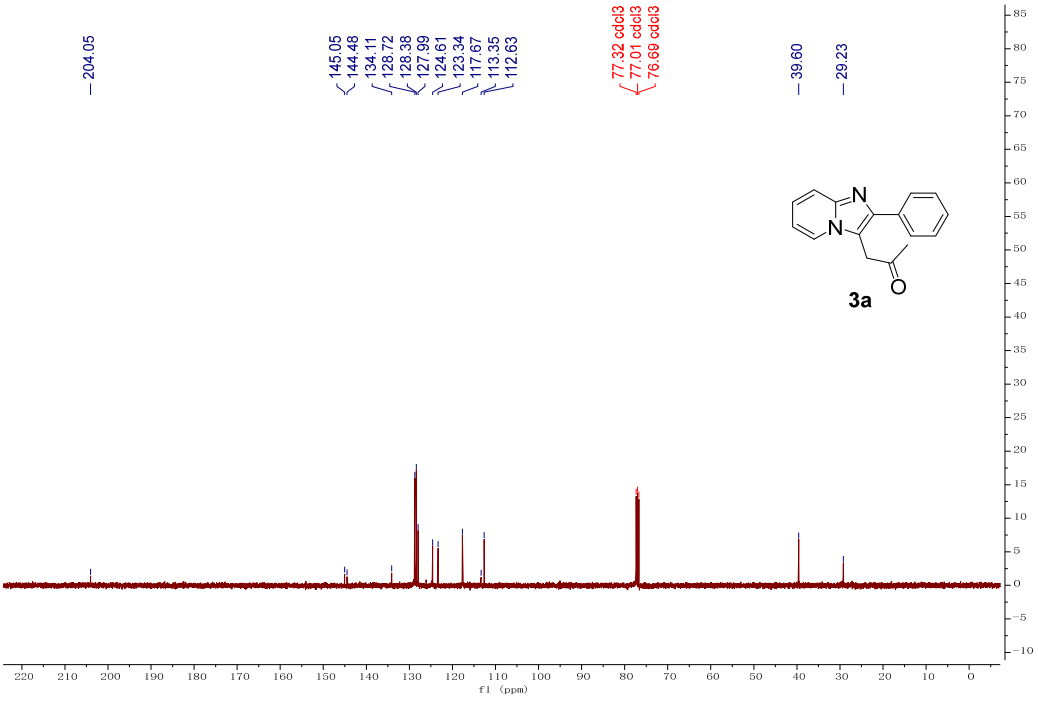
Purified by using a flash column chromatography; isolated yield = 39 %, 22.6 mg; yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 6.9 Hz, 1H), 7.64 (d, *J* = 8.9 Hz, 1H), 7.57 (d, *J* = 7.1 Hz, 2H), 7.45 (t, *J* = 7.3 Hz, 2H), 7.40 – 7.35 (m, 1H), 7.20 – 7.14 (m, 1H), 6.75 (t, *J* = 6.5 Hz, 1H), 4.35 – 4.29 (m, 1H), 2.67 – 2.49 (m, 2H), 2.27 – 2.19 (m, 2H), 2.10 – 1.97 (m, 2H), 1.89 – 1.80 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.38, 145.30, 134.82, 130.90, 130.27, 128.90, 128.58, 127.77, 125.26, 124.00, 117.94, 111.61, 48.31, 42.25, 31.17, 27.03, 25.37. HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{18}N_2O$ 291.1492; Found:291.1492.

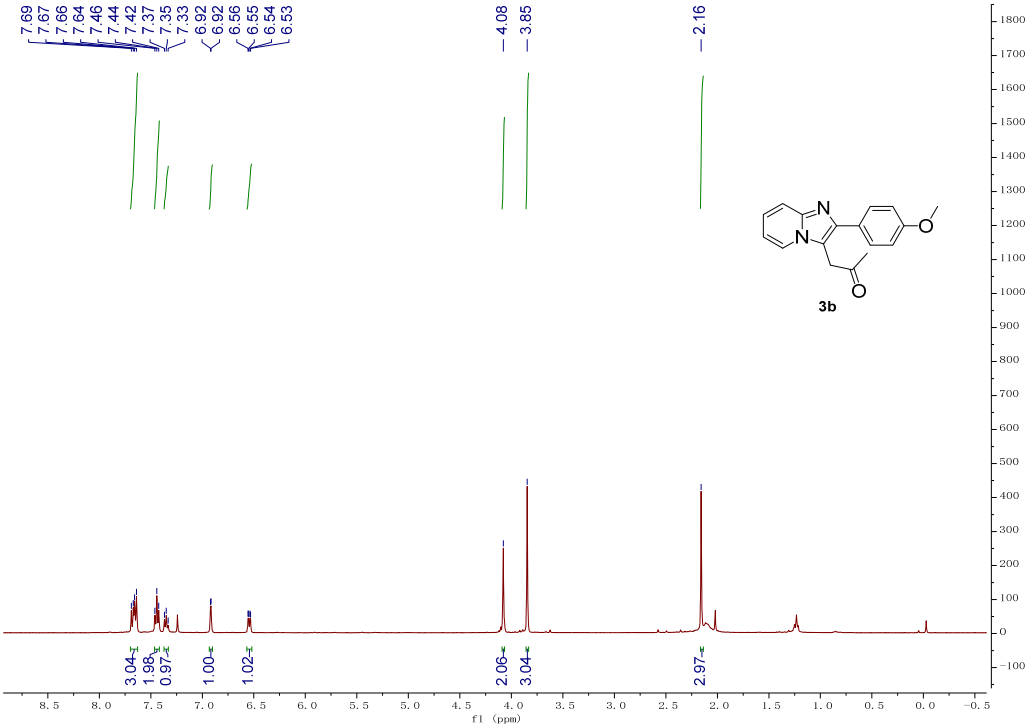
REFERENCE

(1) S.-C. Wang, X.-H. Huang, Z.-M. Ge, X. Wang, R.-T. Li. *RSC Adv.,* 2016, **6**, 68, 63532-63535.

