Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Copper–Catalyzed Transformation of Ketones to Amides via C(CO)–C(alkyl) Bond Cleavage Directed by Picolinamide

Haojie Ma, Xiaoqiang Zhou, Zhenzhen Zhan, Daidong Wei, Chong Shi, Xingxing Liu, Guosheng Huang*

State Key Laboratory of Applied Organic Chemistry, Key Laboratory of Nonferrous Metal Chemistry and Resources Utilization of Gansu Province, Department of Chemistry, Lanzhou University, Lanzhou, P. R. China.

E-mail: hgs@lzu.edu.cn

Experimental Section

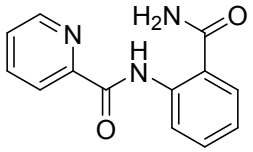
General experimental details

^{1}H-NMR spectra were recorded at 400 MHz. Chemical shifts (in ppm) were referenced to DMSO (δ = 2.50 ppm) in DMSO as an internal standard. ^{13}C-NMR spectra were obtained at 100 MHz and were calibrated with DMSO (δ = 39.50 ppm). The high resolution mass spectra (HRMS) were recorded on an FT-ICR mass spectrometer using electrospray ionization (ESI). Products were purified by flash chromatography on 200–300 mesh silica gels. Unless otherwise noted, commercially available reagents and solvents were used without further purification. All melting points were determined without correction.

Typical procedure for the preparation of N-(2-carbamoylphenyl)picolinamide (2a)

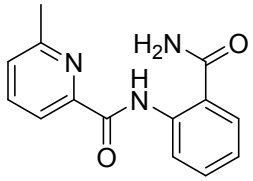
A test tube was charged with **1a** (0.2mmol), NaN_3 (0.6 mmol), $Cu(OAc)_2$ (20 mol %), AcOH (4 equiv) in DMSO (1 mL). The reaction tube was evacuated and back-filled with O_2 (3 times, balloon).Then the reaction mixture was stirred at 80 ^{o}C (oil bath temperature) under O_2 atmosphere. After cooling to room temperature, the solvent was extracted with ethyl acetate and washed with brine, dried with Na_2SO_4. After the solvent was evaporated in vacuo, the residues were purified by column chromatography, eluting with petroleum ether / ethyl acetate to afford pure **2a**.

Analytical data for products



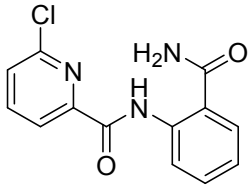
N-(2-carbamoylphenyl)picolinamide (2a).

White solid (41.0 mg, yield 85%), m.p. 219-221 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 13.08 (s, 1H), 8.73 (m, 2H), 8.30 – 8.20 (m, 1H), 8.17 (dd, *J* = 7.8, 1.0 Hz, 1H), 8.06 (m, 1H), 7.80 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.67 (m, 2H), 7.59 – 7.53 (m, 1H), 7.19 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 170.3, 162.6, 149.8, 148.7, 138.5, 138.1, 131.9, 128.6, 127.0, 122.9, 122.3, 121.6, 120.3. HRMS calcd for $C_{13}H_{12}N_3O_2$ $[M+H]^+$ 242.0924, found 242.0927.



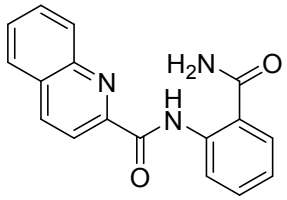
N-(2-carbamoylphenyl)-6-methylpicolinamide (2b).

White solid (41.8 mg, 82%), m.p. 239-241 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 13.23 (s, 1H), 8.77 (dd, *J* = 8.3, 0.7 Hz, 1H), 8.19 (d, *J* = 22.4 Hz, 1H), 7.94 (m, 2H), 7.82 (m, 1H), 7.77 (s, 1H), 7.58 – 7.52 (m, 1H), 7.51 (d, *J* = 6.9 Hz, 1H), 7.17 (m, 1H), 2.59 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 170.4, 162.8, 157.2, 149.1, 138.8, 138.1, 132.0, 128.6, 126.4, 122.8, 121.3, 120.2, 119.4, 23.8. HRMS calcd for $C_{14}H_{14}N_3O_2$ $[M+H]^+$ 256.1081, found 256.1082.



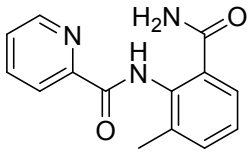
N-(2-carbamoylphenyl)-6-chloropicolinamide (2c).

White solid (28.6 mg, 52%), m.p. 243-245 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.23 (s, 1H), 8.75 (dd, *J* = 8.3, 0.7 Hz, 1H), 8.30 (d, *J* = 25.6 Hz, 1H), 8.17 – 8.09 (m, 2H), 7.82 (m, 3H), 7.60 – 7.54 (m, 1H), 7.20 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 170.4, 161.2, 150.5, 149.2, 141.6, 138.6, 132.1, 128.6, 127.6, 123.1, 121.6, 120.9, 120.2. HRMS calcd for $C_{13}H_{11}ClN_3O_2$ $[M+H]^+$ 276.0535, found 276.0539.



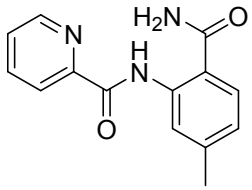
N-(2-carbamoylphenyl)quinoline-2-carboxamide (2d).

White solid (44.2 mg, 76%), m.p. 266-268 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.52 (s, 1H), 8.82 (dd, *J* = 8.4, 0.9 Hz, 1H), 8.66 – 8.60 (m, 1H), 8.34 – 8.22 (m, 2H), 8.15 – 8.08 (m, 2H), 7.94 – 7.85 (m, 2H), 7.79 (d, *J* = 9.1 Hz, 1H), 7.75 (m, 1H), 7.62 – 7.56 (m, 1H), 7.24 – 7.17 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 170.5, 162.7, 149.9, 145.8, 138.8, 138.2, 132.1, 130.7, 129.2, 128.9, 128.7, 128.4, 128.1, 122.9, 121.2, 120.2, 118.6. HRMS calcd for $C_{17}H_{14}N_3O_2$ $[M+H]^+$ 292.1081, found 292.1080.



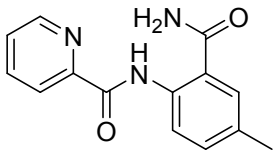
N-(2-carbamoyl-6-methylphenyl)picolinamide (2e).

White solid (27.0 mg, 53%), m.p. 171-173 °C. ^{1}H NMR (400 MHz, DMSO) δ 11.01 (s, 1H), 8.74 (m, 1H), 8.13 (d, *J* = 7.8 Hz, 1H), 8.05 (m, 1H), 7.91 (s, 1H), 7.67 (m, 1H), 7.42 (dd, *J* = 13.5, 8.1 Hz, 3H), 7.26 (t, *J* = 7.6 Hz, 1H), 2.24 (d, *J* = 9.1 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 169.7, 161.9, 149.4, 148.7, 138.0, 135.4, 133.9, 132.4, 132.0, 127.0, 125.7, 125.6, 122.2, 18.8. HRMS calcd for $C_{14}H_{14}N_3O_2$ $[M+H]^+$ 256.1081, found 256.1083.



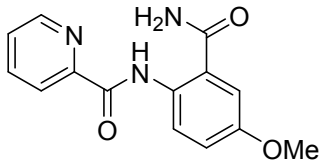
N-(2-carbamoyl-5-methylphenyl)picolinamide (2f).

White solid (38.8 mg, 76%), m.p. 246-248 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.20 (s, 1H), 8.74 – 8.68 (m, 1H), 8.62 (d, *J* = 0.5 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 2H), 8.06 (m, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.65 (m, 1H), 7.56 (s, 1H), 7.00 (dd, *J* = 8.0, 0.9 Hz, 1H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 170.3, 162.6, 149.9, 148.6, 142.0, 138.8, 138.0, 128.6, 126.9, 123.5, 122.3, 120.7, 118.5, 21.4. HRMS calcd for $C_{14}H_{14}N_3O_2$ $[M+H]^+$ 256.1081, found 256.1079.



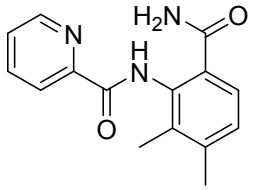
N-(2-carbamoyl-4-methylphenyl)picolinamide (2g).

White solid (42.3 mg, 83%), m.p. 245-247 °C. ^{1}H NMR (400 MHz, DMSO) δ 12.96 (s, 1H), 8.71 (d, *J* = 4.2 Hz, 1H), 8.63 (d, *J* = 8.4 Hz, 1H), 8.22 – 8.13 (m, 2H), 8.05 (m, 1H), 7.68 – 7.60 (m, 3H), 7.37 (dd, *J* = 8.5, 1.4 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 170.3, 162.4, 149.9, 148.6, 138.0, 136.1, 132.3, 132.0, 128.9, 126.9, 122.2, 121.5, 120.3, 20.4. HRMS calcd for $C_{14}H_{14}N_3O_2$ $[M+H]^+$ 256.1081, found 256.1084.



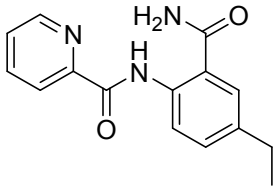
N-(2-carbamoyl-4-methoxyphenyl)picolinamide (2h).

White solid (41.7 mg, 77%), m.p. 246-248 °C. ^{1}H NMR (400 MHz, DMSO) δ 12.80 (s, 1H), 8.71 (d, *J* = 4.1 Hz, 1H), 8.67 – 8.62 (m, 1H), 8.25 (s, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 8.05 (m, 1H), 7.70 – 7.61 (m, 2H), 7.35 (d, *J* = 2.9 Hz, 1H), 7.16 (dd, *J* = 9.1, 2.9 Hz, 1H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 169.9, 162.1, 154.5, 150.0, 148.6, 138.0, 131.7, 126.8, 122.9, 122.2, 121.9, 117.3, 113.6, 55.5. HRMS calcd for $C_{14}H_{14}N_3O_3$ $[M+H]^+$ 272.1030, found 272.1032.



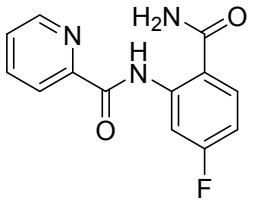
N-(6-carbamoyl-2,3-dimethylphenyl)picolinamide (2i).

White solid (29.6 mg, 55%), m.p. 206-208 °C. ^{1}H NMR (400 MHz, DMSO) δ 11.11 (s, 1H), 8.76 – 8.71 (m, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 8.04 (m, 1H), 7.85 (s, 1H), 7.66 (m, 1H), 7.40 – 7.32 (m, 2H), 7.16 (d, *J* = 7.8 Hz, 1H), 2.32 (s, 3H), 2.08 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 169.8, 162.1, 149.5, 148.7, 139.8, 138.0, 134.0, 133.9, 129.3, 126.9, 125.1, 122.2, 20.3, 15.4. HRMS calcd for $C_{15}H_{16}N_3O_2$ $[M+H]^+$ 270.1237, found 270.1238.



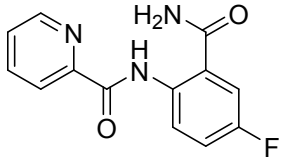
N-(2-carbamoyl-4-ethylphenyl)picolinamide (2j).

White solid (44.7 mg, 83%), m.p. 243-245 °C. ^{1}H NMR (400 MHz, DMSO) δ 12.98 (s, 1H), 8.76 – 8.67 (m, 1H), 8.65 (d, *J* = 8.5 Hz, 1H), 8.27 – 8.10 (m, 2H), 8.05 (m, 1H), 7.69 – 7.54 (m, 3H), 7.39 (m, 1H), 2.62 (m, 2H), 1.22 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 170.4, 162.4, 149.9, 148.6, 138.3, 138.0, 136.3, 131.1, 127.8, 126.9, 122.2, 121.6, 120.4, 27.6, 15.4. HRMS calcd for $C_{15}H_{16}N_3O_2$ $[M+H]^+$ 270.1237, found 270.1240.



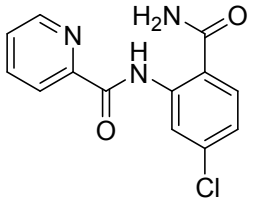
N-(2-carbamoyl-5-fluorophenyl)picolinamide (2k).

