Supporting Information

Copper Catalysed Direct Amidation of Methyl Groups with N-H Bonds

Yao Huang, Tieqiao Chen,* Qiang Li, Yongbo Zhou, and Shuang-Feng Yin*

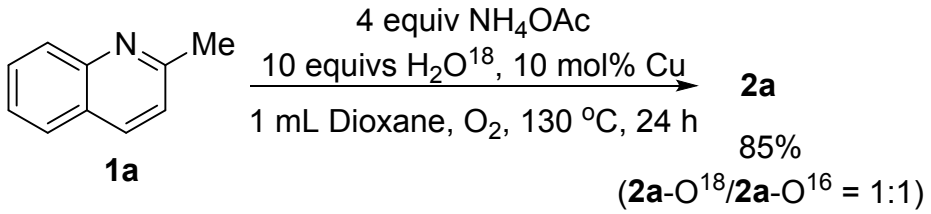
State Key Laboratory of Chemo/Biosensing and Chemometrics, College of Chemistry and Chemical Engineering, Hunan University, Changsha 410082, China; National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Ibaraki 305-8565, Japan.

General information

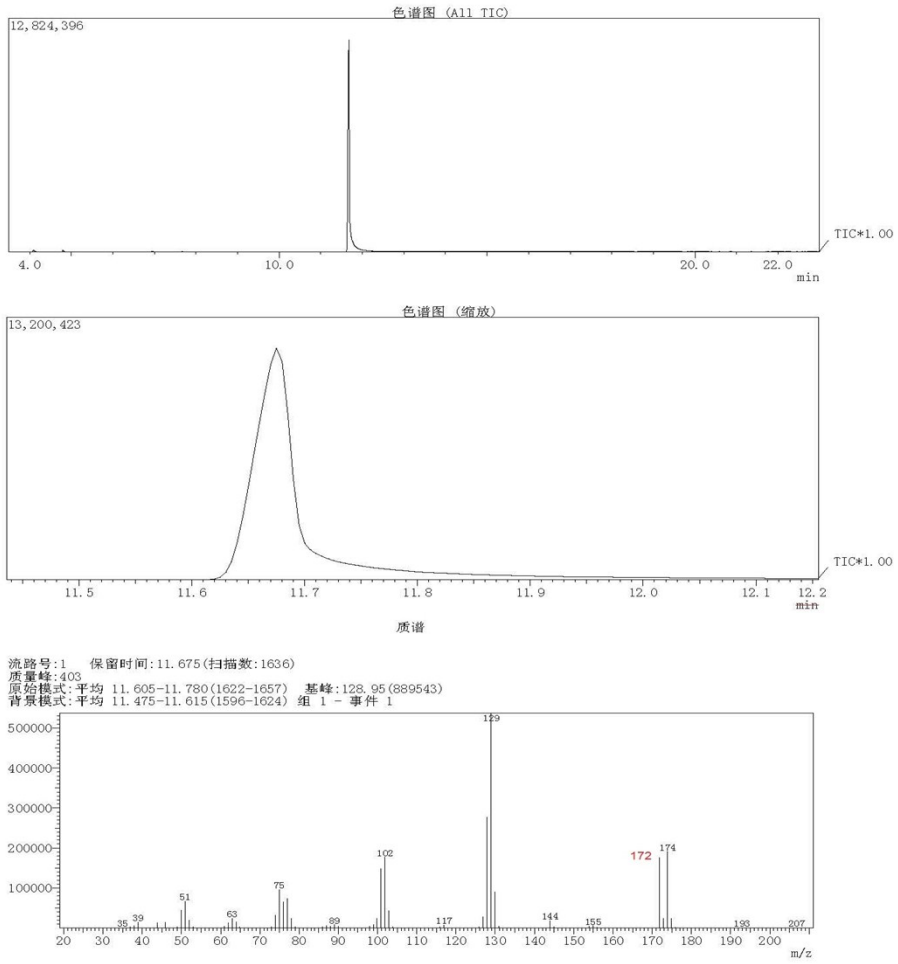
All reactions were carried out under dioxygen atmosphere (1 atm). Column chromatography was performed using Silica Gel 60 (particle size 37-54 μm). The pure products were obtained by column chromatography using ethyl acetate/petroleum ether as an eluent. GC analysis was performed on GC 7820A (Shimadzu). GC-MS results were recorded on GC-MS QP2010 (Shimadzu). The ^{1}H NMR and ^{13}C NMR data were data were acquired on a Brucker ADVANCE III spectrometer (400 MHz for ^{1}H NMR spectroscopy and 100 MHz for ^{13}C NMR spectroscopy). Exact mass was conducted by the Analytical Center at Hunan University, China.