4.^{1}H NMR and ^{13}C NMR for products



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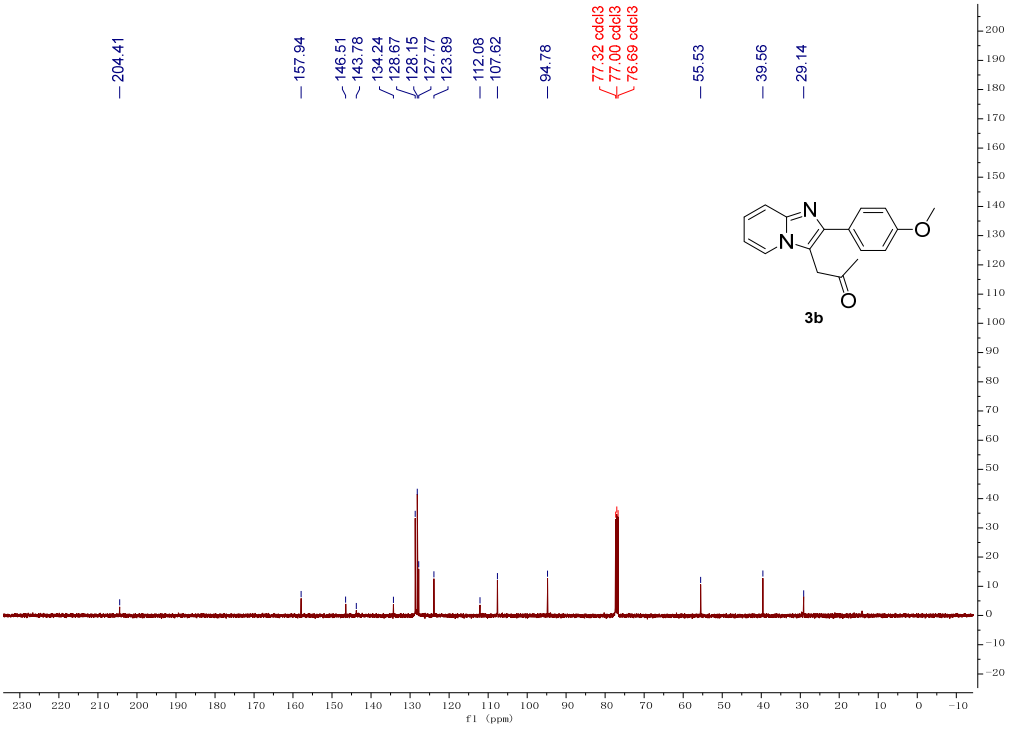
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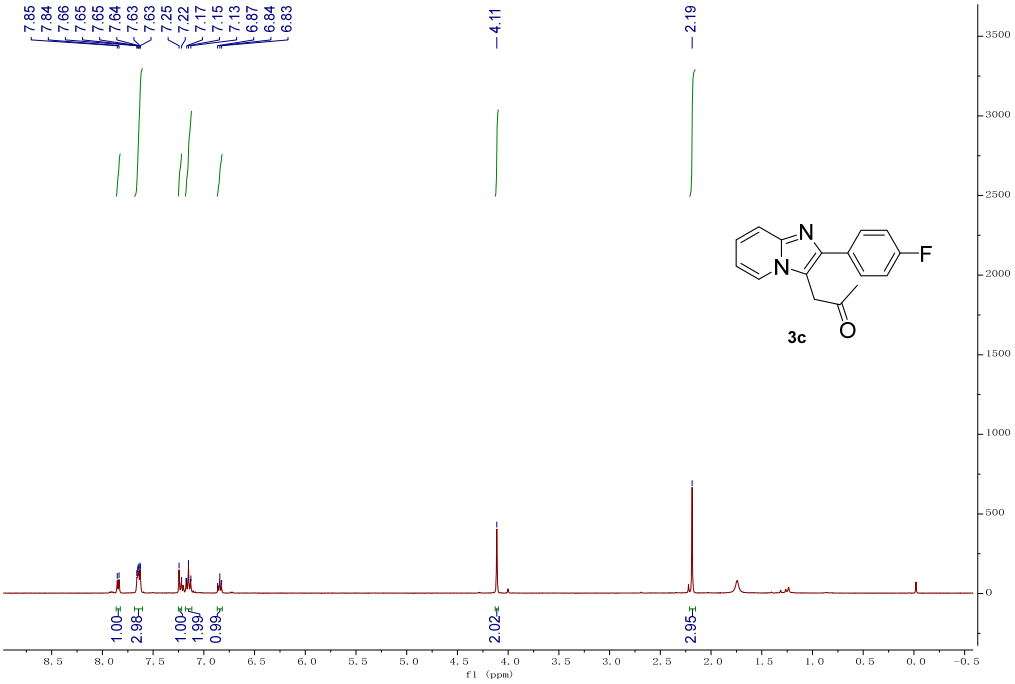
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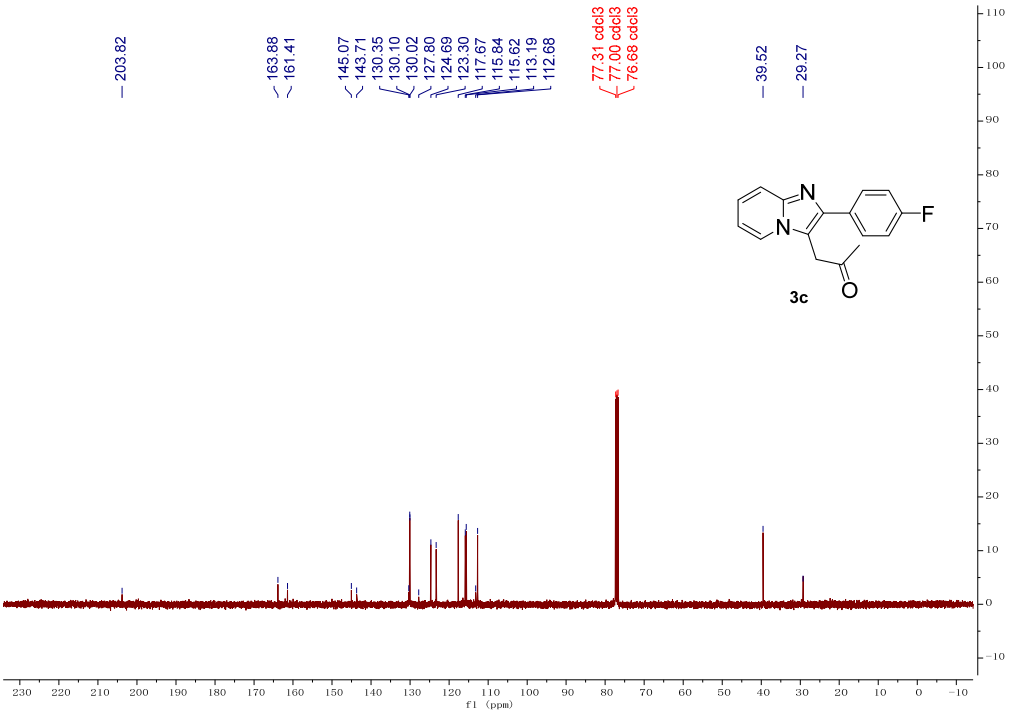
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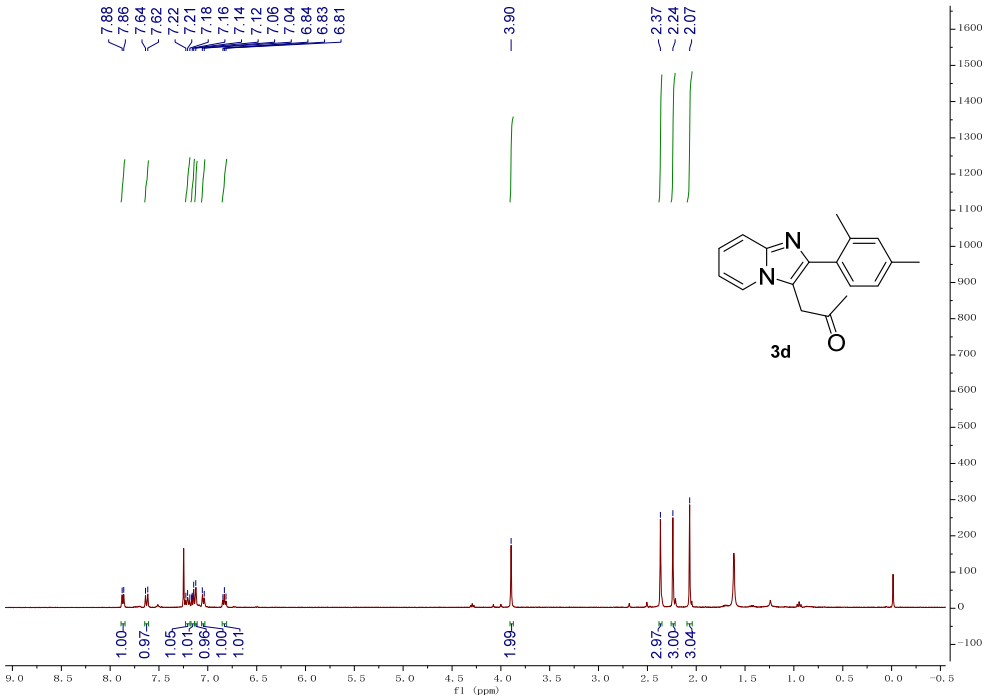
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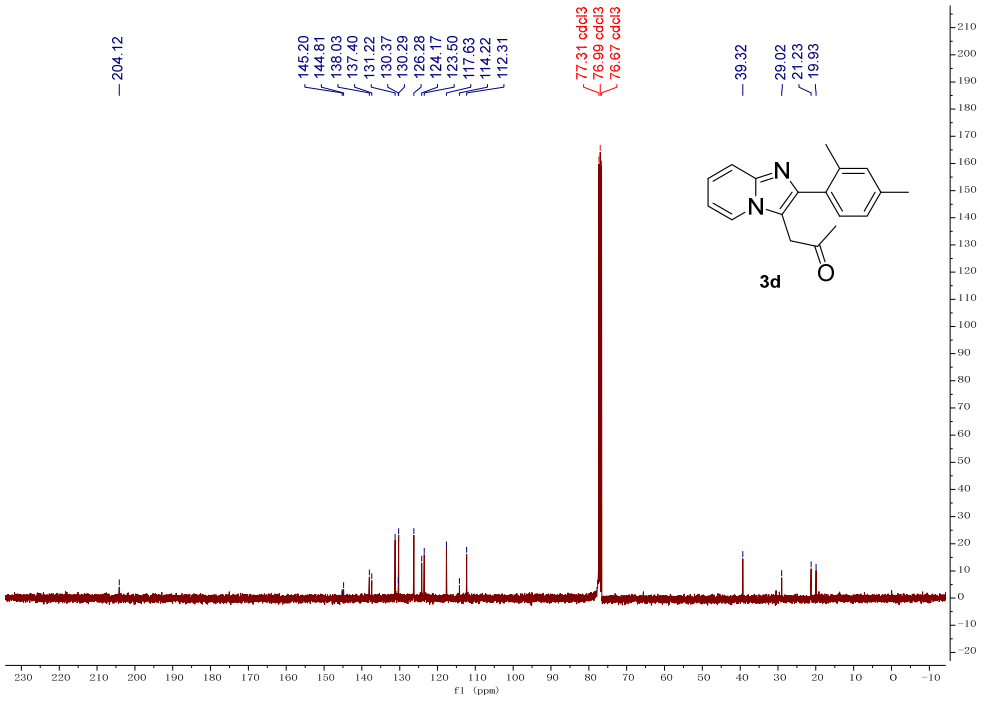
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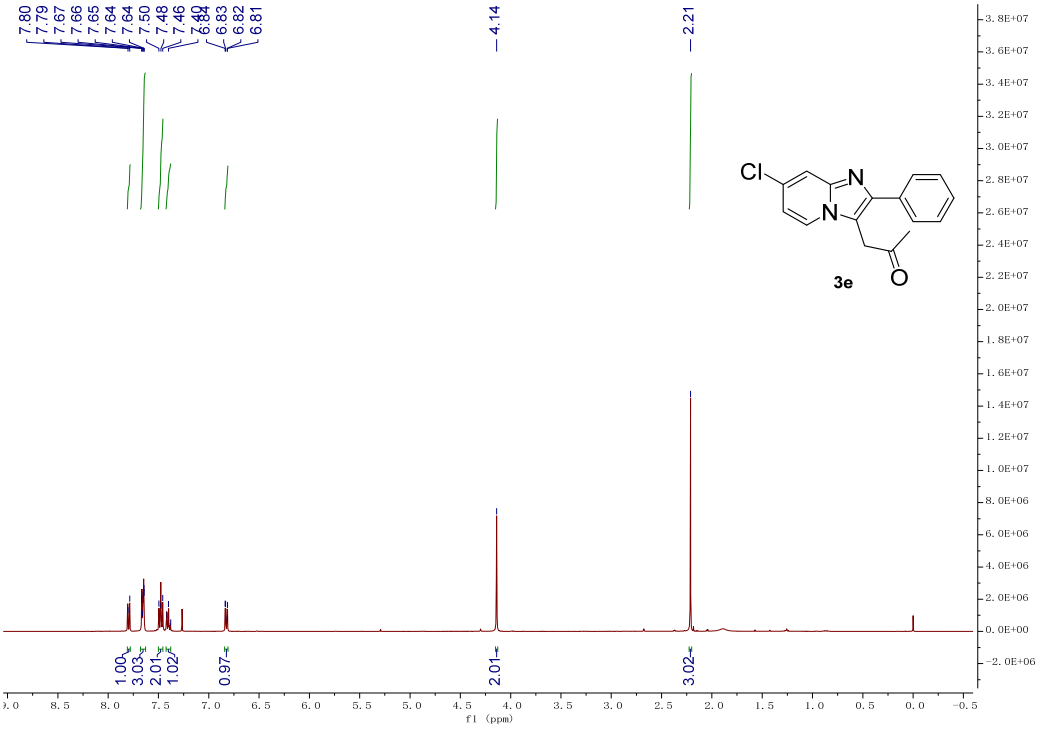
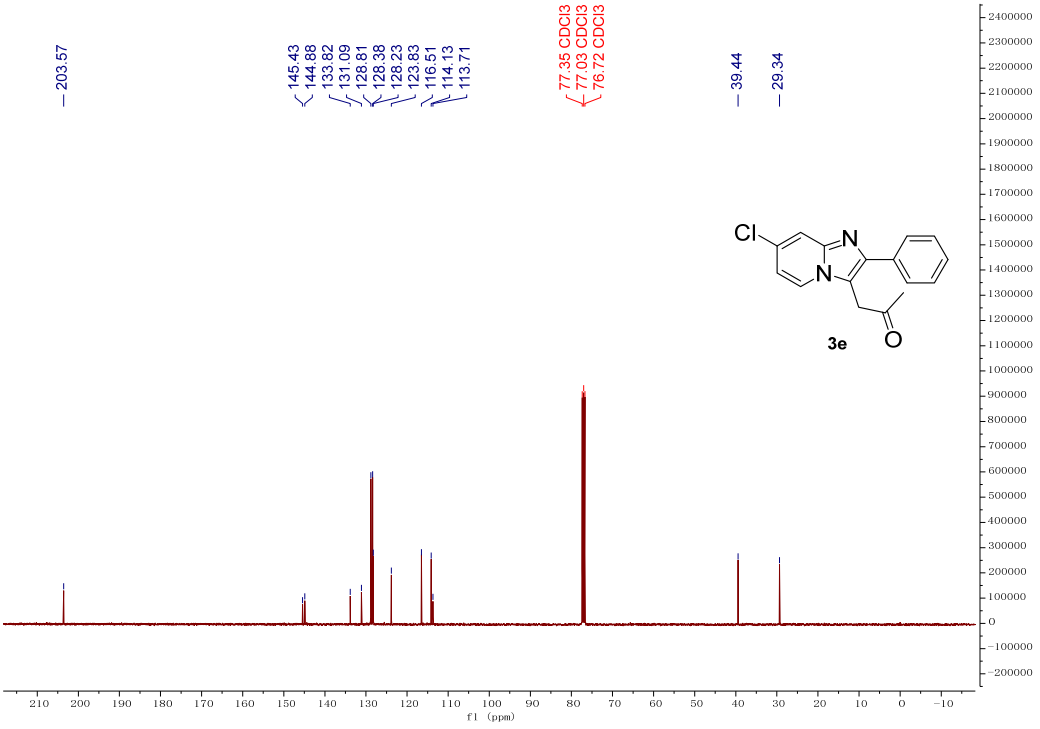
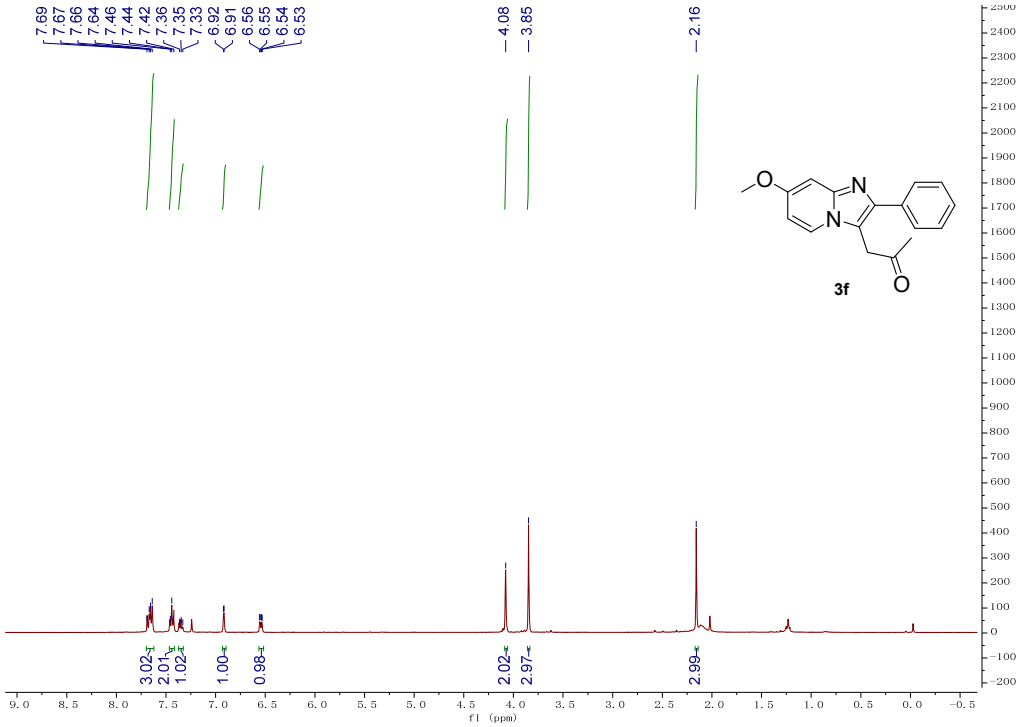
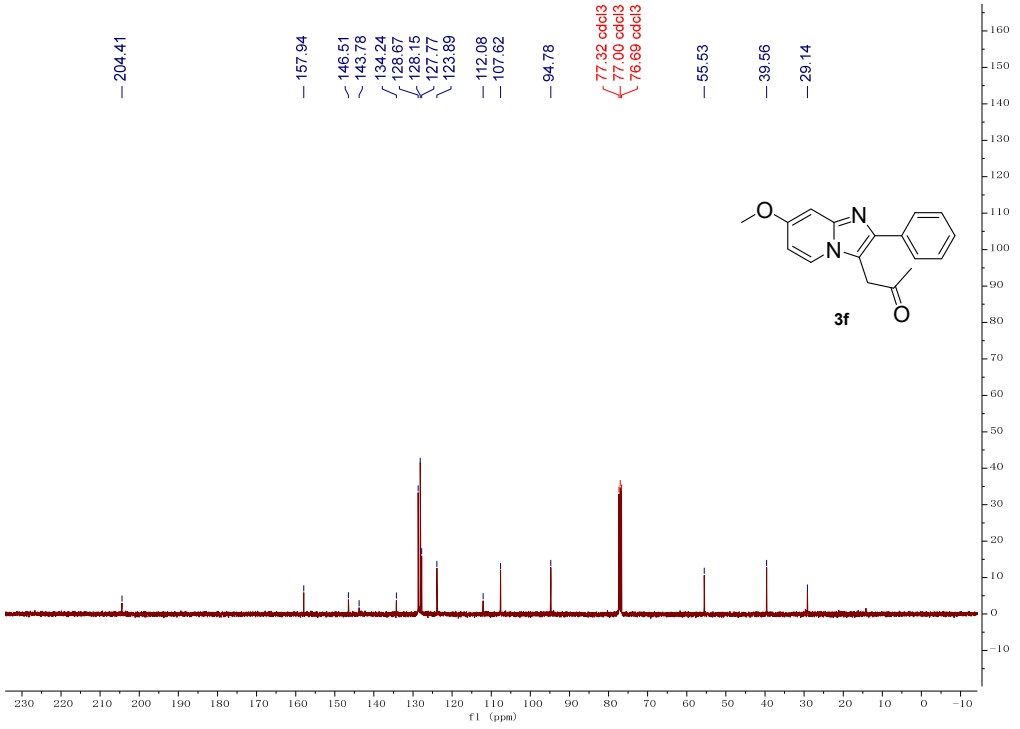
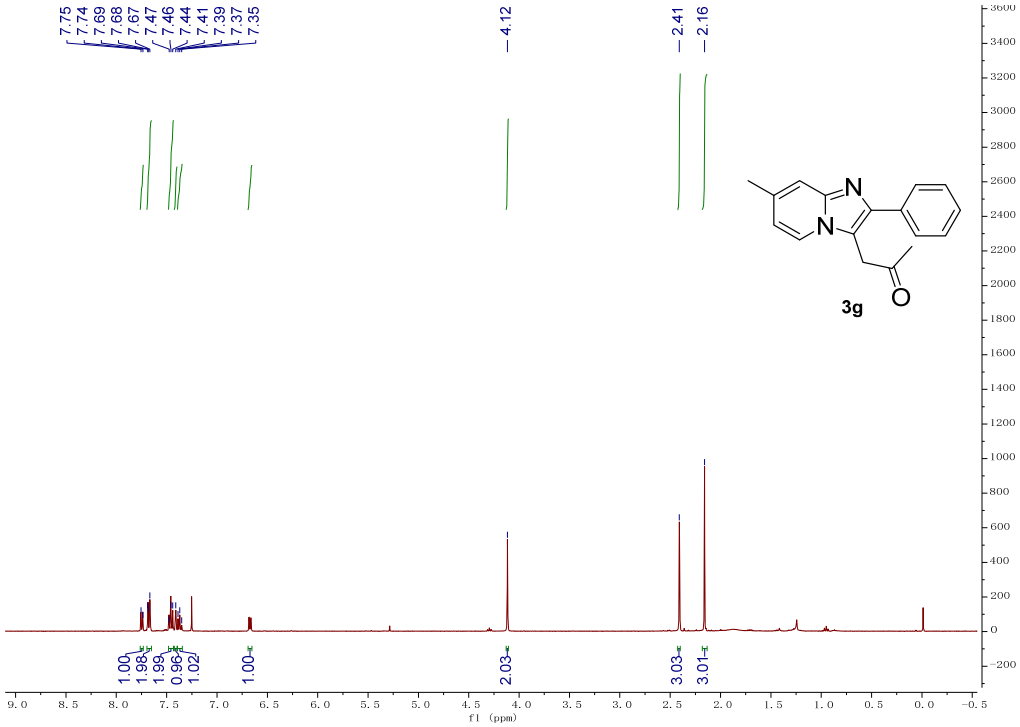