White solid (44 mg, 85%), m.p. 210-212 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.40 (s, 1H), 8.72 (m, 1H), 8.61 (dd, *J* = 12.3, 2.7 Hz, 1H), 8.26 (s, 1H), 8.20 – 8.14 (m, 1H), 8.06 (m, 1H), 7.91 (dd, *J* = 8.8, 6.5 Hz, 1H), 7.75 – 7.60 (m, 2H), 7.09 – 6.99 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 169.5, 163.6 (d, *J* = 245 Hz), 163.1, 149.4, 148.7, 140.9 (d, *J* = 12 Hz), 138.1, 131.0 (d, *J* = 10 Hz), 127.2, 122.5, 117.5 (d, *J* = 2 Hz), 109.6 (d, *J* = 22 Hz), 107.9 (d, *J* = 28 Hz). HRMS calcd for $C_{13}H_{11}FN_3O_2$ $[M+H]^+$ 260.0830, found 260.0832.



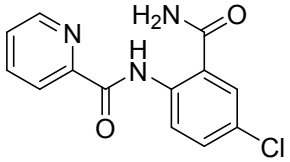
N-(2-carbamoyl-4-fluorophenyl)picolinamide (2l).

White solid (47.7 mg, 92%), m.p. 276-278 °C. ^{1}H NMR (400 MHz, DMSO) δ 12.96 (s, 1H), 8.76 (dd, *J* = 9.2, 5.4 Hz, 1H), 8.72 (m, 1H), 8.30 (s, 1H), 8.20 – 8.14 (m, 1H), 8.06 (m, 1H), 7.80 (s, 1H), 7.69 – 7.63 (m, 2H), 7.48 – 7.41 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 169.0 (d, *J* = 1 Hz), 162.5, 157.0 (d, *J* = 240 Hz), 149.6, 148.7, 138.1, 135.0 (d, *J* = 2 Hz), 127.0, 123.1 (d, *J* = 6 Hz), 122.3, 122.2, 118.6 (d, *J* = 21 Hz), 115.2 (d, *J* = 24 Hz). HRMS calcd for $C_{13}H_{11}FN_3O_2$ $[M+H]^+$ 260.0830, found 260.0834.



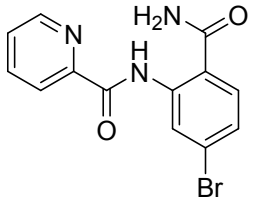
N-(2-carbamoyl-5-chlorophenyl)picolinamide (2m).

White solid (41.8 mg,76%), m.p. 270-272 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.27 (s, 1H), 8.86 (d, *J* = 2.2 Hz, 1H), 8.73 (dd, *J* = 4.7, 0.6 Hz, 1H), 8.31 (s, 1H), 8.18 (d, *J* = 7.8 Hz, 1H), 8.07 (m, 1H), 7.84 (d, *J* = 8.5 Hz, 1H), 7.80 – 7.71 (m, 1H), 7.67 (m, 1H), 7.28 (dd, *J* = 8.5, 2.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 169.4, 162.9, 149.3, 148.7, 139.9, 138.1, 136.4, 130.3, 127.2, 122.6, 122.5, 119.8, 119.6. HRMS calcd for $C_{13}H_{11}ClN_3O_2$ $[M+H]^+$ 276.0535, found 276.0533.



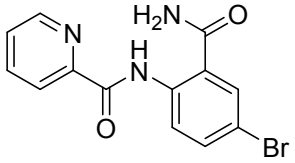
N-(2-carbamoyl-4-chlorophenyl)picolinamide (2n).

White solid (44.0 mg, 80%), m.p. 273-275 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.04 (s, 1H), 8.77 (d, *J* = 9.0 Hz, 1H), 8.74 – 8.69 (m, 1H), 8.35 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.05 (m, 1H), 7.87 (d, *J* = 2.5 Hz, 1H), 7.79 (d, *J* = 8.7 Hz, 1H), 7.68 – 7.60 (m, 2H). ^{13}C NMR (100 MHz, DMSO) δ 168.9, 162.7, 149.5, 148.7, 138.1, 137.4, 131.6, 128.2, 127.1, 126.7, 123.1, 122.4, 122.0. HRMS calcd for $C_{13}H_{11}ClN_3O_2$ $[M+H]^+$ 276.0535, found 276.0532.



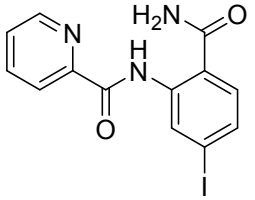
N-(5-bromo-2-carbamoylphenyl)picolinamide (2o).

White solid (35.7 mg, 56%), m.p. 274-276 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.23 (s, 1H), 9.01 (d, *J* = 2.0 Hz, 1H), 8.73 (m, 1H), 8.32 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.07 (m, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.68 (m, 1H), 7.41 (dd, *J* = 8.4, 2.0 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 169.5, 162.9, 149.3, 148.7, 139.9, 138.2, 130.4, 127.2, 125.6, 125.2, 122.6, 122.5, 120.2. HRMS calcd for $C_{13}H_{11}BrN_3O_2$ $[M+H]^+$ 320.0029, found 320.0027.



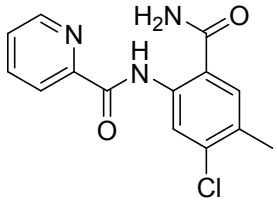
N-(4-bromo-2-carbamoylphenyl)picolinamide (2p).

White solid (41.5 mg, 65%), m.p. 269-271 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.04 (s, 1H), 8.76 – 8.67 (m, 2H), 8.36 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.06 (m, 1H), 7.99 (d, *J* = 2.3 Hz, 1H), 7.80 (s, 1H), 7.75 (dd, *J* = 8.9, 2.2 Hz, 1H), 7.66 (dd, *J* = 6.7, 4.9 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.8, 162.7, 149.5, 148.7, 138.1, 137.8, 134.5, 131.0, 127.1, 123.4, 122.4, 122.2, 114.6. HRMS calcd for $C_{13}H_{11}BrN_3O_2$ $[M+H]^+$ 320.0029, found 320.0025.



N-(2-carbamoyl-5-iodophenyl)picolinamide (2q).

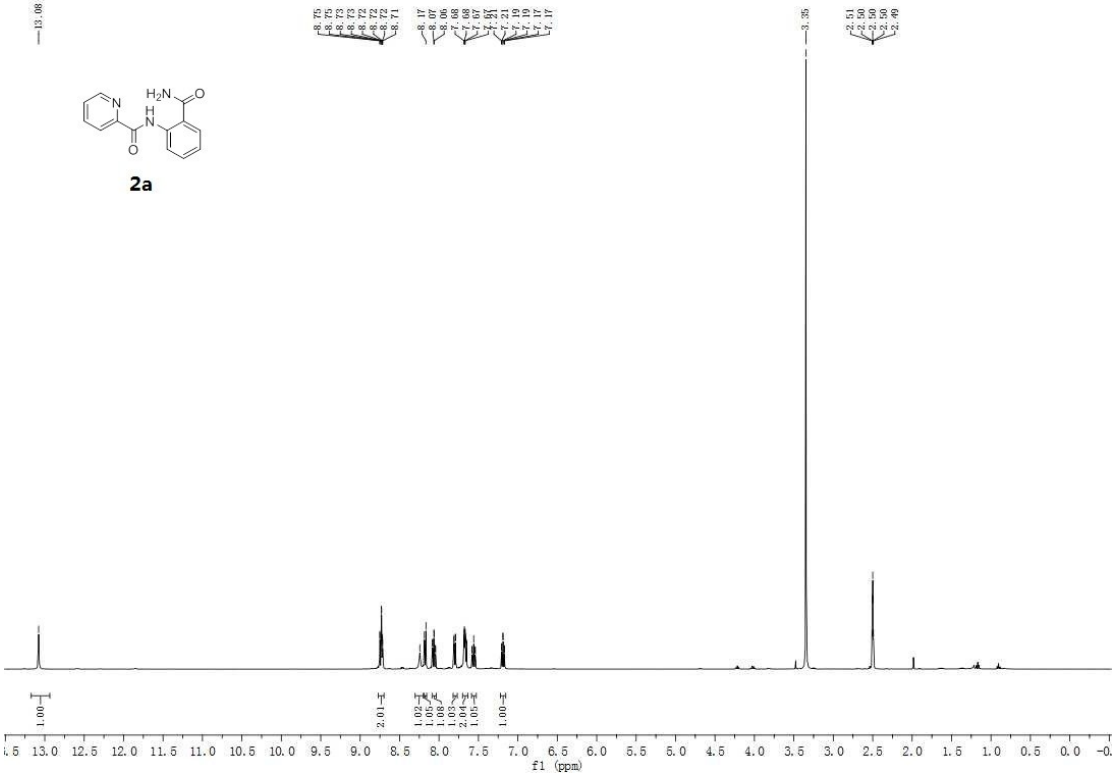
White solid (36.7 mg, 50%), m.p. 259-261 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.15 (s, 1H), 9.19 (s, 1H), 8.72 (dd, *J* = 4.7, 0.6 Hz, 1H), 8.29 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.07 (m, 1H), 7.78 – 7.70 (m, 1H), 7.69 – 7.65 (m, 1H), 7.58 (d, *J* = 0.8 Hz, 2H). ^{13}C NMR (100 MHz, DMSO) δ 169.7, 162.8, 149.4, 148.7, 139.5, 138.2, 131.5, 130.2, 128.5, 127.2, 122.4, 120.6, 99.2. HRMS calcd for $C_{13}H_{11}IN_3O_2$ $[M+H]^+$ 367.9891, found 367.9894.