^{18}O experiments

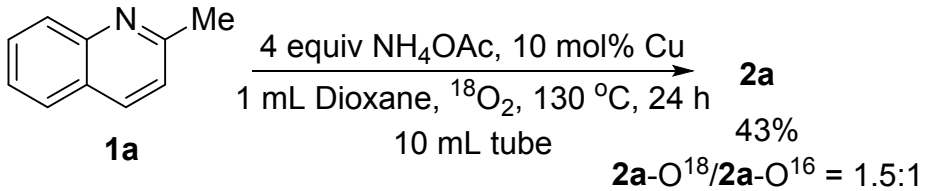
A.



Under an oxygen atmosphere (1 atm), **1a** (0.2 mmol), NH_4OAc (0.8 mmol, dried in vacuum), metallic Cu (0.02 mol), H_2O^{18} (2 mmol) and dioxane (1.0 mL, dried by CaH_2) were placed into a glass tube (25 mL) and sealed. The mixture was heated at 130 °C for 24 hours. The yield and ratio were determined by GC and GC-MS.

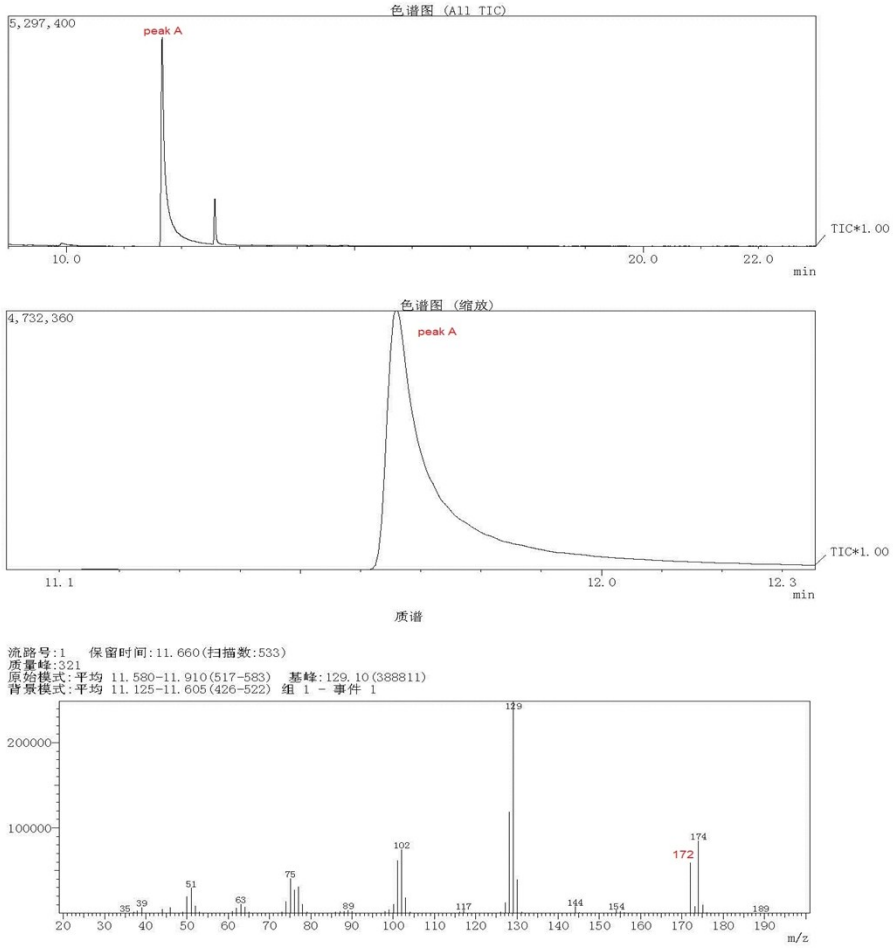


B.