7.80
7.79
7.67
7.66
7.65
7.64
7.64
7.50
7.48
7.46
6.84
6.83
6.82
6.81
4.14
2.21
Cl
N
N
O
3e
1.00
3.03
2.01
1.02
0.97
2.01
3.02
f1 (ppm)

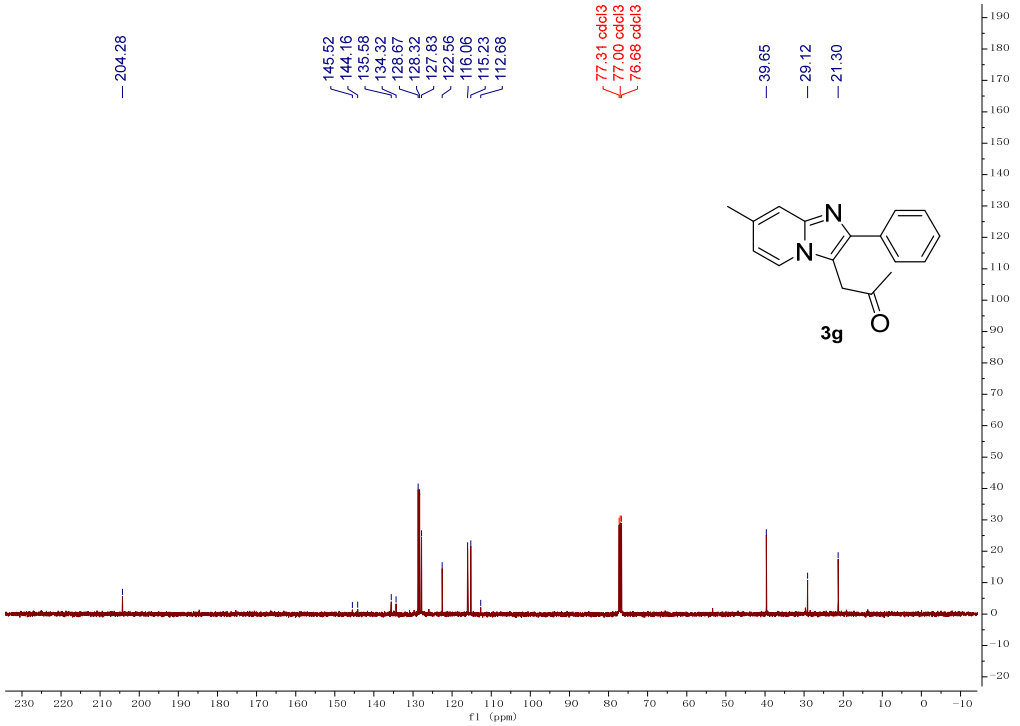

203.57
145.43
144.88
133.82
131.09
128.81
128.38
128.23
123.83
116.51
114.13
113.71
77.35 CDCl3
77.03 CDCl3
76.72 CDCl3
39.44
29.34
Cl
N
N
O
3e
f1 (ppm)


7.69
7.67
7.66
7.64
7.46
7.44
7.42
7.36
7.35
7.33
6.92
6.91
6.56
6.55
6.54
6.53
4.08
3.85
2.16
3.02
2.01
1.02
1.00
0.98
2.02
2.97
2.99
f1 (ppm)
3f