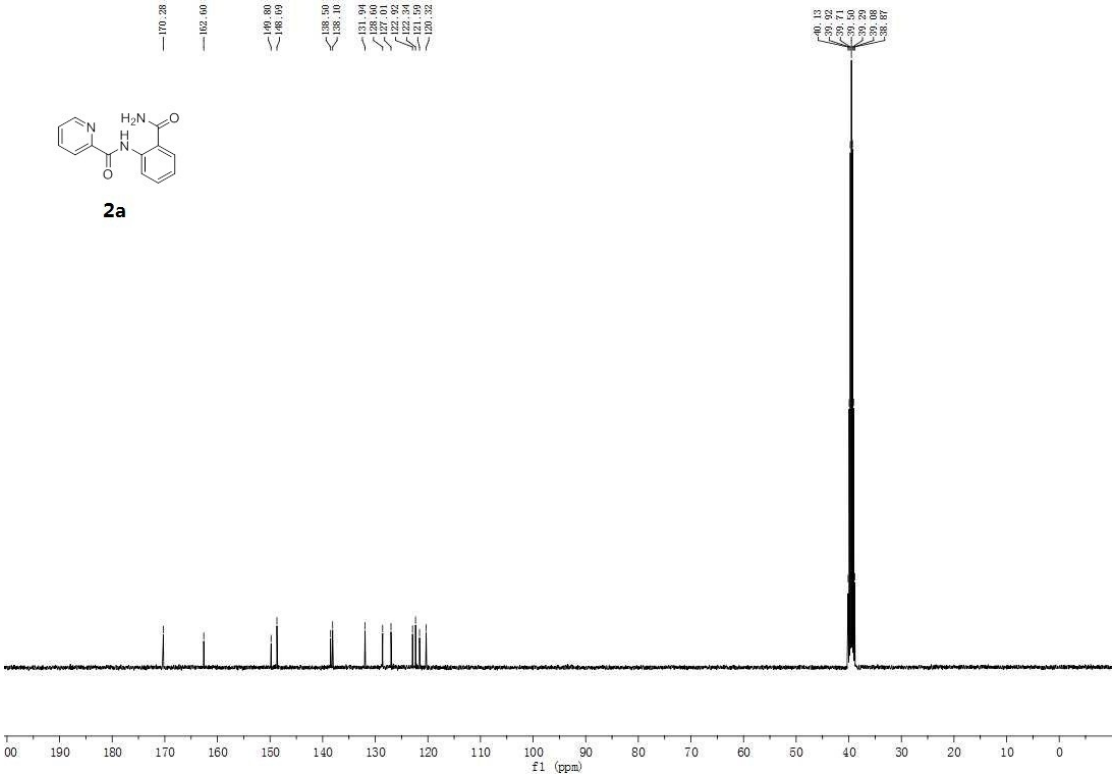


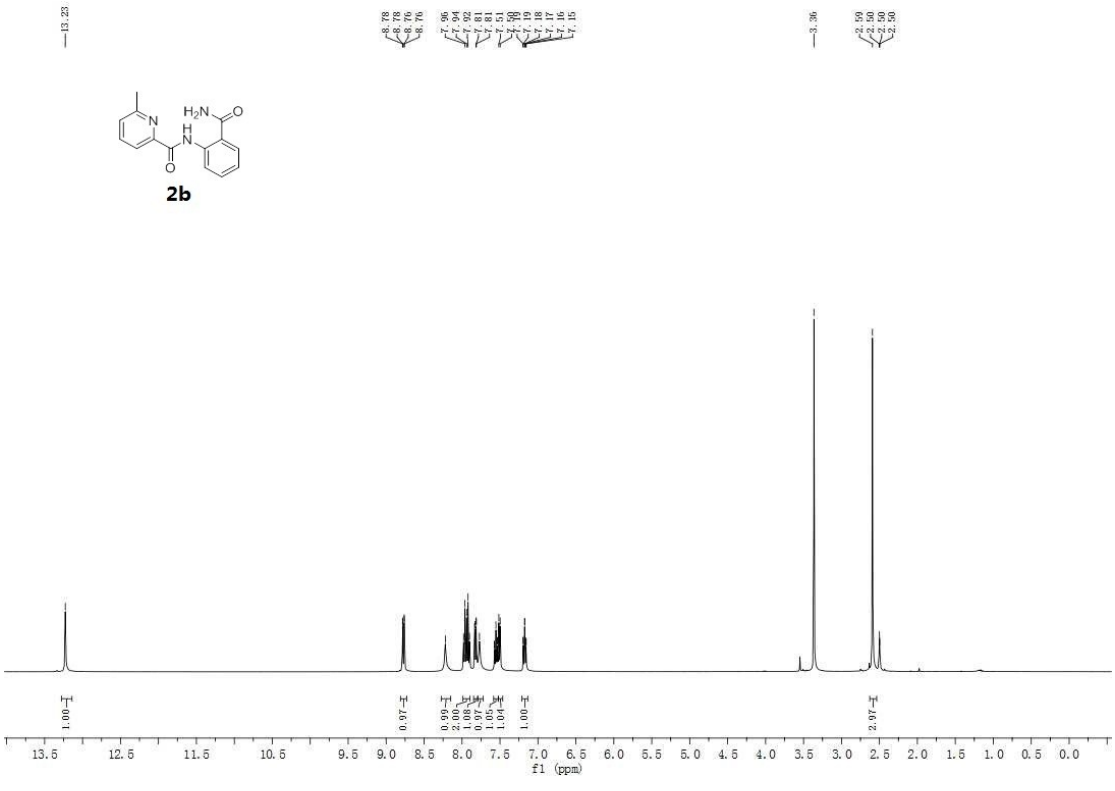
N-(2-carbamoyl-5-chloro-4-methylphenyl)picolinamide (2r).

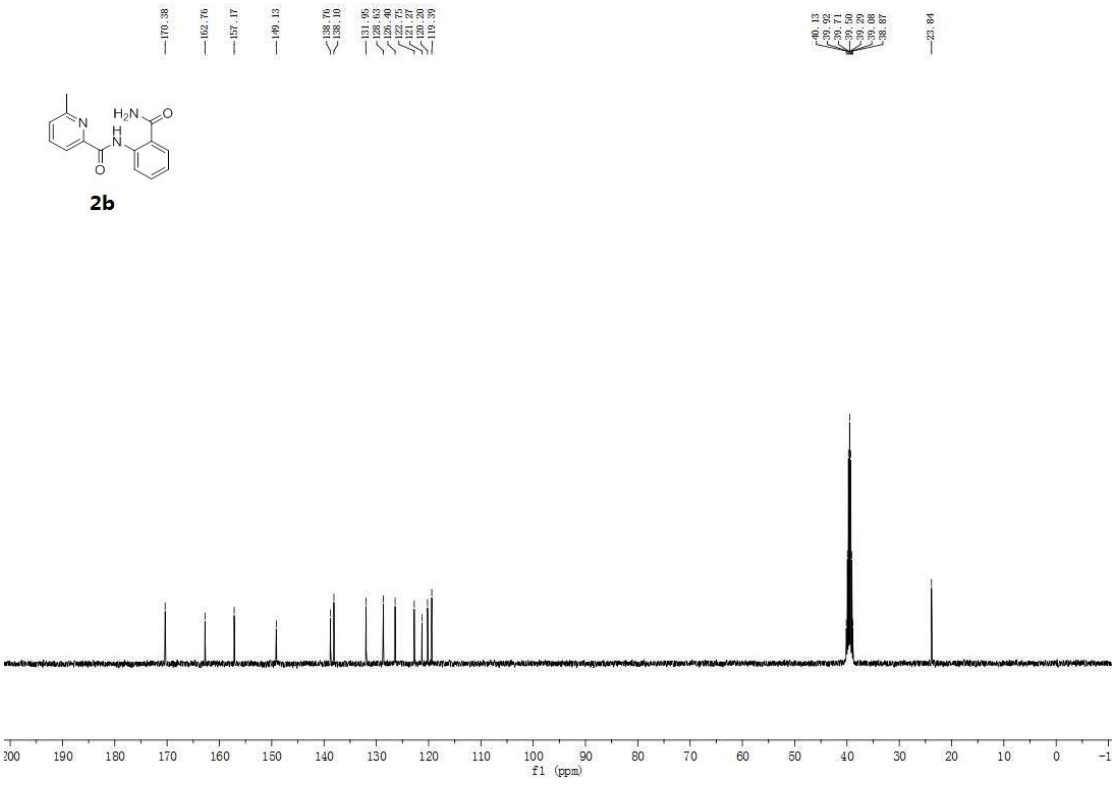
White solid (40.5 mg, 70%), m.p. 271-273 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.13 (s, 1H), 8.85 (s, 1H), 8.72 (m, 1H), 8.25 (s, 1H), 8.18 – 8.14 (m, 1H), 8.06 (m, 1H), 7.82 (s, 1H), 7.70 (d, *J* = 13.0 Hz, 1H), 7.66 (m, 1H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 169.4, 162.7, 149.5, 148.7, 138.1, 137.6, 136.3, 131.0, 129.7, 127.1, 122.4, 120.0, 119.9, 19.0. HRMS calcd for $C_{14}H_{13}ClN_3O_2$ $[M+H]^+$ 290.0691, found 290.0692.