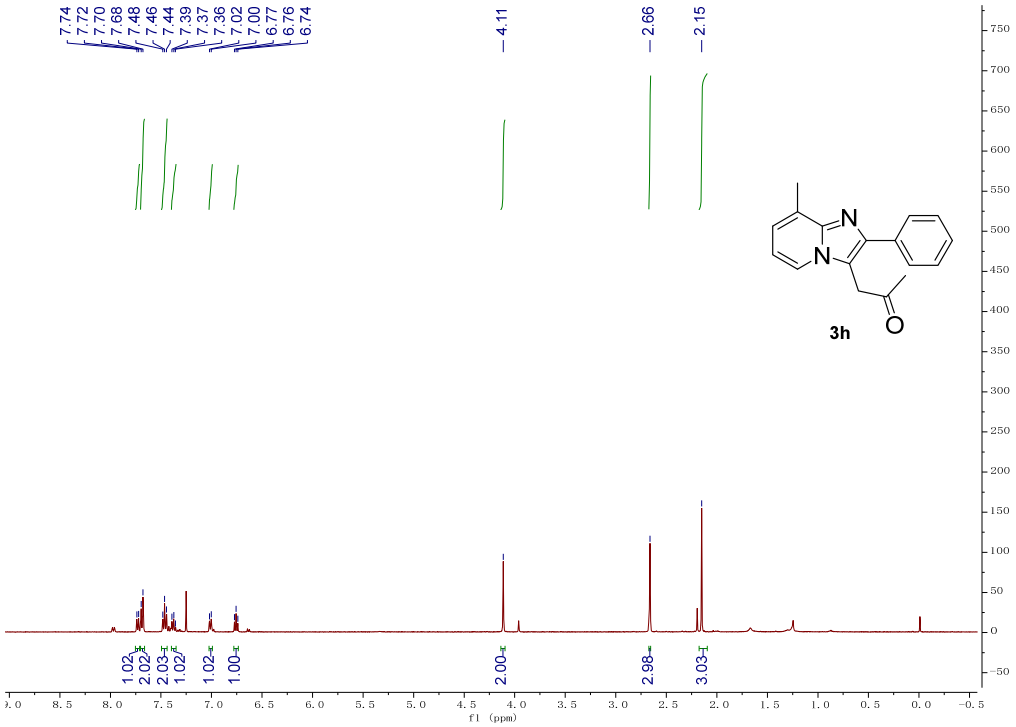

204.41
157.94
146.51
143.78
134.24
128.67
128.15
127.77
123.89
112.08
107.62
94.78
77.32 cdcl3
77.00 cdcl3
76.69 cdcl3
55.53
39.56
29.14
f1 (ppm)
3f

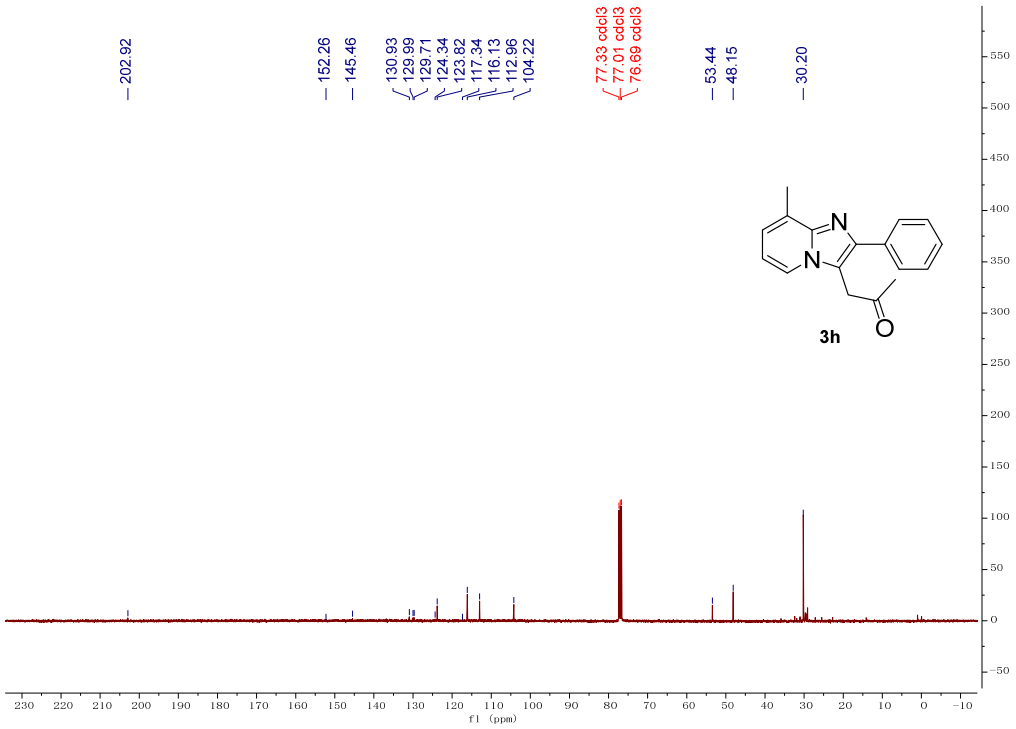

7.75
7.74
7.69
7.68
7.67
7.47
7.46
7.44
7.41
7.39
7.37
7.35
4.12
2.41
2.16
1.00
1.98
1.99
0.96
1.02
1.00
2.03
3.03
3.01
f1 (ppm)

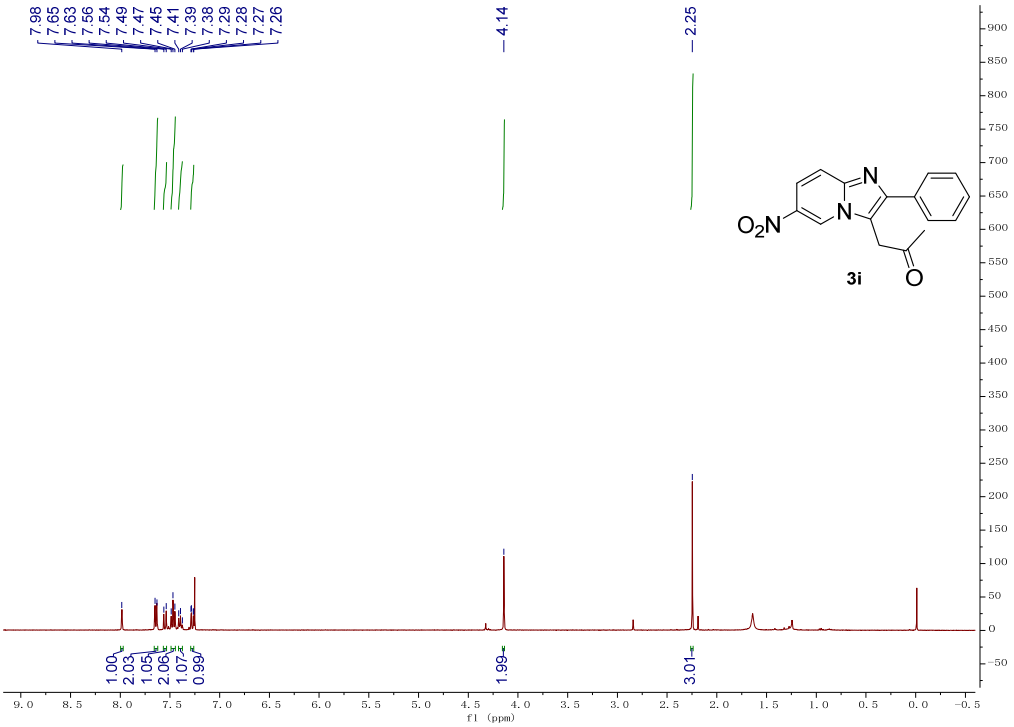
3g

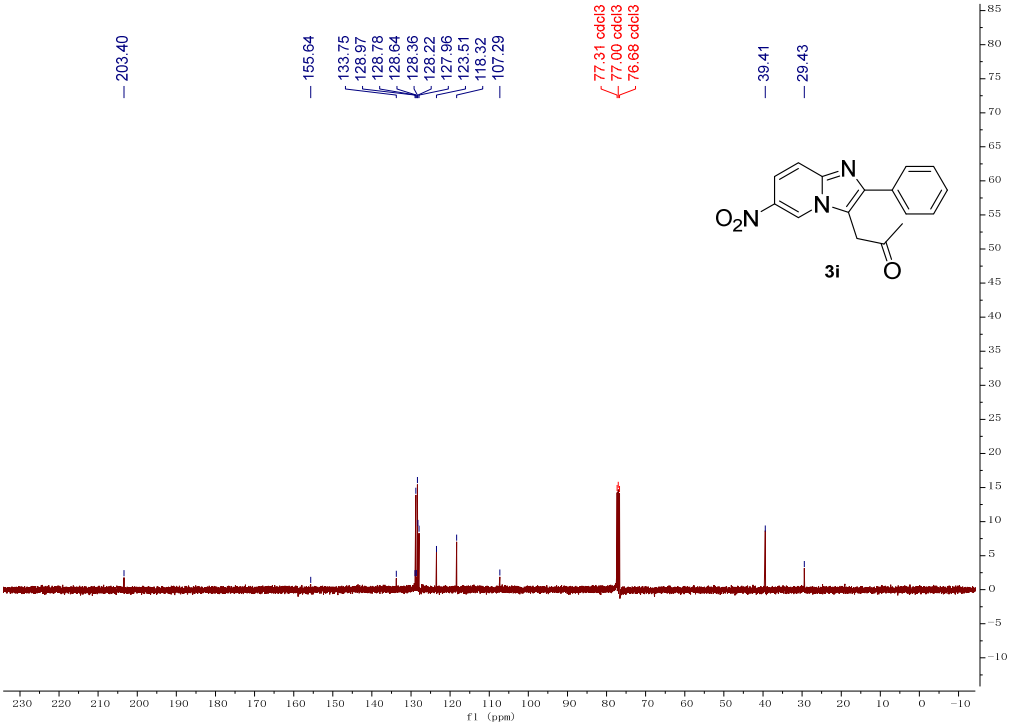

204.28
145.52
144.16
135.58
134.32
128.67
128.32
127.83
122.56
116.06
115.23
112.68
77.31 cdcl3
77.00 cdcl3
76.68 cdcl3
39.65
29.12
21.30
f1 (ppm)

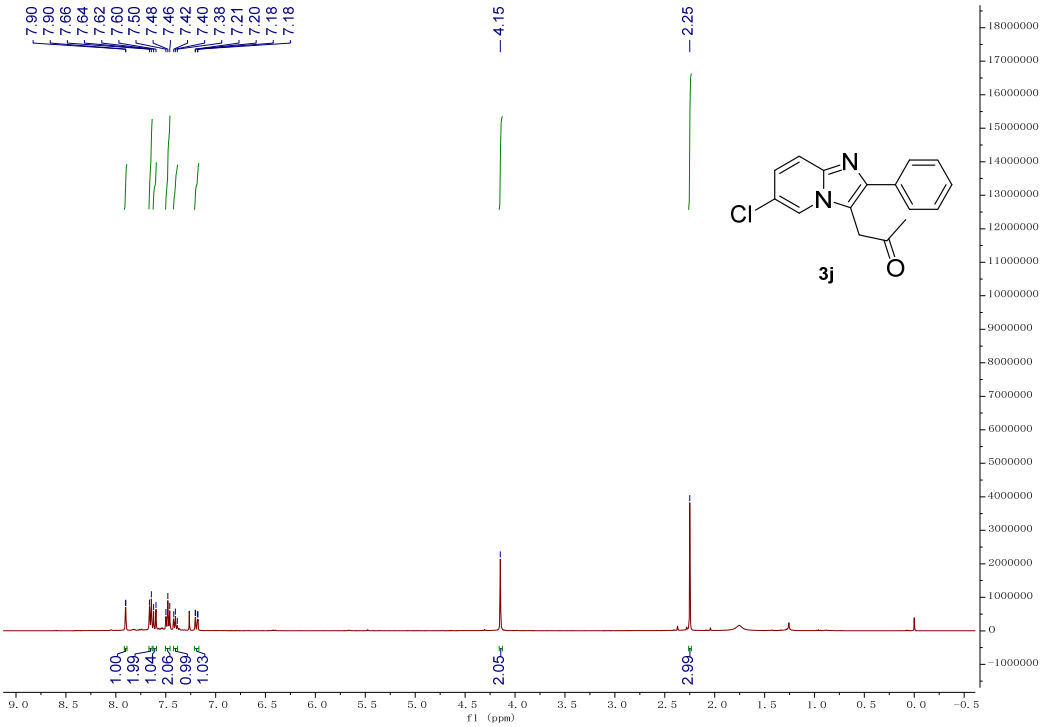
3g

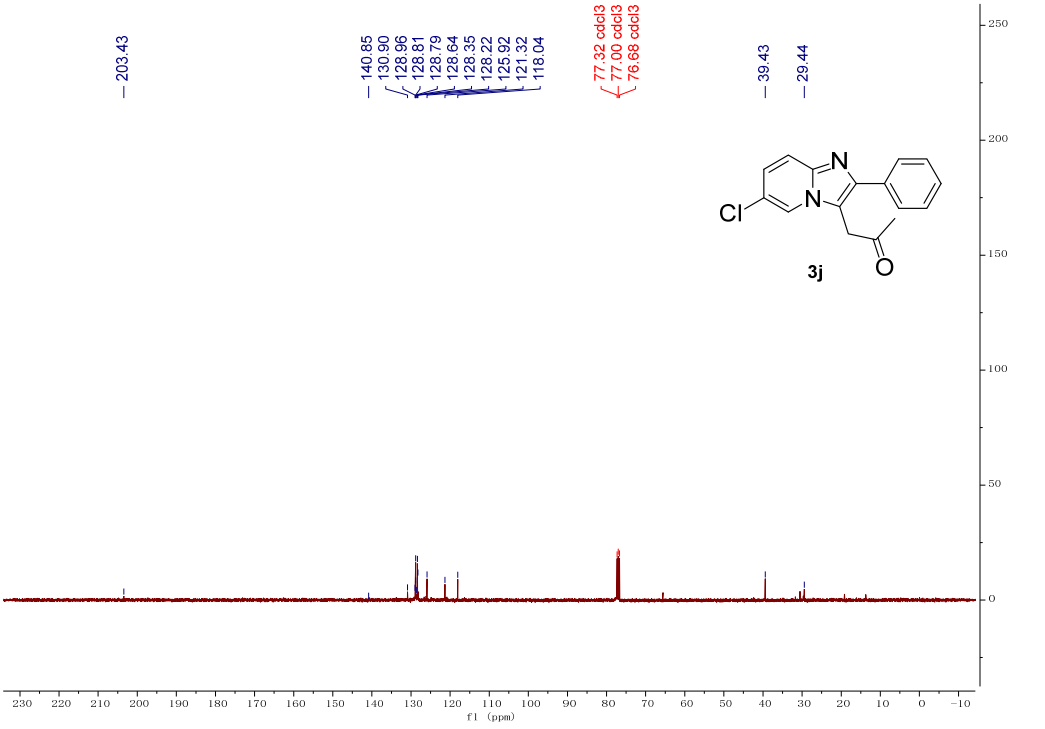

7.74
7.72
7.70
7.68
7.48
7.46
7.44
7.39
7.37
7.36
7.02
7.00
6.77
6.76
6.74
4.11
2.66
2.15
1.02
2.02
2.03
1.02
1.02
1.00
2.00
2.98
3.03
f1 (ppm)
3h

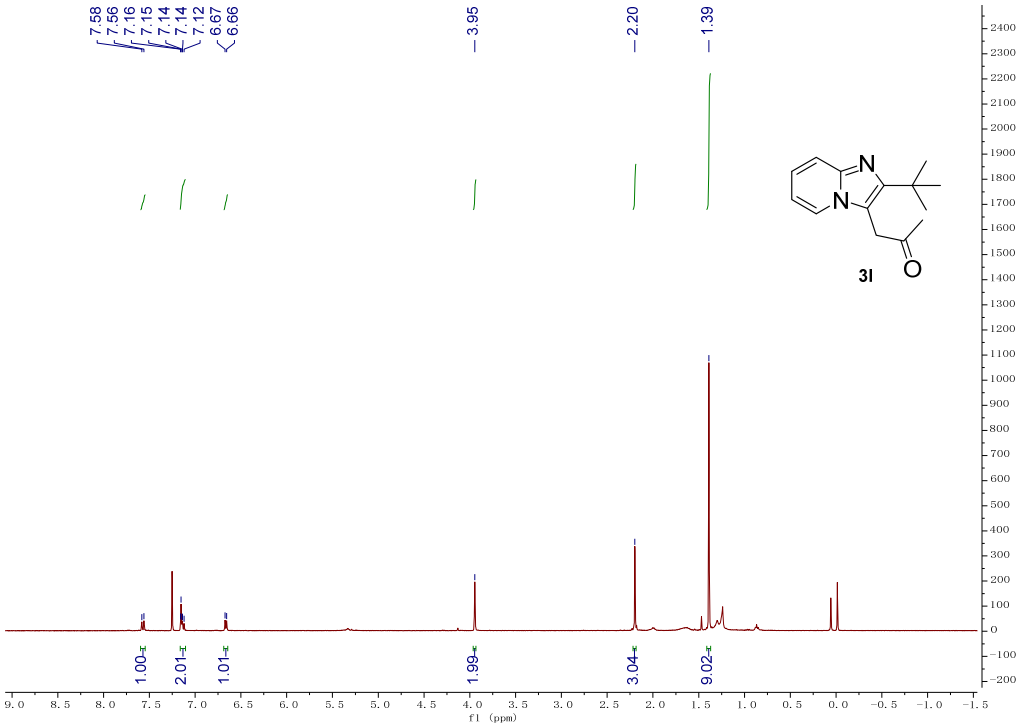

202.92
152.26
145.46
130.93
129.99
129.71
124.34
123.82
117.34
116.13
112.96
104.22
77.33 cdcl3
77.01 cdcl3
76.69 cdcl3
53.44
48.15
30.20
f1 (ppm)
3h

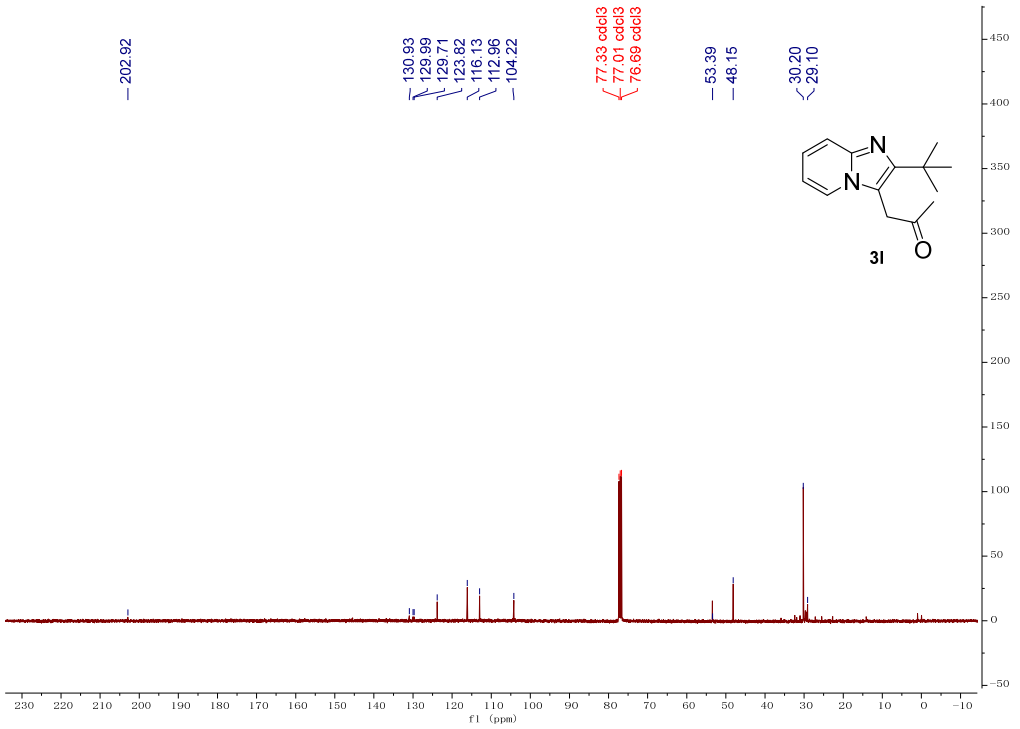

7.98
7.65
7.63
7.56
7.54
7.49
7.47
7.45
7.41
7.39
7.38
7.29
7.28
7.27
7.26
4.14
2.25
1.00
2.03
1.05
2.06
1.07
0.99
1.99
3.01
f1 (ppm)
3i