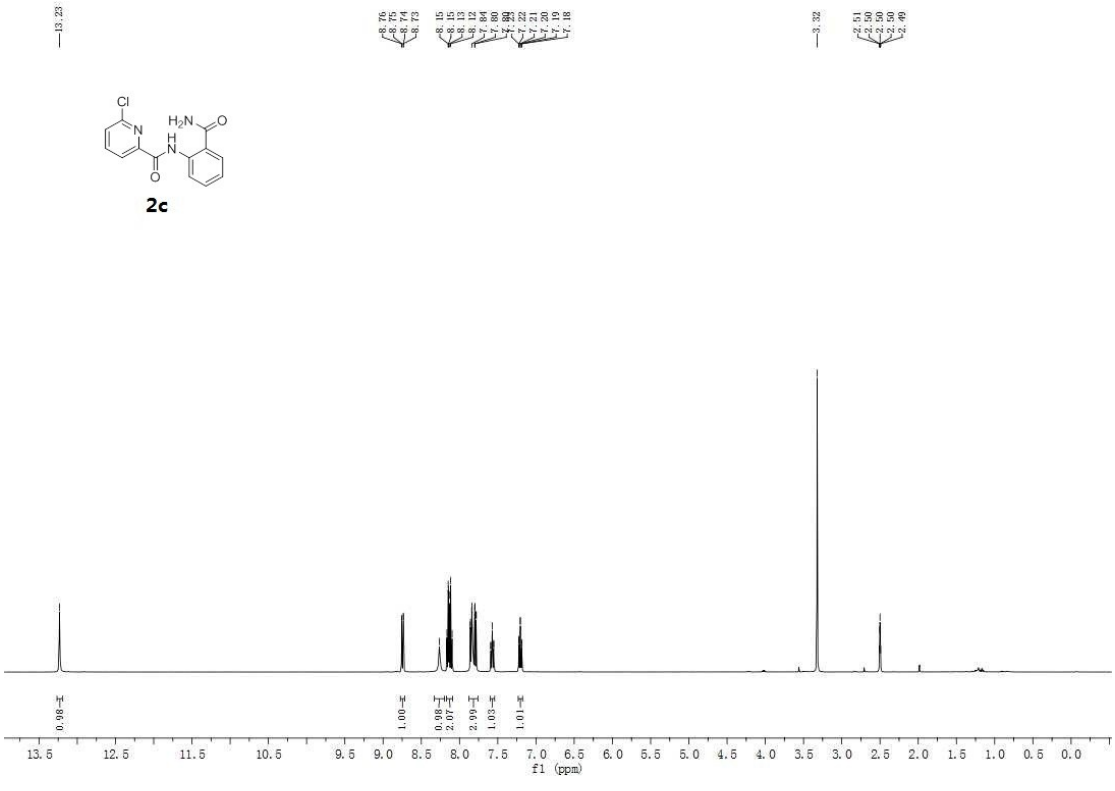
NMR spectra of all compounds

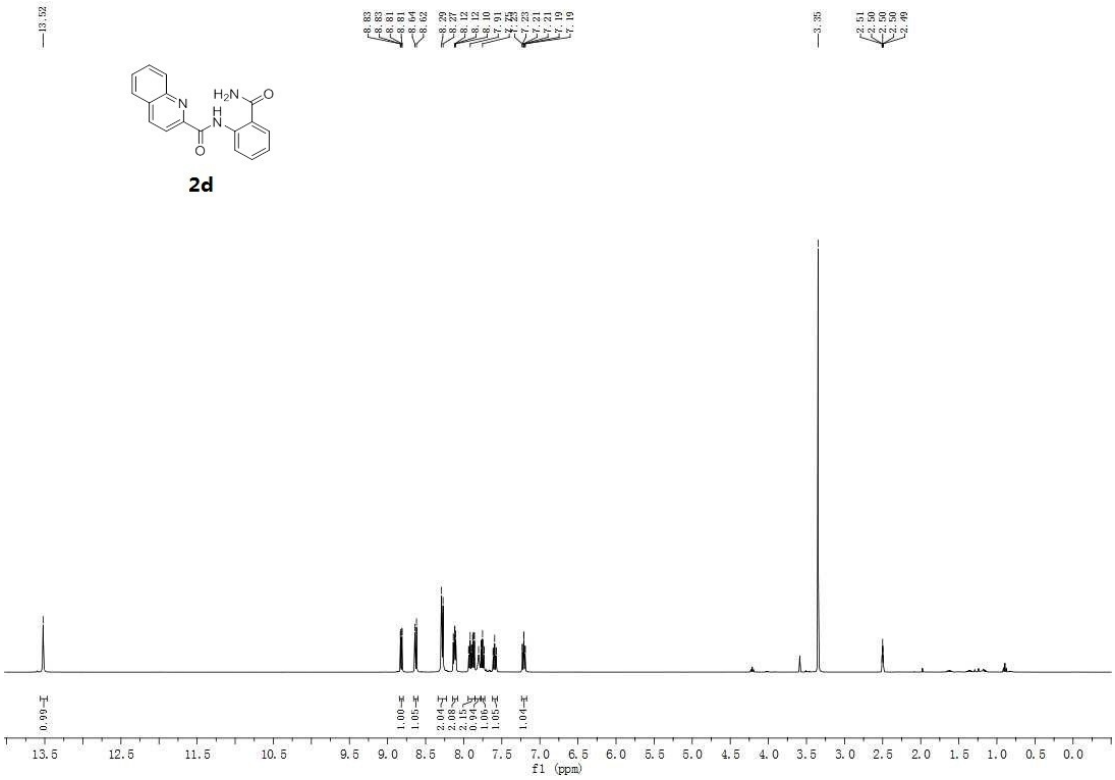


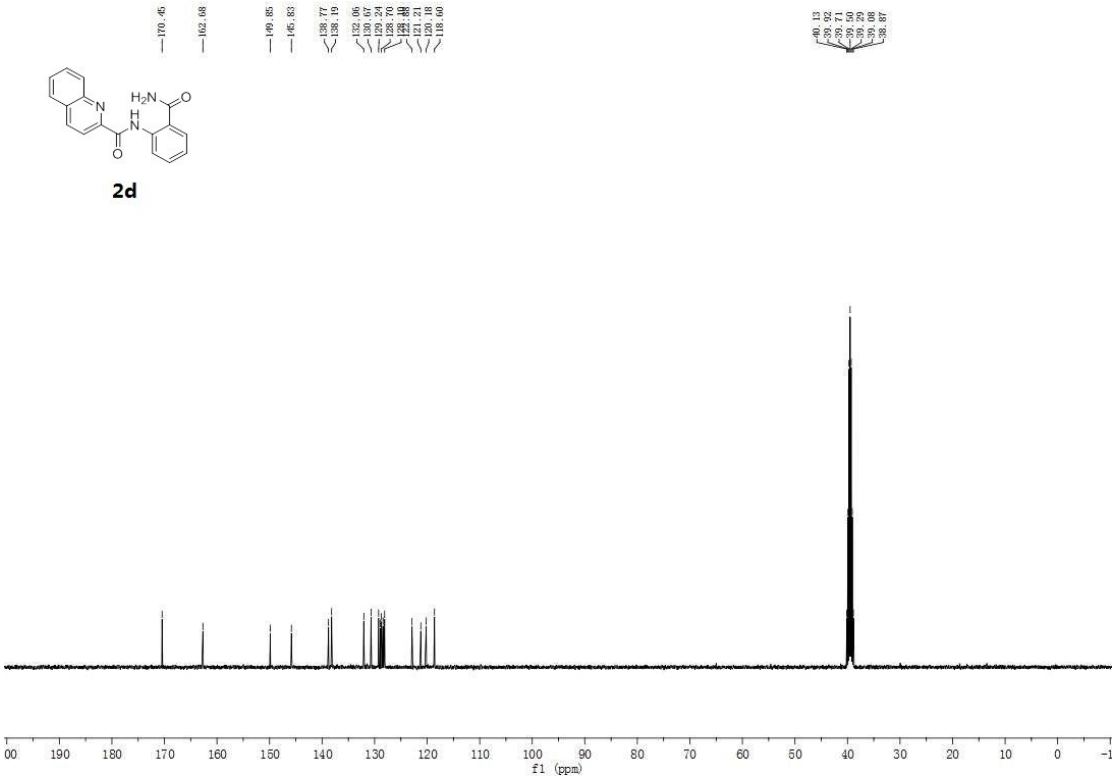


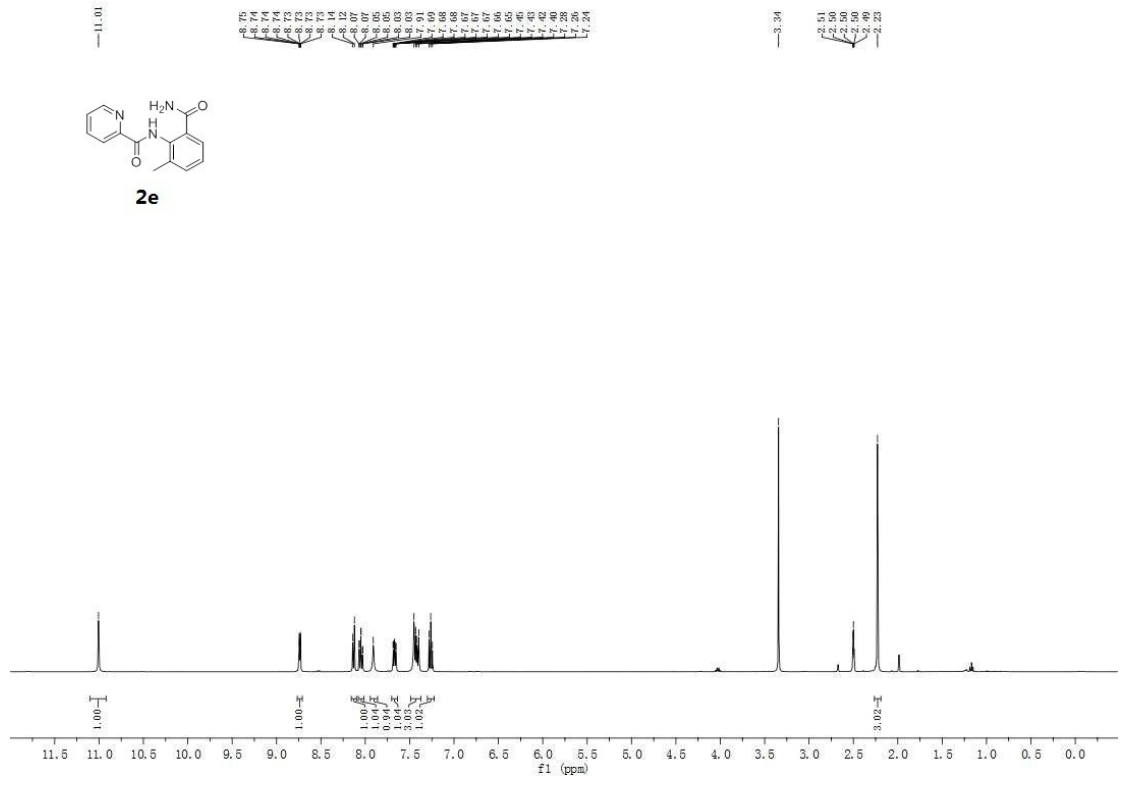

13.23
8.78
8.78
8.76
8.76
7.96
7.94
7.92
7.81
7.81
7.51
7.19
7.18
7.17
7.16
7.15
3.36
2.59
2.56
2.50
2.50
2b
1.00
0.97
0.99
2.09
1.04
0.97
1.05
1.04
1.00
2.97
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.38
162.76
157.17
149.13
138.76
138.10
131.95
128.63
126.40
122.75
121.27
120.30
119.39
40.13
39.92
39.71
39.50
39.29
39.08
38.87
23.84
2b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

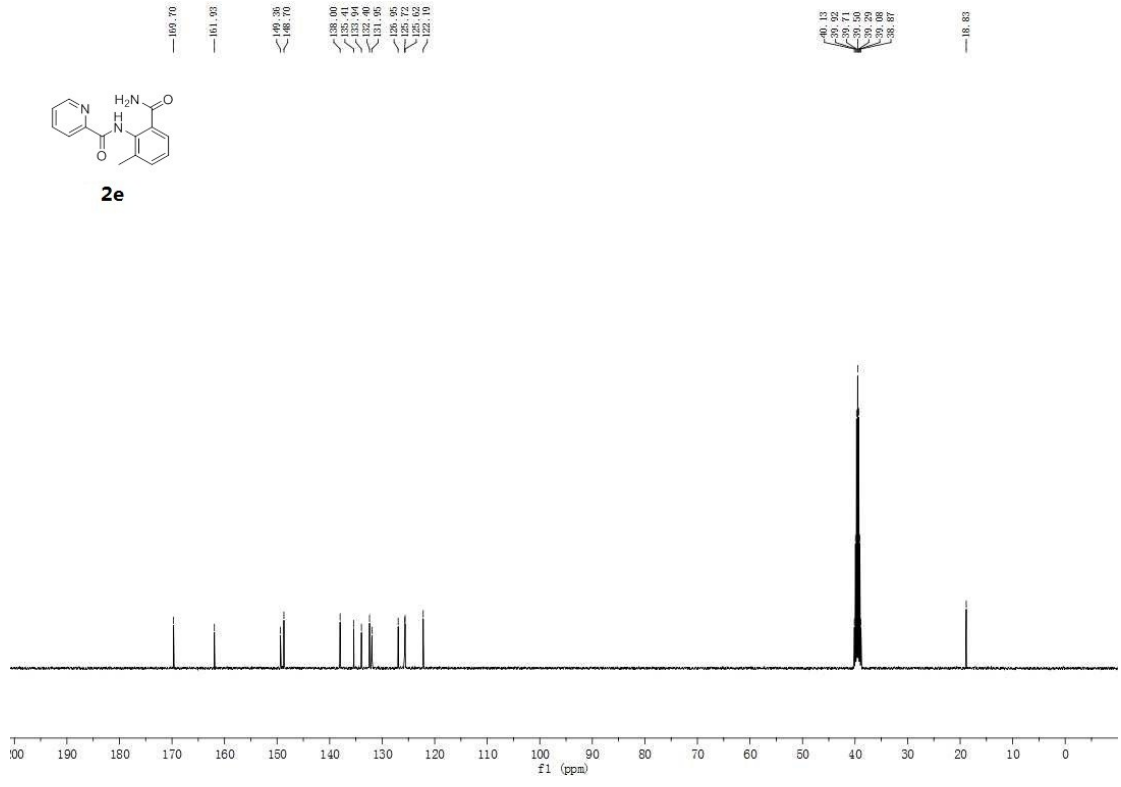

13.23
8.76
8.75
8.74
8.73
8.15
8.15
8.13
8.12
7.84
7.80
7.72
7.71
7.20
7.19
7.18
3.32
2.51
2.50
2.50
2.49
Cl
N
H2N
O
H
N
O
2c
0.98
1.00
0.98
2.07
2.99
1.03
1.01
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
170.38
161.16
150.49
149.19
141.56
138.62
132.09
128.63
127.64
123.09
121.59
120.91
120.24
40.13
39.92
39.71
39.50
39.29
39.08
38.87
Cl
N
H2N
O
H
N
O
2c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


2d
f1 (ppm)

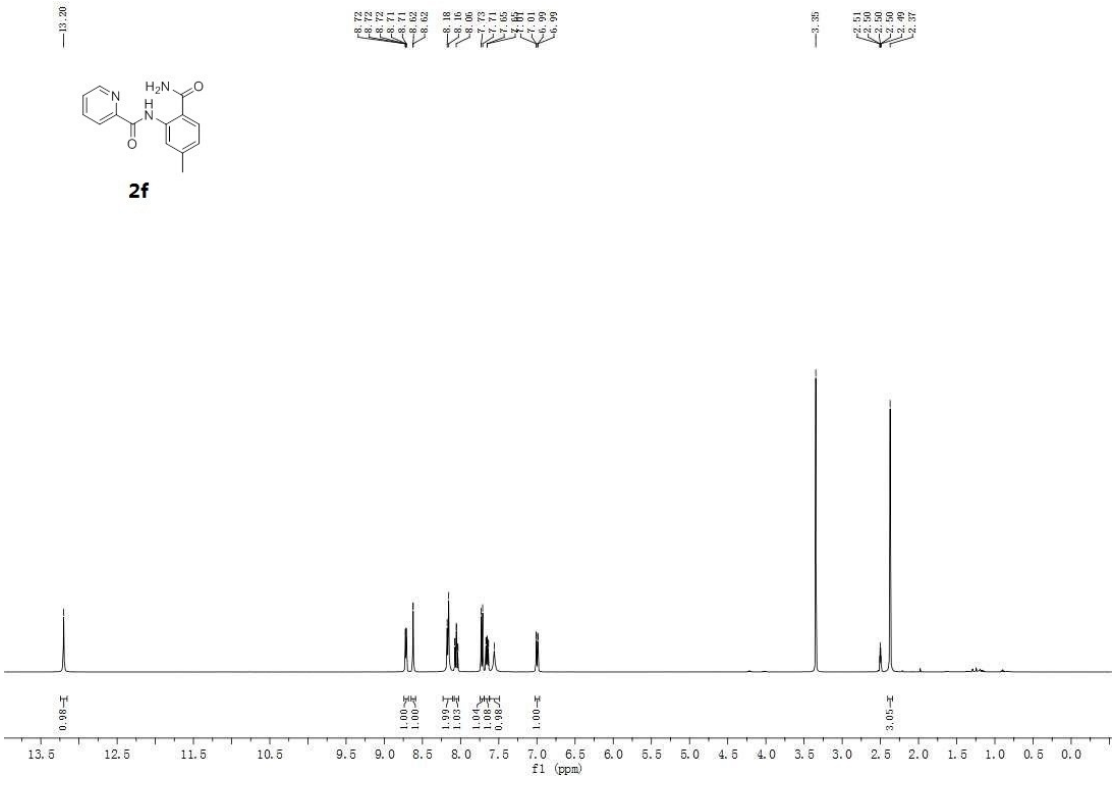

2d
f1 (ppm)