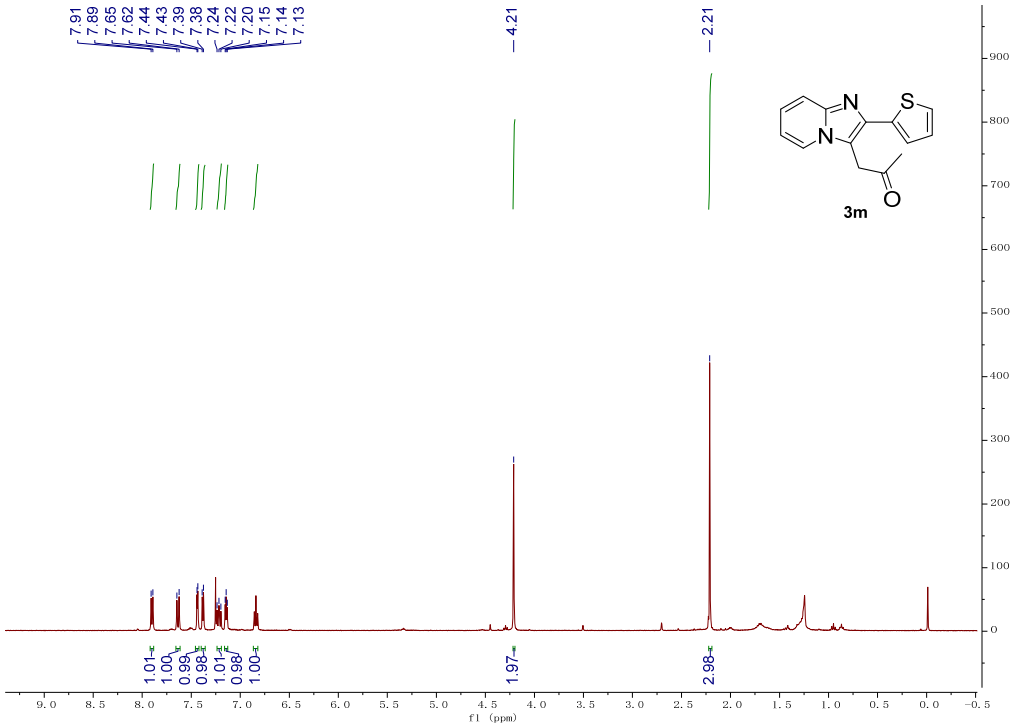

203.40
155.64
133.75
128.97
128.78
128.64
128.36
128.22
127.96
123.51
118.32
107.29
77.31 cdcl3
77.00 cdcl3
76.68 cdcl3
39.41
29.43
f1 (ppm)
3i


7.90
7.90
7.66
7.64
7.62
7.60
7.50
7.48
7.46
7.42
7.40
7.38
7.21
7.20
7.18
7.18
4.15
2.25
Cl
N
N
O
3j
1.00
1.99
1.04
2.06
0.99
1.03
2.05
2.99
f1 (ppm)


203.43
140.85
130.90
128.96
128.81
128.79
128.64
128.35
128.22
125.92
121.32
118.04
77.32 cdcl3
77.00 cdcl3
76.68 cdcl3
39.43
29.44
Cl
N
N
O
3j
f1 (ppm)

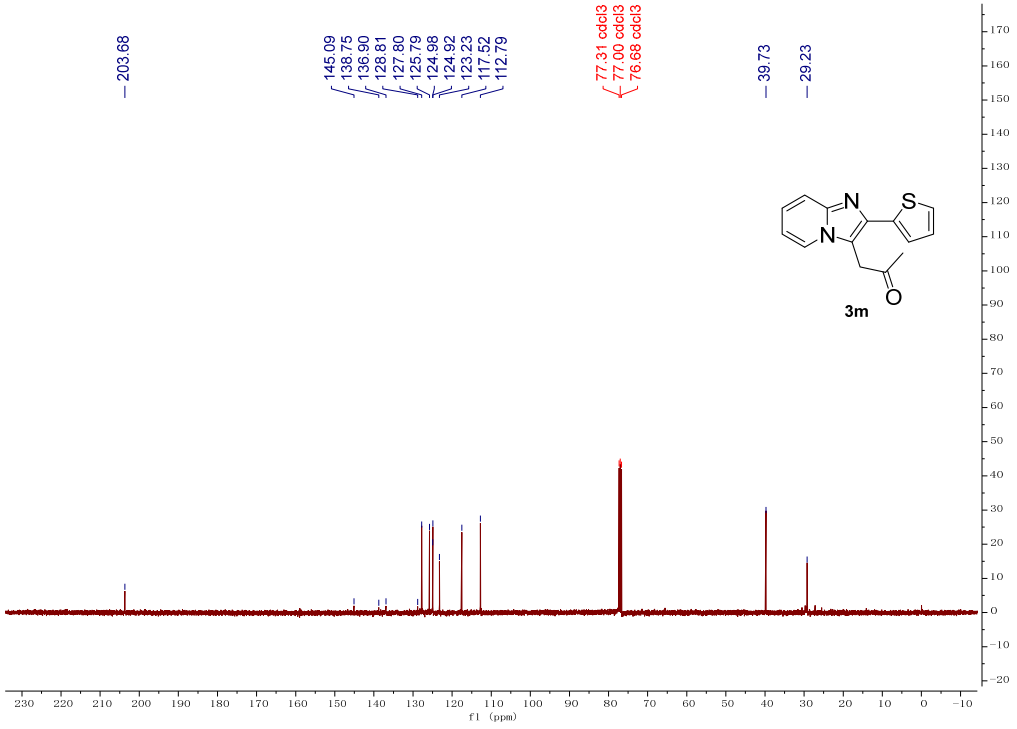

7.58
7.56
7.16
7.15
7.14
7.14
7.12
6.67
6.66
3.95
2.20
1.39
1.00
2.01
1.01
1.99
3.04
9.02
f1 (ppm)
3l


202.92
130.93
129.99
129.71
123.82
116.13
112.96
104.22
77.33 cdcl3
77.01 cdcl3
76.69 cdcl3
53.39
48.15
30.20
29.10
f1 (ppm)
3l


7.91
7.89
7.65
7.62
7.44
7.43
7.39
7.38
7.24
7.22
7.20
7.15
7.14
7.13
4.21
2.21
1.01
1.00
0.99
0.98
1.01
0.98
1.00
1.97
2.98
f1 (ppm)

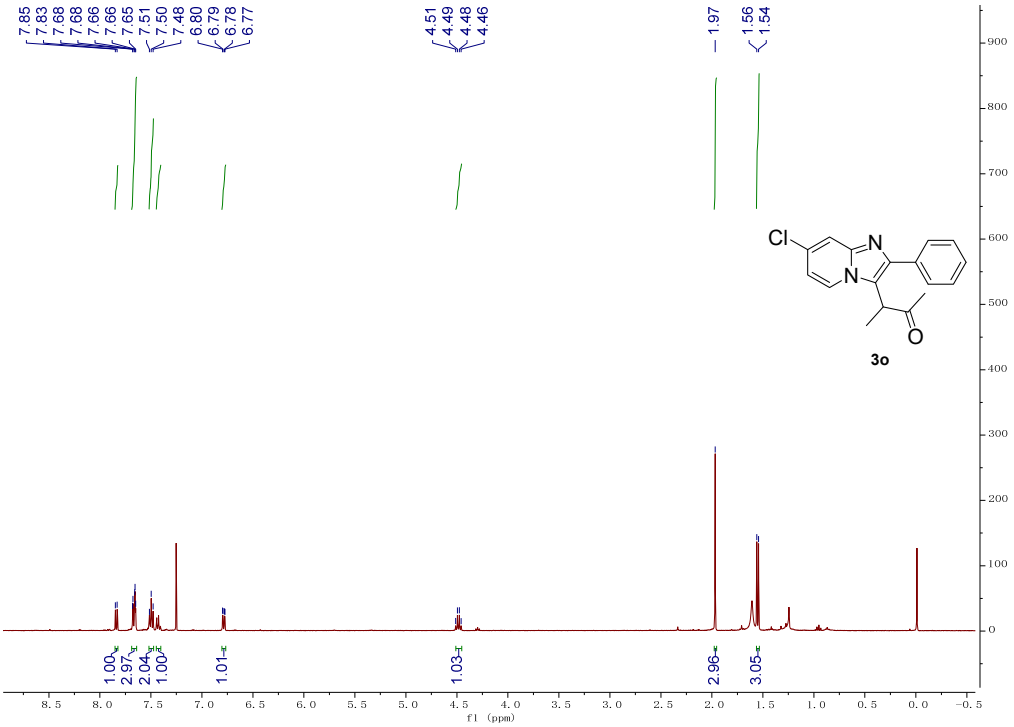
N
S
N
O

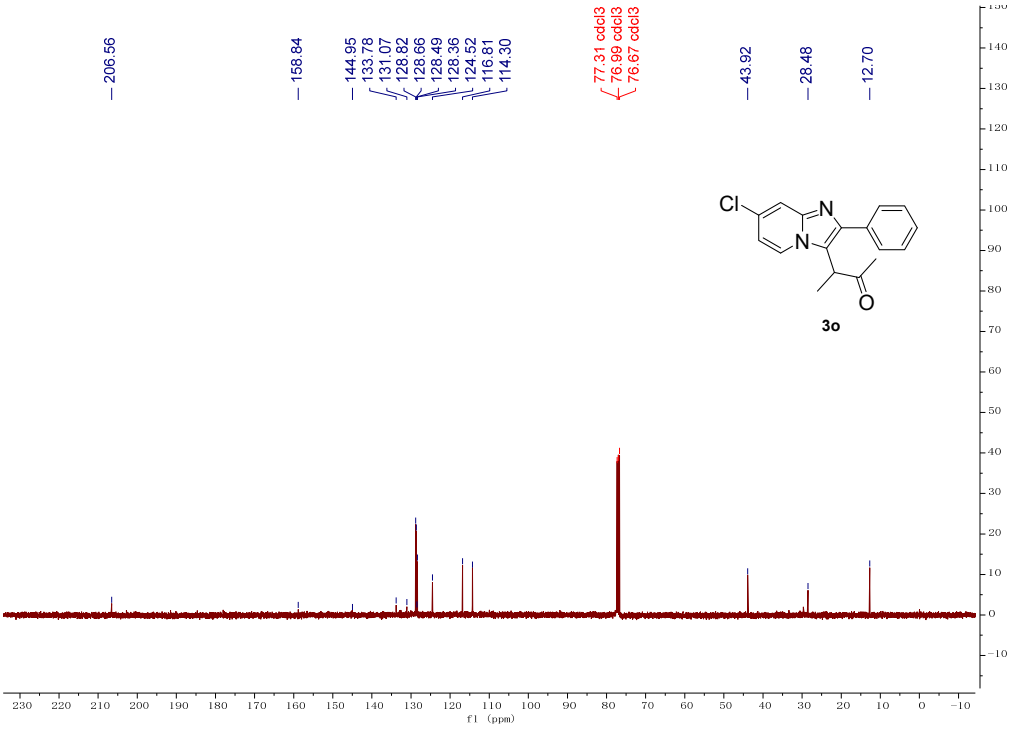
3m

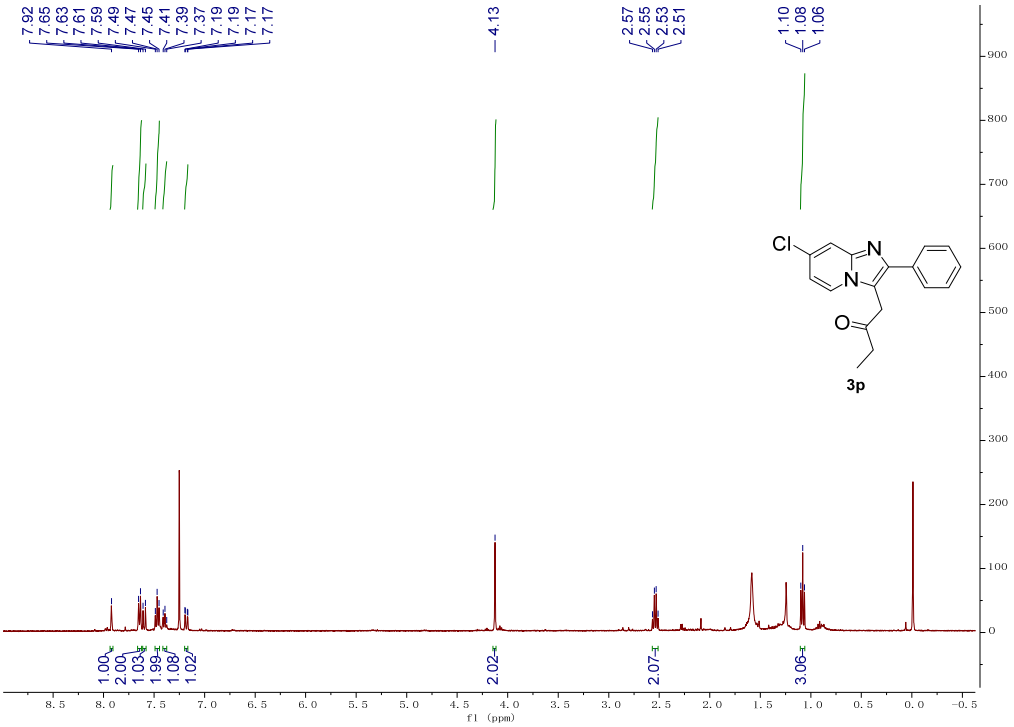

203.68
145.09
138.75
136.90
128.81
127.80
125.79
124.98
124.92
123.23
117.52
112.79
77.31 cdcl3
77.00 cdcl3
76.68 cdcl3
39.73
29.23
f1 (ppm)

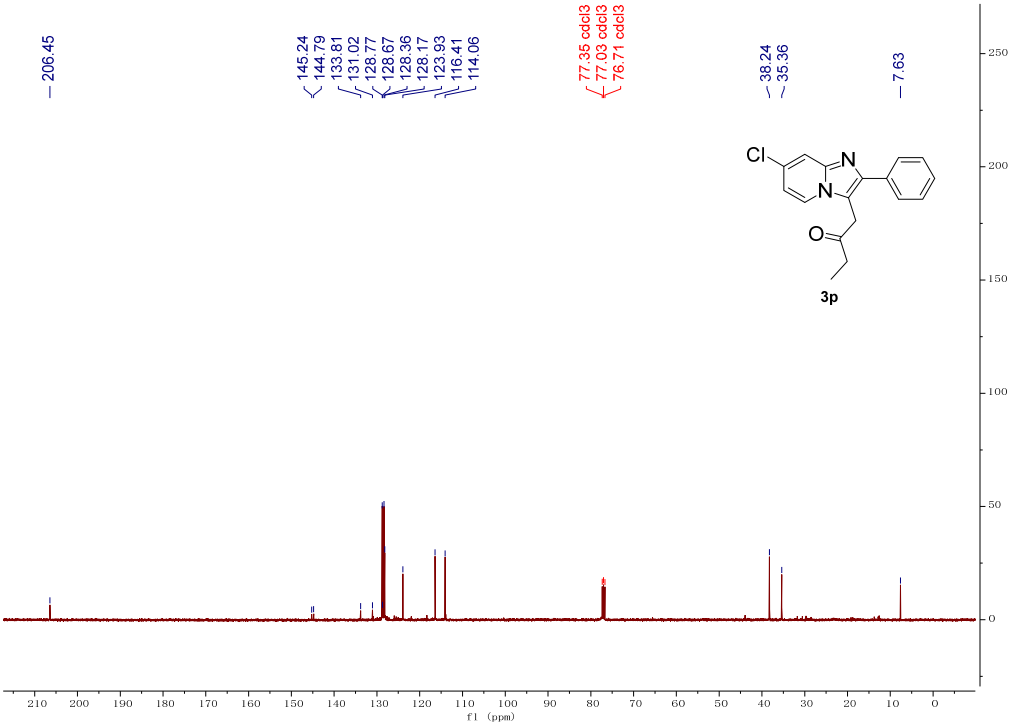
N
S
N
O

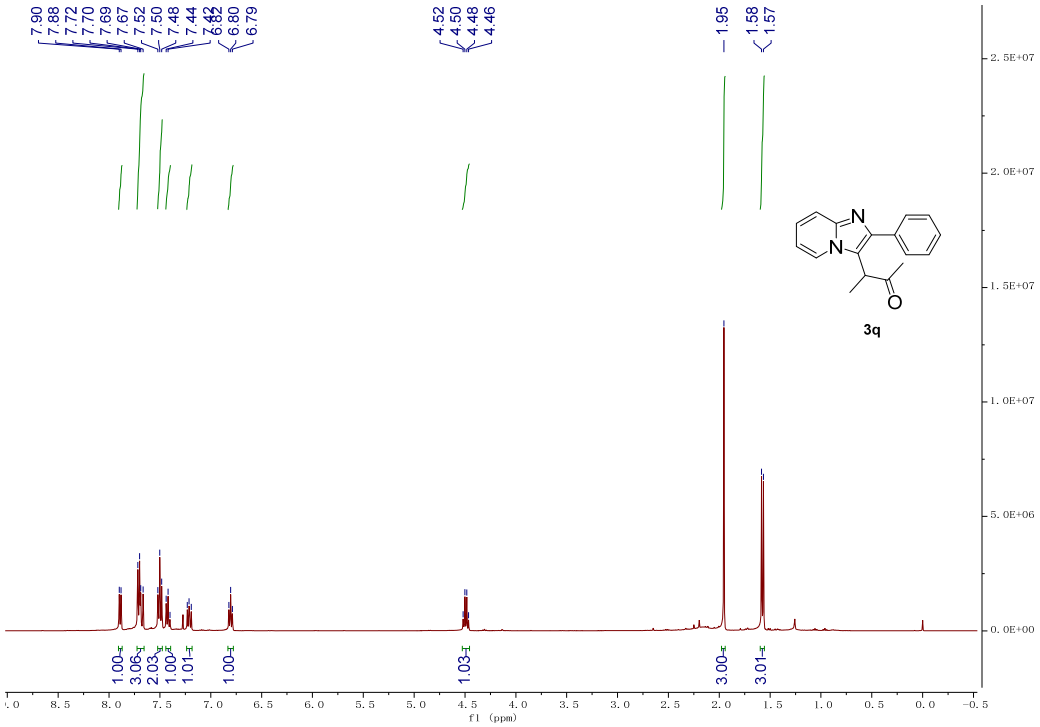
3m


7.85
7.83
7.68
7.68
7.66
7.66
7.65
7.51
7.50
7.48
6.80
6.79
6.78
6.77
4.51
4.49
4.48
4.46
1.97
1.56
1.54
Cl
N
N
O
3o
1.00
2.97
2.04
1.00
1.01
1.03
2.96
3.05
f1 (ppm)

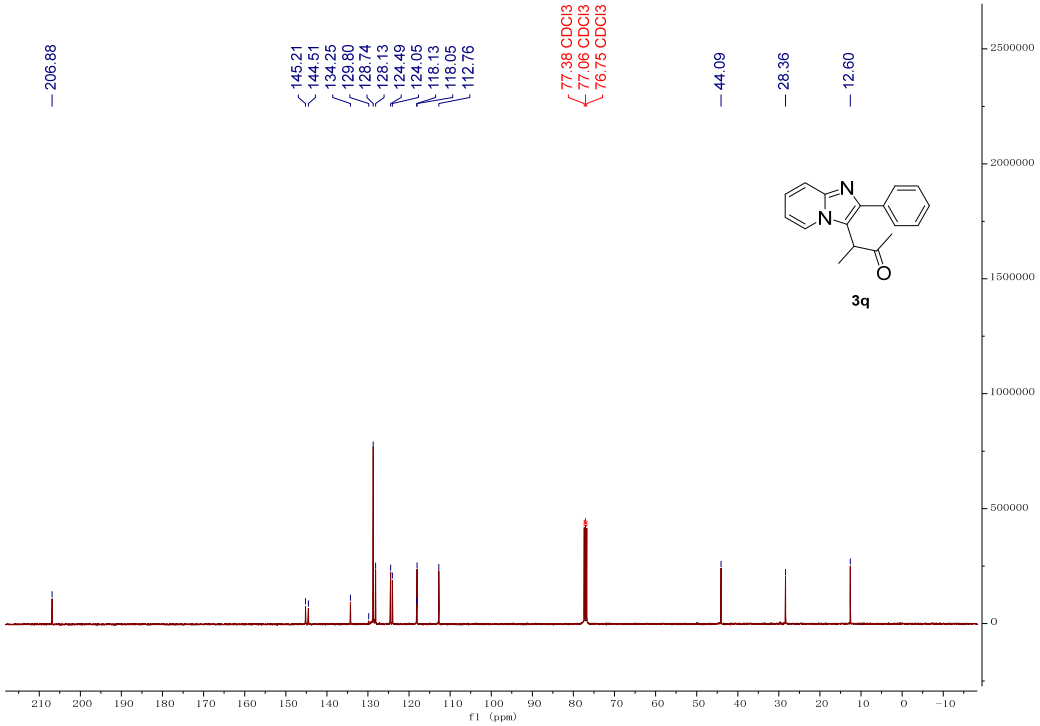

206.56
158.84
144.95
133.78
131.07
128.82
128.66
128.49
128.36
124.52
116.81
114.30
77.31 cdcl3
76.99 cdcl3
76.67 cdcl3
43.92
28.48
12.70
Cl
N
N
O
3o
f1 (ppm)


7.92
7.65
7.63
7.61
7.59
7.49
7.47
7.45
7.41
7.39
7.37
7.19
7.17
7.17
4.13
2.57
2.55
2.53
2.51
1.10
1.08
1.06
Cl
N
N
O
3p
1.00
2.00
1.03
1.99
1.08
1.02
2.02
2.07
3.06
f1 (ppm)