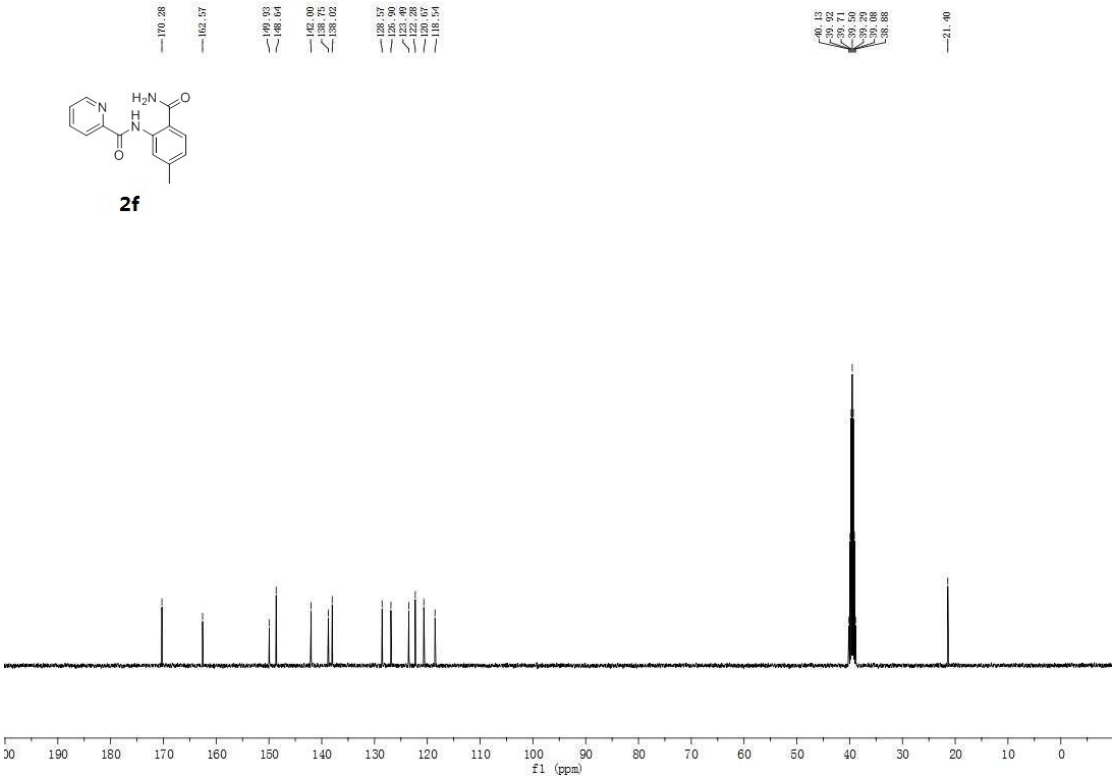
2e



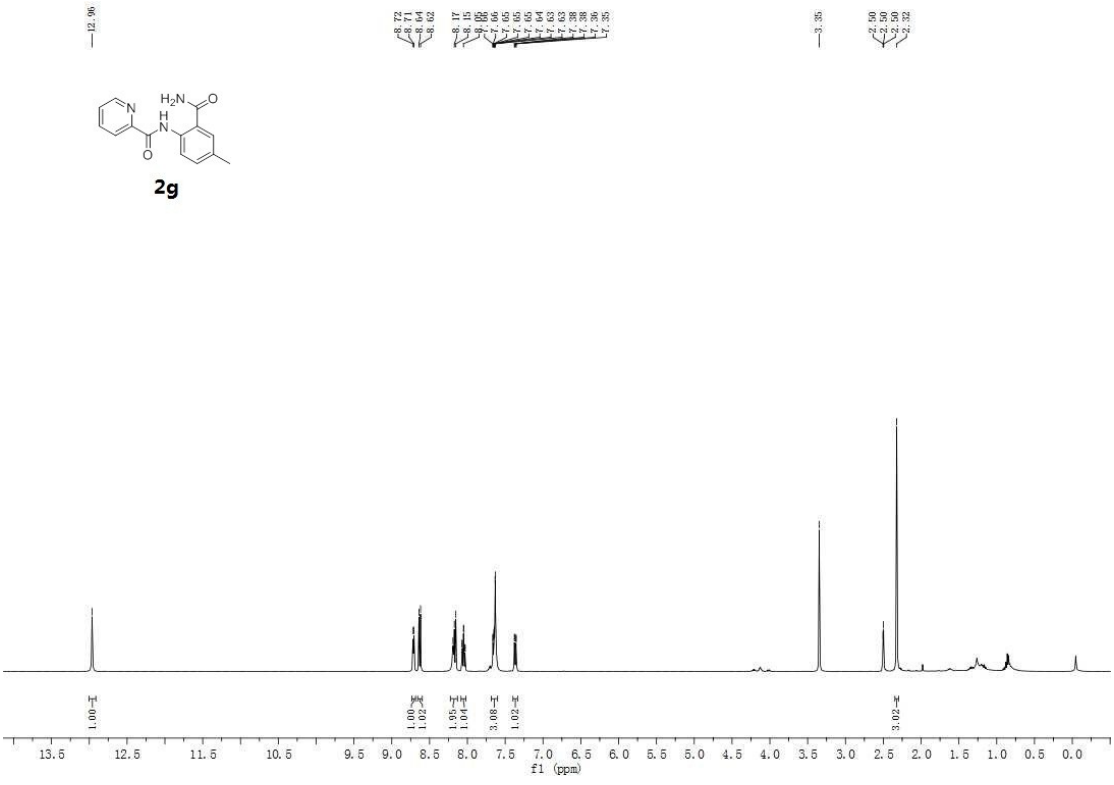
2e


13.20
8.72
8.72
8.72
8.71
8.71
8.62
8.62
8.18
8.16
8.06
7.73
7.71
7.71
7.65
7.65
7.01
6.99
6.99
3.35
2.51
2.50
2.50
2.50
2.49
2.37
0.98
1.00
1.00
1.99
1.03
1.04
1.08
0.98
1.00
3.05
13.5
12.5
11.5
10.5
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)

2f

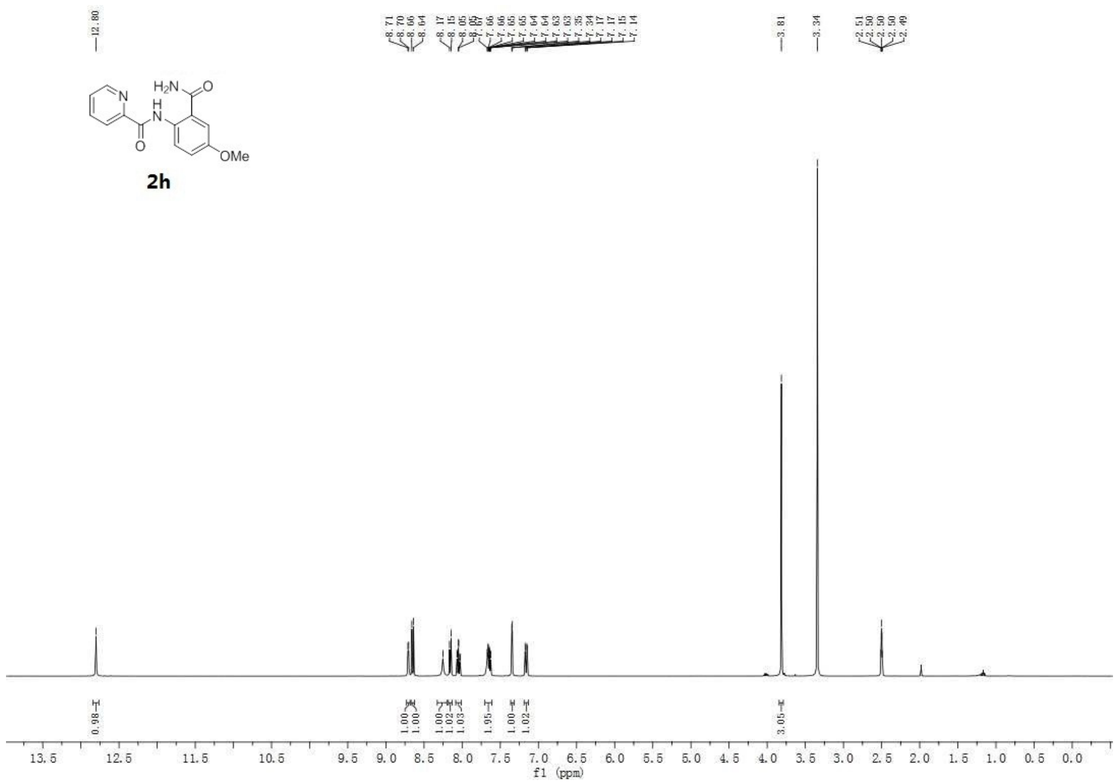

170.28
162.57
149.93
148.64
142.00
138.75
138.02
128.57
126.90
123.49
122.28
120.67
118.54
40.13
39.92
39.71
39.50
39.29
39.08
38.88
21.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

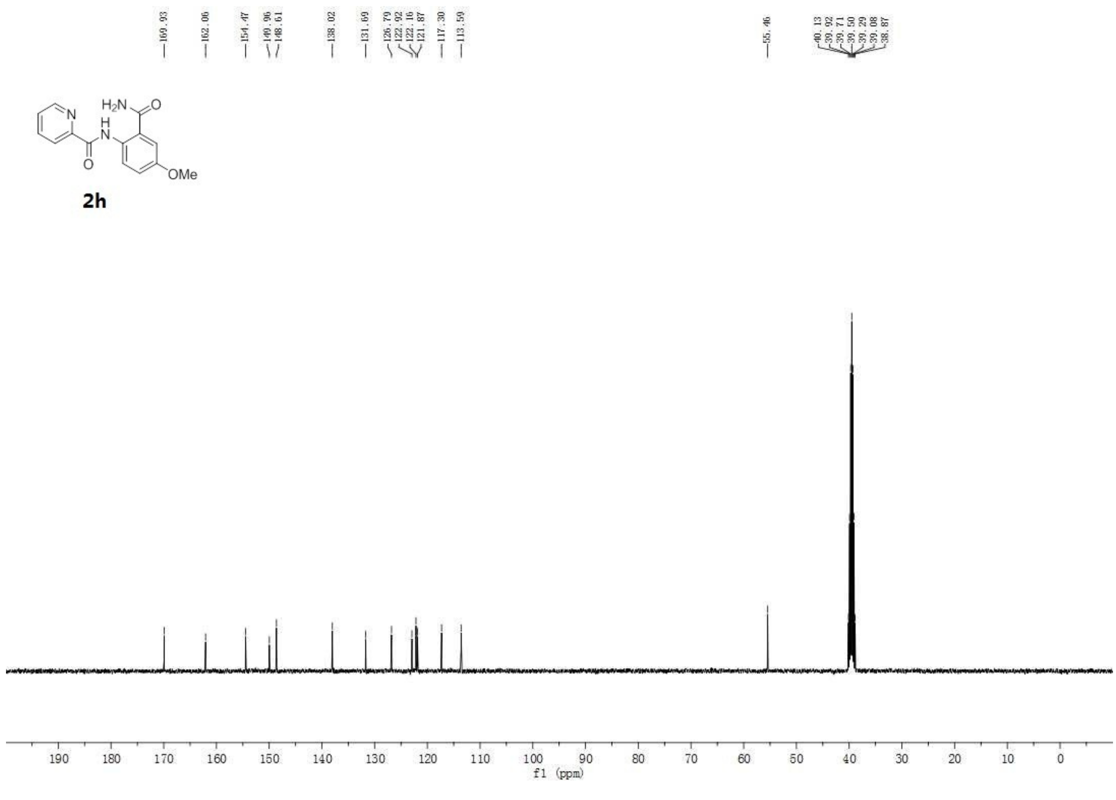
2f

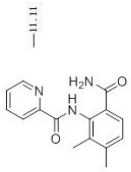

12.96
8.72
8.71
8.64
8.62
8.17
8.15
7.66
7.65
7.65
7.64
7.63
7.63
7.38
7.36
7.35
3.35
2.50
2.50
2.50
2.32
1.00
1.00
1.02
1.95
1.04
3.08
1.03
3.02
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
170.33
162.35
149.91
148.63
138.02
136.06
132.30
131.06
128.92
126.87
122.23
121.52
120.27
40.13
39.92
39.71
39.50
39.29
39.08
38.87
20.37
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2g

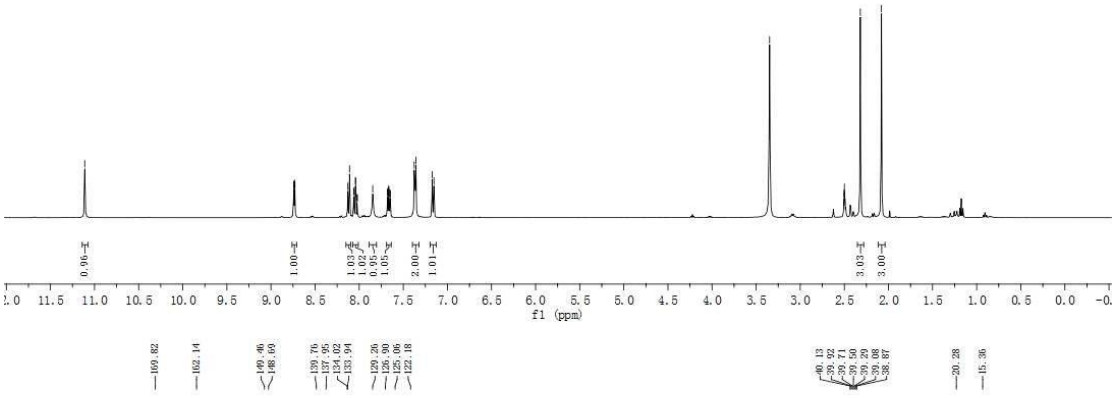
2g

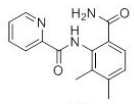

12.80
8.71 8.70 8.66 8.64
8.17 8.15 8.05 8.05 7.66 7.66 7.65 7.65 7.64 7.64 7.63 7.63 7.35 7.34 7.17 7.17 7.15 7.14
3.81
3.34
2.51 2.50 2.50 2.50 2.49
H2N
O
N
H
N
O
OMe
2h
0.98
1.00 1.00
1.00 1.02 1.03
1.95
1.00 1.02
3.05
f1 (ppm)