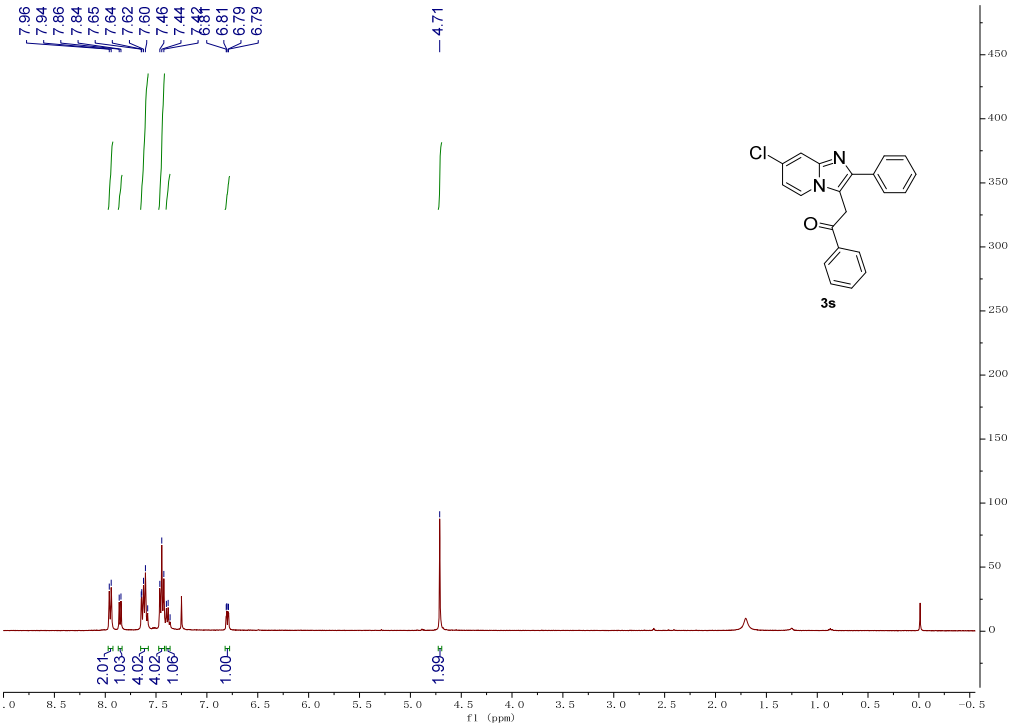

206.45
145.24
144.79
133.81
131.02
128.77
128.67
128.36
128.17
123.93
116.41
114.06
77.35 cdcl3
77.03 cdcl3
76.71 cdcl3
38.24
35.36
7.63
Cl
N
N
O
3p
f1 (ppm)

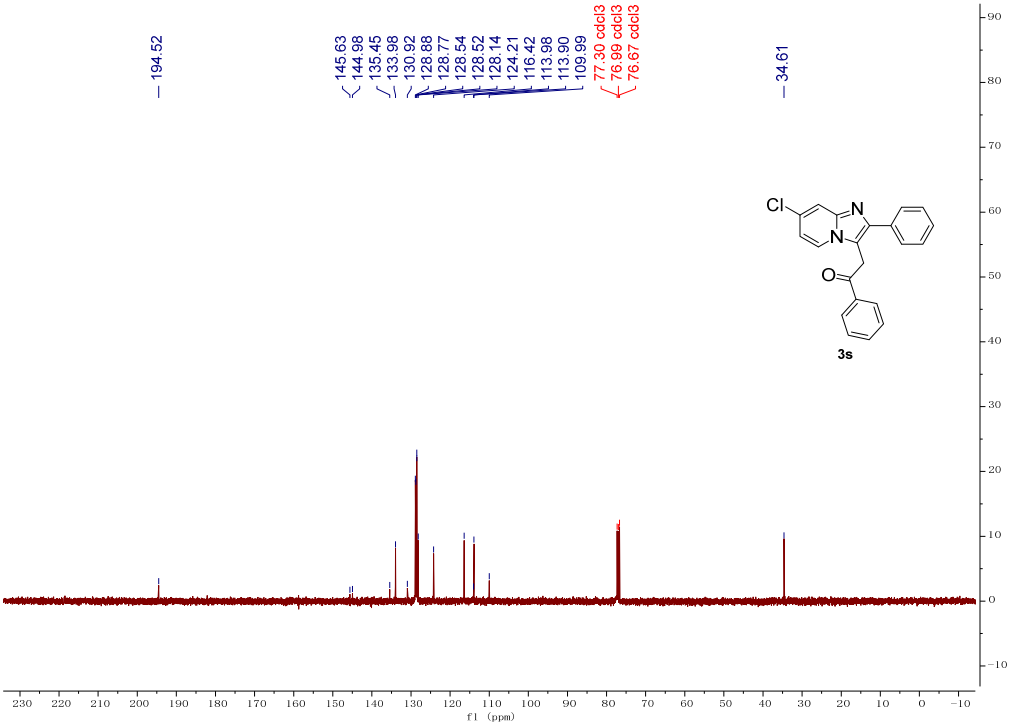

7.90
7.88
7.72
7.70
7.69
7.67
7.52
7.50
7.48
7.44
7.42
6.82
6.80
6.79
4.52
4.50
4.48
4.46
1.95
1.58
1.57
1.00
3.06
2.03
1.00
1.01
1.00
1.03
3.00
3.01
f1 (ppm)

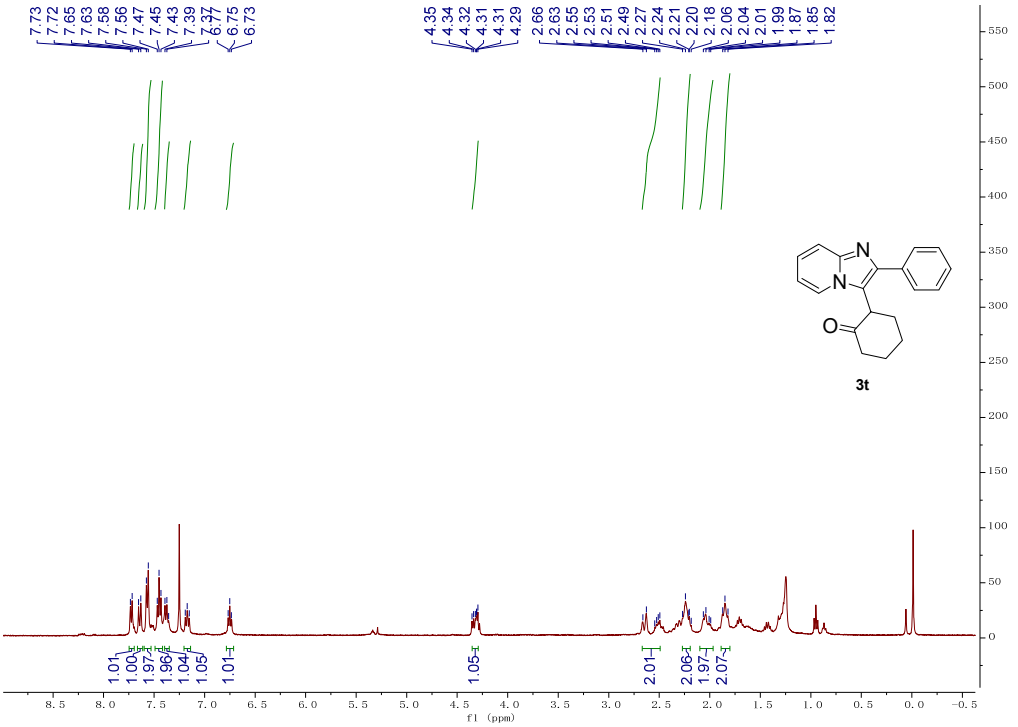
3q


206.88
145.21
144.51
134.25
129.80
128.74
128.13
124.49
124.05
118.13
118.05
112.76
77.38 CDCl3
77.06 CDCl3
76.75 CDCl3
44.09
28.36
12.60
f1 (ppm)

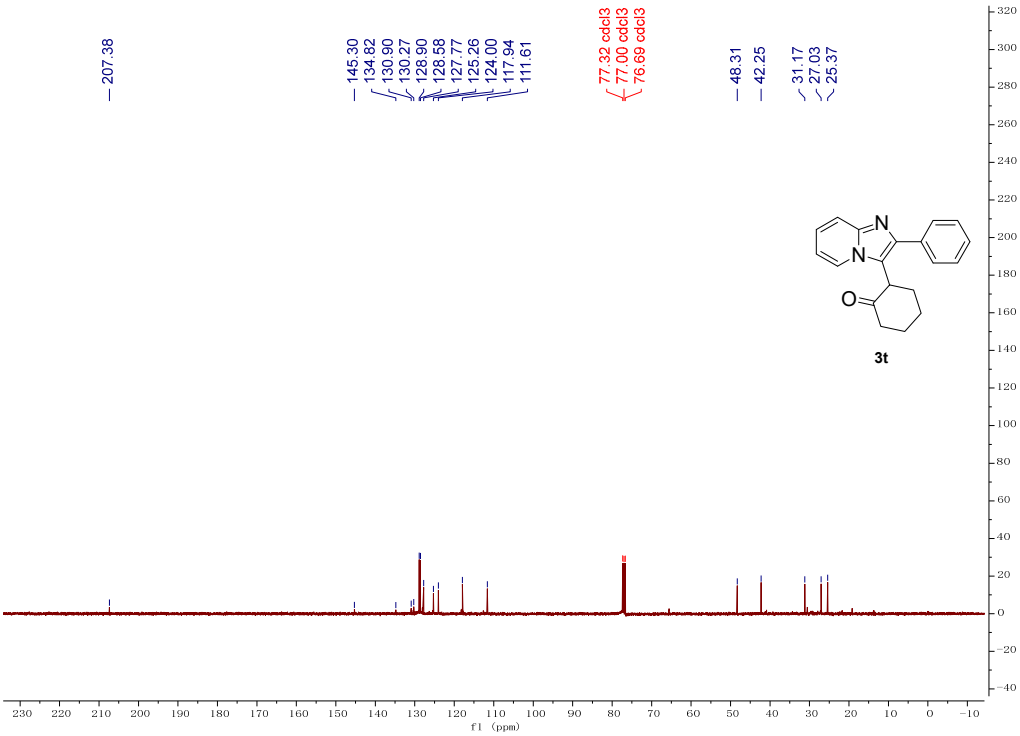
3q


7.96
7.94
7.86
7.84
7.65
7.64
7.62
7.60
7.46
7.44
7.42
6.81
6.79
6.79
4.71
Cl
N
N
O
3s
2.01
1.03
4.02
4.02
1.06
1.00
1.99
f1 (ppm)


194.52
145.63
144.98
135.45
133.98
130.92
128.88
128.77
128.54
128.52
128.14
124.21
116.42
113.98
113.90
109.99
77.30 cdcl3
76.99 cdcl3
76.67 cdcl3
34.61
Cl
N
N
O
3s
f1 (ppm)


7.73
7.72
7.65
7.63
7.58
7.56
7.47
7.45
7.43
7.39
7.37
6.77
6.75
6.73
4.35
4.34
4.32
4.31
4.29
2.66
2.63
2.55
2.53
2.51
2.49
2.27
2.24
2.21
2.20
2.18
2.06
2.04
2.01
1.99
1.87
1.85
1.82
1.01
1.00
1.97
1.96
1.04
1.05
1.01
1.05
2.01
2.06
1.97
2.07
f1 (ppm)

3t


207.38
145.30
134.82
130.90
130.27
128.90
128.58
127.77
125.26
124.00
117.94
111.61
77.32 cdcl3
77.00 cdcl3
76.69 cdcl3
48.31
42.25
31.17
27.03
25.37
f1 (ppm)

3t

5. GC-MS spectra of compounds 4 and 5