169.93
162.06
154.47
149.96 148.61
138.02
131.69
126.79 122.92 122.16 121.87
117.30
113.59
55.46
40.13 39.92 39.71 39.50 39.29 39.08 38.87
H2N
O
N
H
N
O
OMe
2h
f1 (ppm)

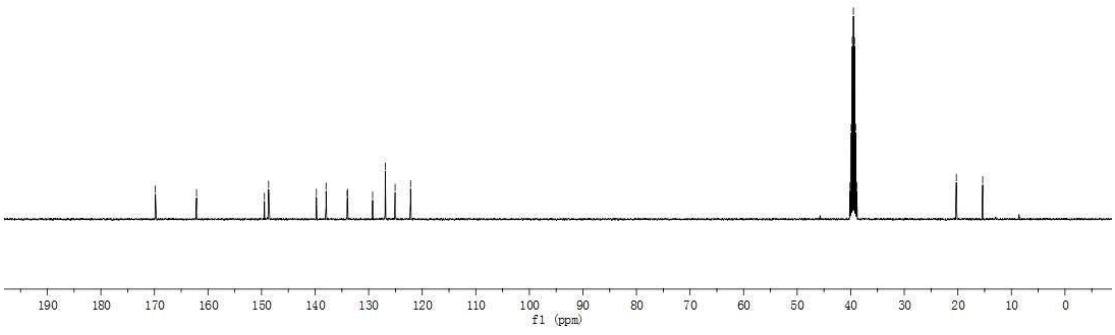


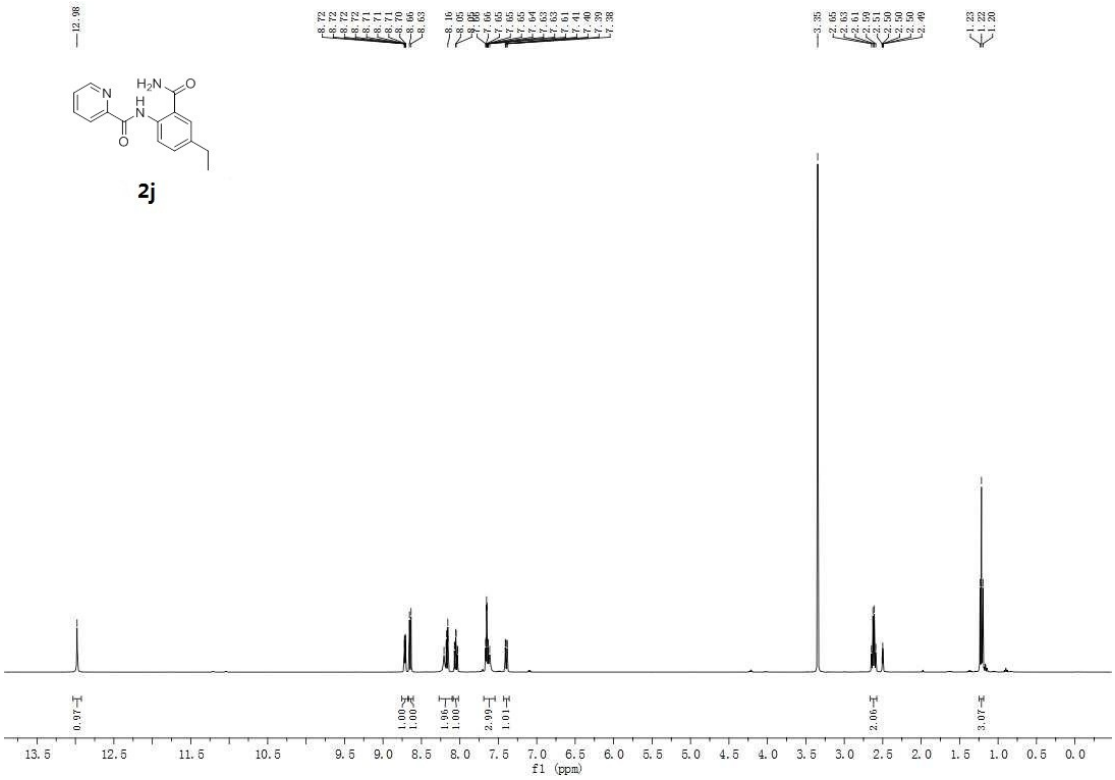
2i

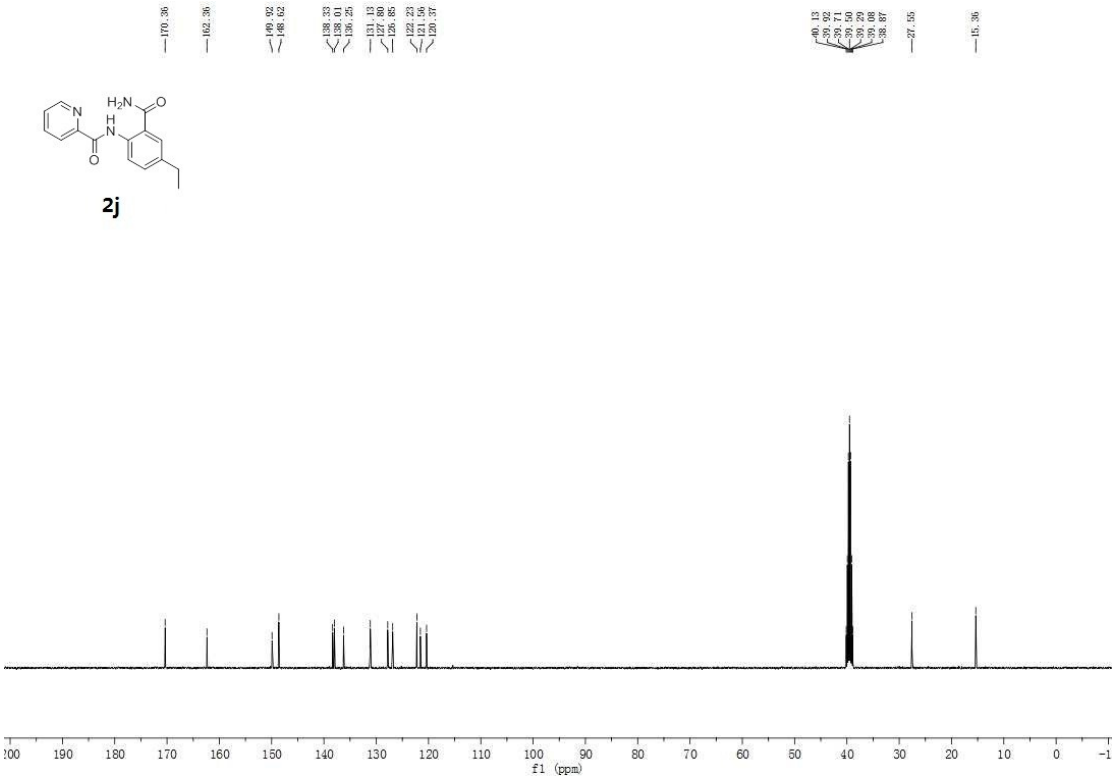


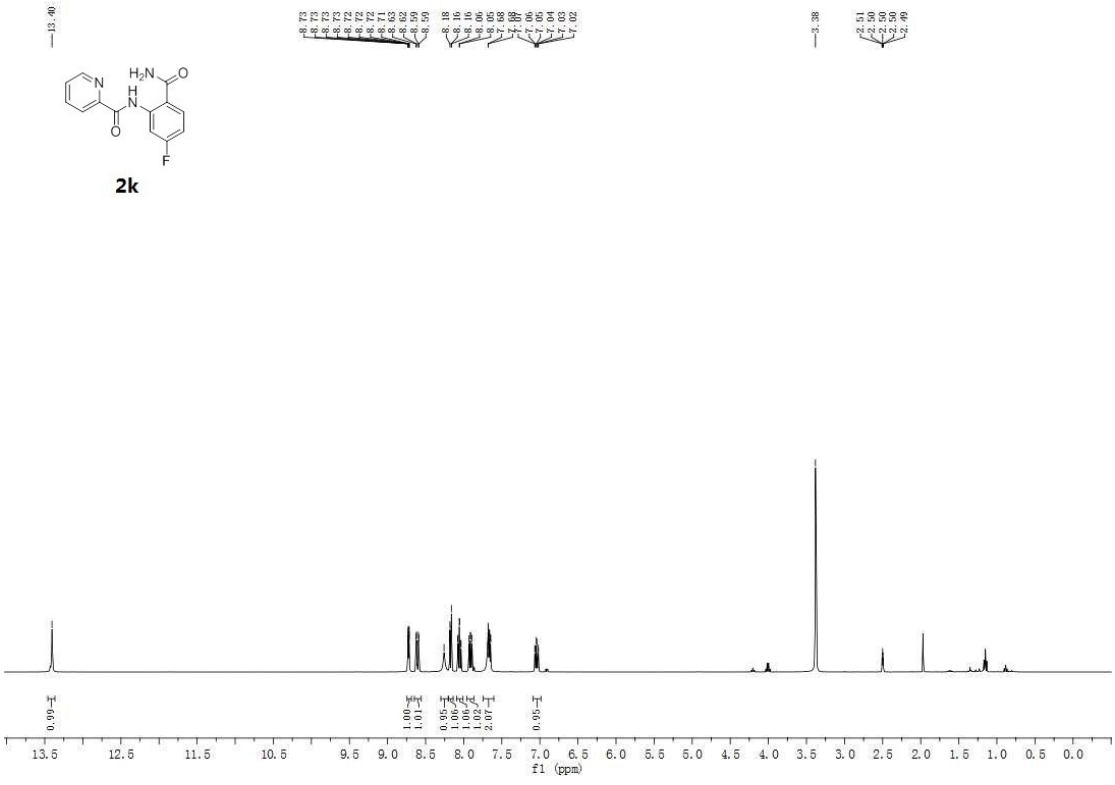


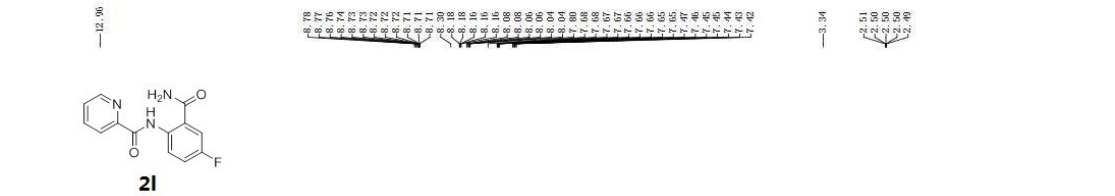
2i



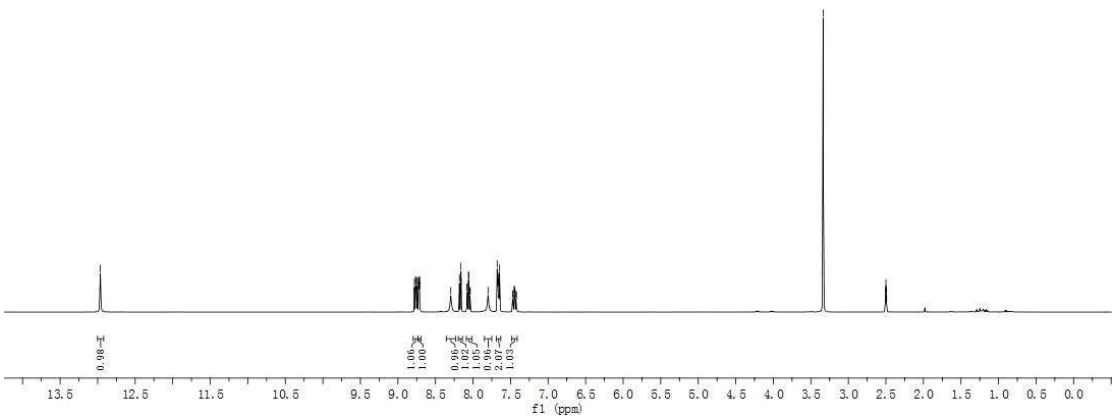

12.98
8.72
8.72
8.72
8.72
8.71
8.71
8.71
8.71
8.70
8.66
8.65
8.16
8.05
8.05
7.66
7.65
7.65
7.64
7.64
7.63
7.63
7.61
7.44
7.40
7.39
7.38
3.35
2.65
2.63
2.61
2.59
2.51
2.50
2.50
2.50
2.49
1.23
1.22
1.20
H2N
O
N
H
N
O
2j
0.97
1.00
1.00
1.96
1.00
2.99
1.01
2.06
3.07
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
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)


170.36
162.36
149.92
148.62
138.33
136.01
136.25
131.13
127.80
125.85
122.23
121.56
120.37
40.13
39.92
39.71
39.50
39.29
39.08
38.87
27.55
15.36
H2N
O
N
H
N
O
2j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

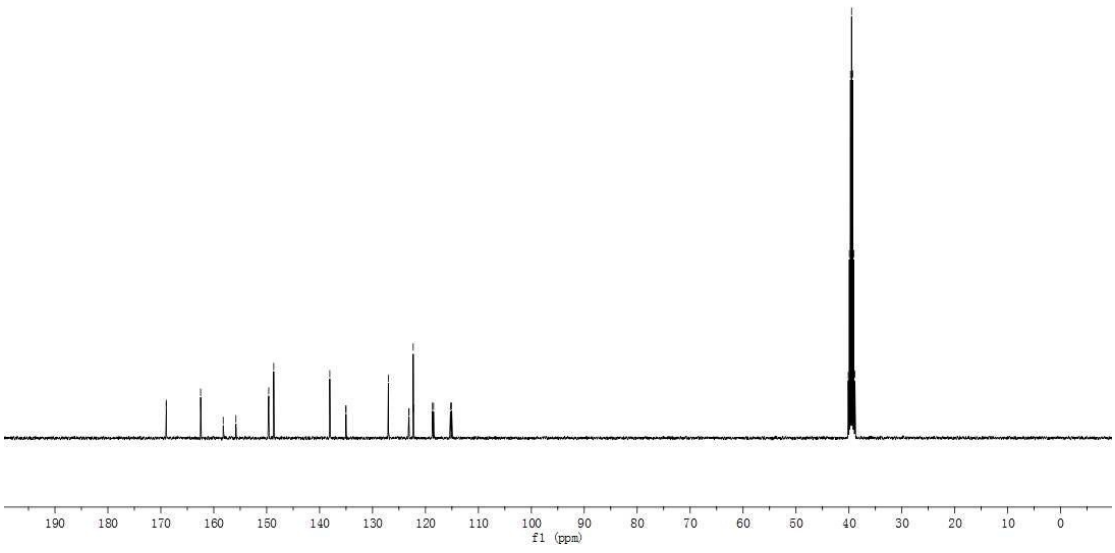

2k
f1 (ppm)
2k
f1 (ppm)

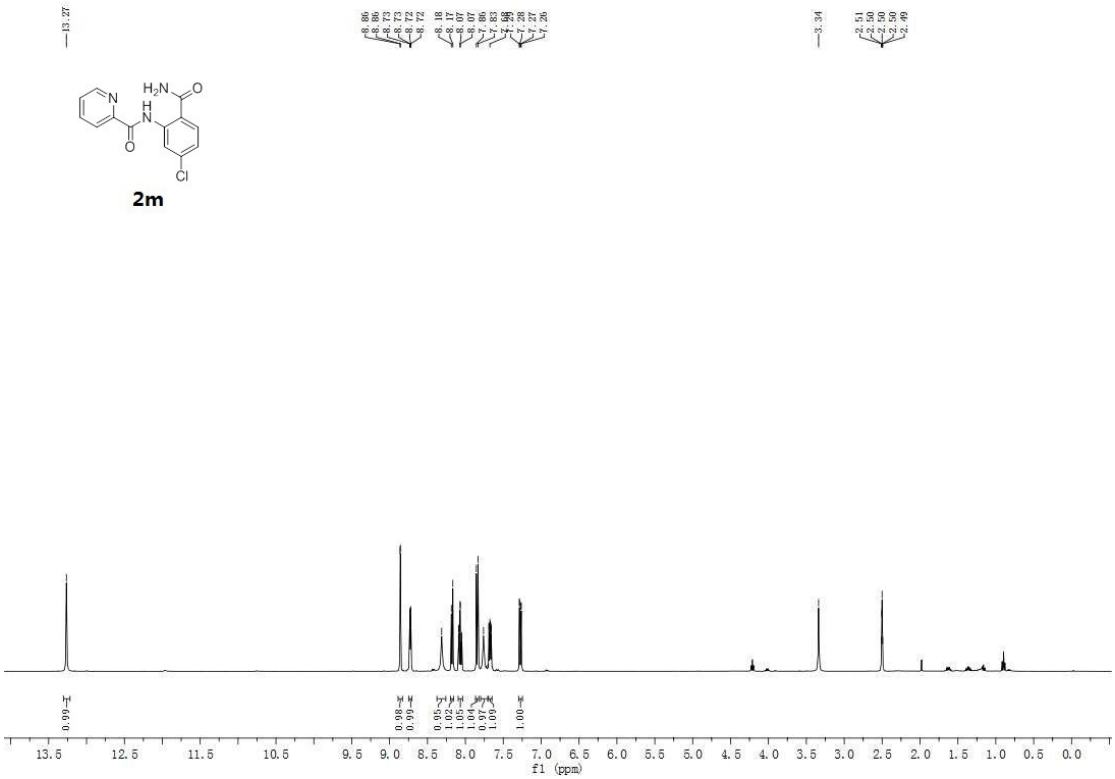


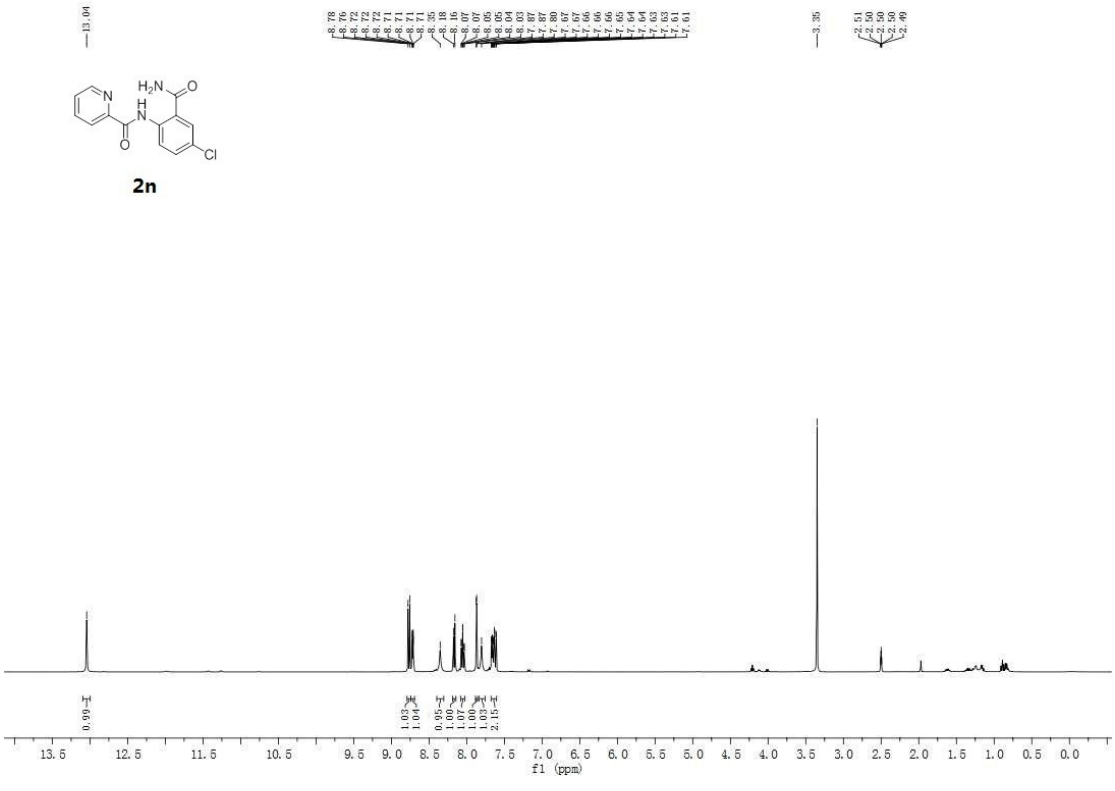
2l

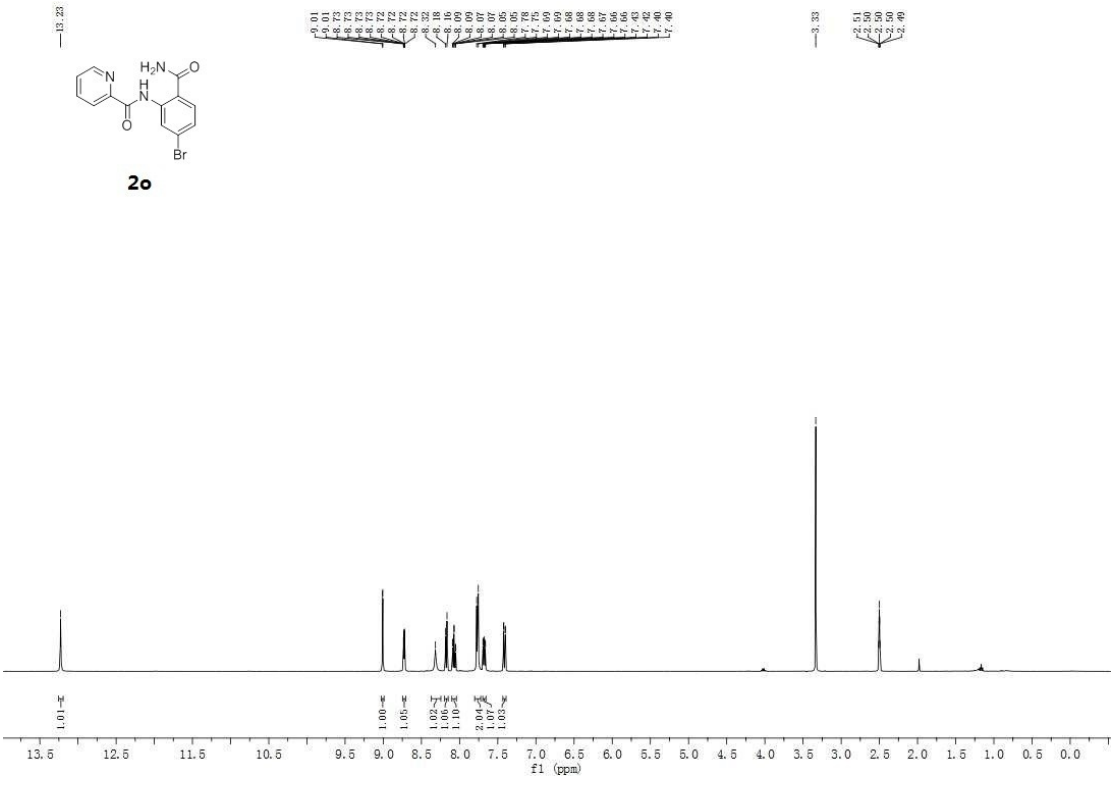


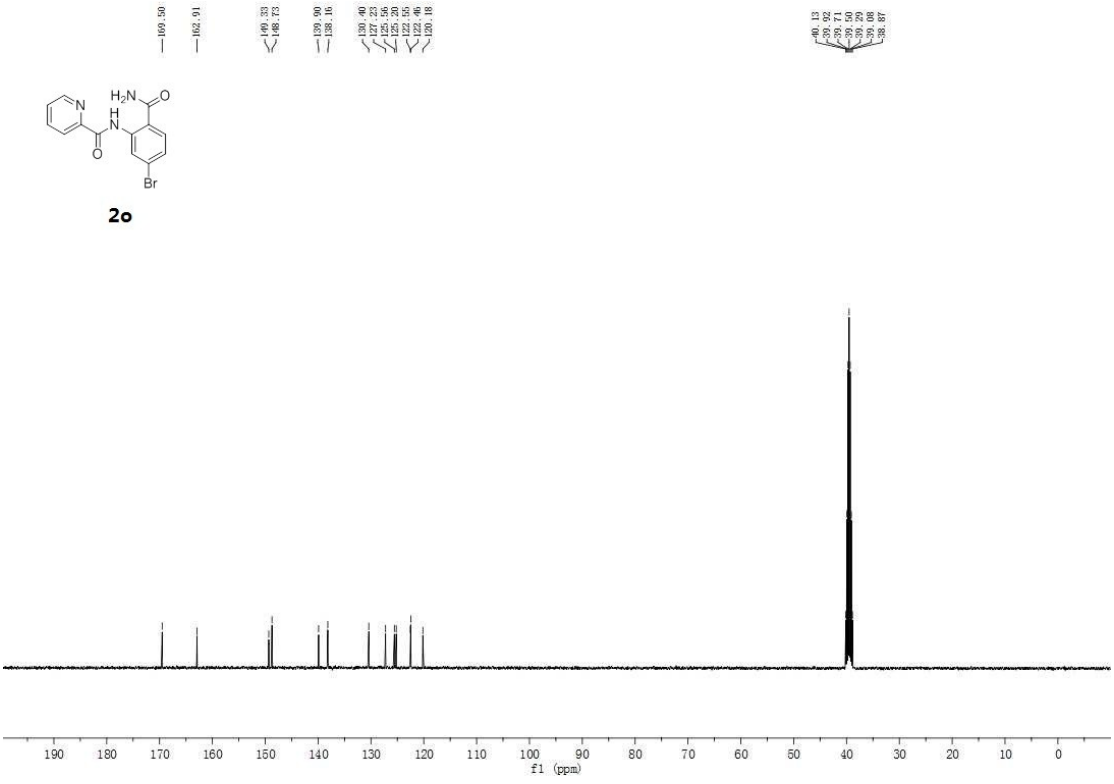
2l

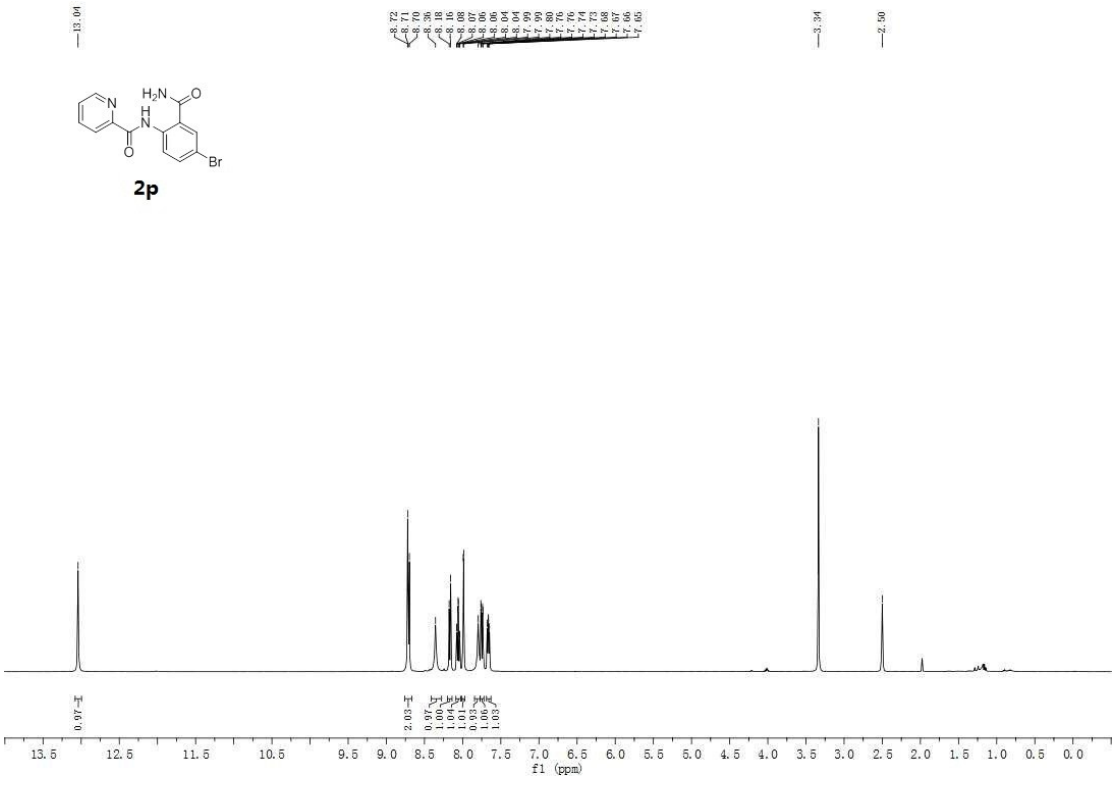



13.27
8.86
8.86
8.73
8.73
8.72
8.72
8.18
8.17
8.07
8.07
7.86
7.83
7.29
7.27
7.26
3.34
2.51
2.50
2.50
2.49
2m
0.99
0.98
0.99
0.95
1.02
1.05
1.04
0.97
1.09
1.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
169.41
162.94
149.34
148.71
139.91
138.13
136.37
130.29
127.21
122.60
122.46
119.80
119.62
40.13
39.92
39.71
39.50
39.29
39.08
38.87
2m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

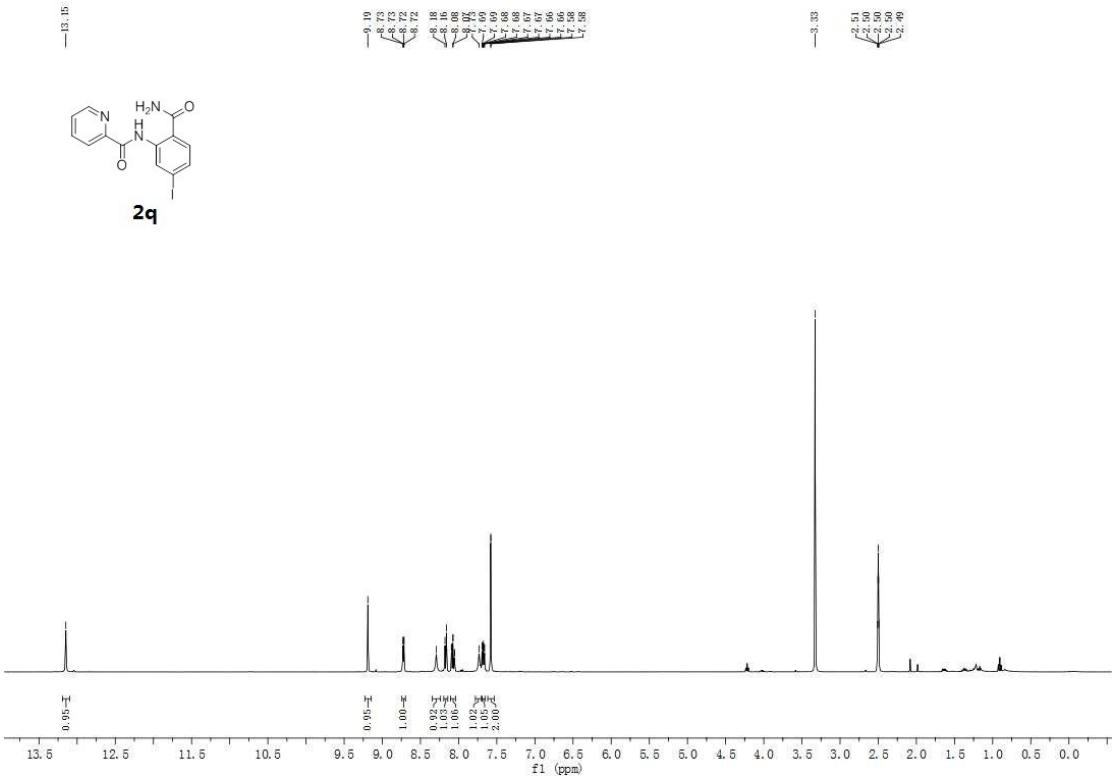

2n
f1 (ppm)
2n
f1 (ppm)

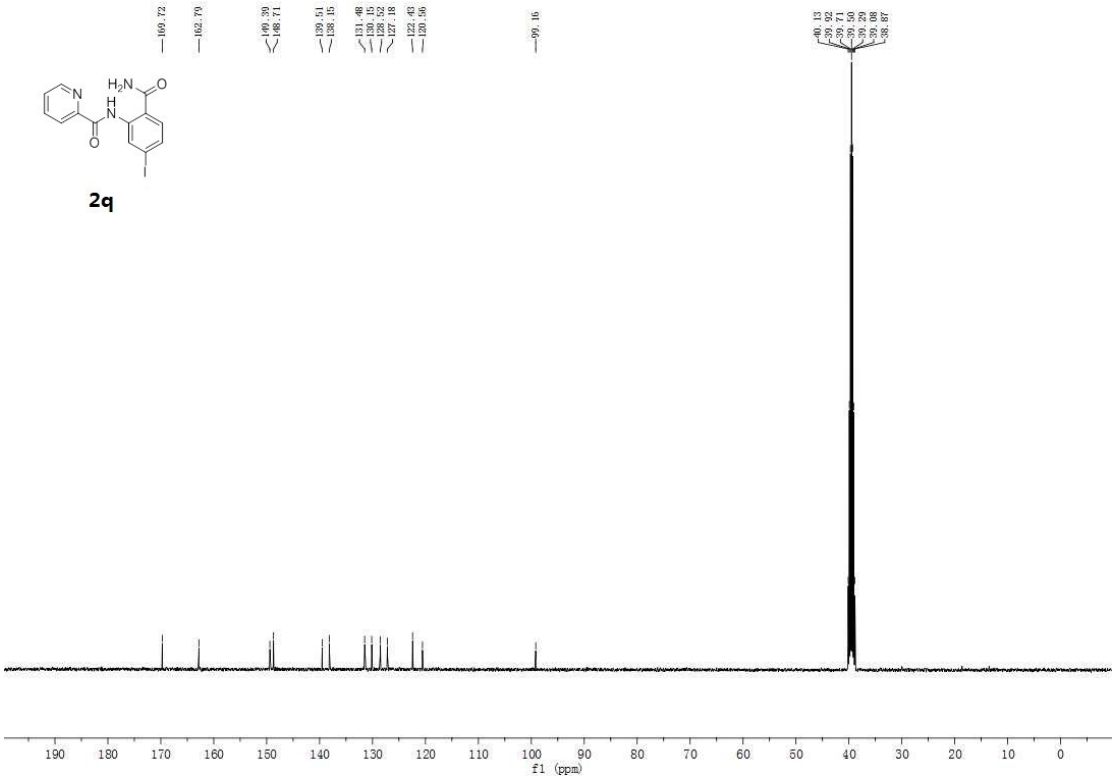

2o
f1 (ppm)

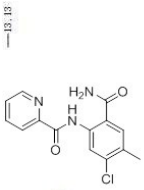

2o
f1 (ppm)


13.04
8.72
8.71
8.70
8.36
8.18
8.15
8.08
8.07
8.06
8.06
8.04
8.04
7.99
7.99
7.80
7.76
7.76
7.74
7.73
7.68
7.67
7.66
7.65
3.34
2.50
0.97
2.03
0.97
1.00
1.04
1.01
0.93
1.06
1.03
13.5
12.5
11.5
10.5
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)
168.83
162.65
149.48
148.68
138.10
137.79
134.54
131.65
127.11
123.37
122.38
122.24
114.64
40.13
39.92
39.71
39.50
39.29
39.08
38.87
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

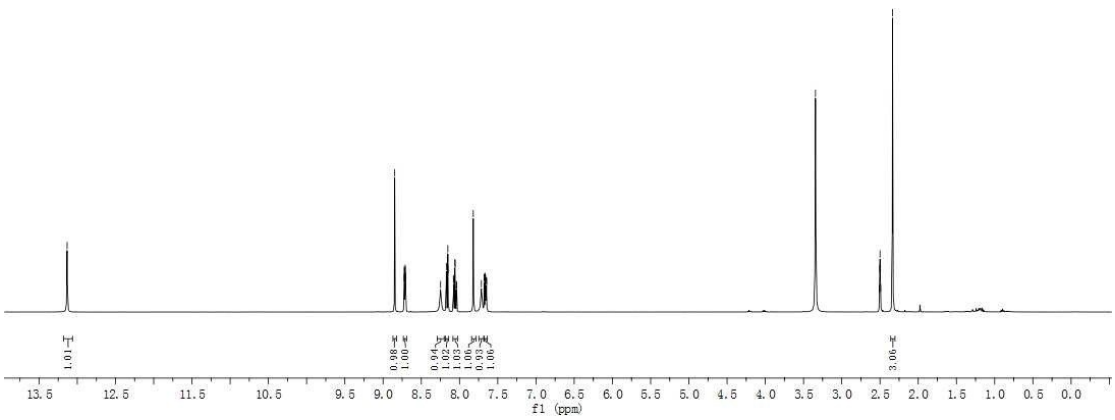
2p

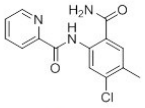

13.15
9.19
8.73
8.72
8.18
8.16
8.08
7.69
7.68
7.67
7.66
7.58
3.33
2.51
2.50
2.49
2q
0.95
0.95
1.00
0.93
1.03
1.06
1.02
1.05
2.00
f1 (ppm)


169.72
162.79
149.39
148.71
139.51
138.15
131.48
130.15
128.52
127.18
122.43
120.56
99.16
40.13
39.92
39.71
39.50
39.29
39.08
38.87
2q
f1 (ppm)



2r





2r