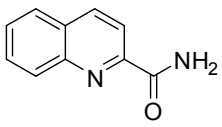


1a

Under an $^{18}O_2$ atmosphere (1 atm), **1a** (0.2 mmol), NH_4OAc (0.8 mmol, dried in vacuum), metallic Cu (0.02 mol) and dioxane (1.0 mL, dried by CaH_2) were placed into a glass tube (10 mL) and sealed. The mixture was heated at 130 °C for 24 hours. The yield and ratio were determined by GC and GC-MS.

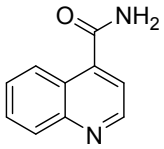


Characterization and analytical data of products



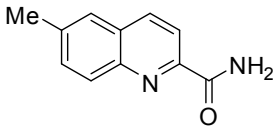
2a[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (b, 2H), 8.13 (d, *J* = 8.4 Hz, 2H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.76-7.80 (m, 1H), 7.62-7.66 (m, 1H), 6.01 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 149.3, 146.6, 137.6, 130.2, 129.8, 129.4, 128.2, 127.8, 118.9.



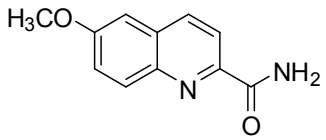
2b[2]

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.92 (d, J = 4.4 Hz, 1 H), 8.36 (s, 1 H (0.4 H, partial H exchanged with DMSO-d_6)), 8.16 (d, J = 8.4 Hz, 1 H), 8.07 (d, J = 8.0 Hz, 1 H), 7.91 (s, 1 H (0.4 H, partial H exchanged with DMSO-d_6)), 7.80-7.84 (m, 1 H), 7.66-7.70 (m, 1 H), 7.55 (d, J = 4.4 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 169.7 (m), 150.7, 148.0, 142.5 (t), 130.6, 129.4, 128.1, 125.8, 124.3, 119.4.



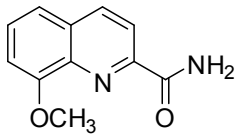
2c

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.43 (d, J = 8.8 Hz, 1H), 8.24 (s, 1H), 8.11 (d, J = 8.8 Hz, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.83 (s, 1H), 7.69-7.71 (m, 2H), 3.32 (mixed with DMSO, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.7, 150.1, 145.1, 138.3, 137.3, 133.1, 129.5, 129.3, 127.1, 119.1, 21.7. HRMS (EI) m/z: [M] Calcd for $C_{11}H_{10}N_2O$ 186.0793, Found 186.0790. m.p. 228-232 ^{o}C.



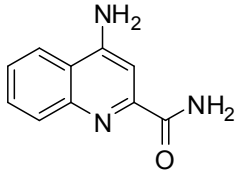
2d

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, J = 8.4 Hz, 1H), 8.19 (d, J = 8.4 Hz, 1H), 8.01 (s, 1 H), 8.00 (d, J = 9.2 Hz, 1H), 7.42 (dd, J_1 = 2.4 Hz, J_2 = 9.2 Hz, 1H), 7.12 (d, J = 2.4 Hz, 1H), 5.69 (s, 1 H), 3.96 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 159.1, 147.1, 142.7, 135.9, 131.3, 130.8, 123.3, 119.3, 104.9, 55.7. HRMS (EI) m/z: [M] Calcd for $C_{11}H_{10}N_2O_2$ 202.0742, Found 202.0733. m.p. 215-218 ^{o}C.



2e[3]

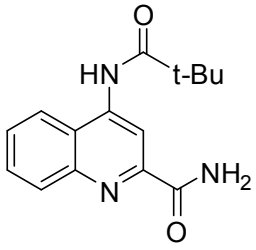
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, J = 8.8 Hz, 1H), 8.29 (d, J = 8.8 Hz, 1H), 8.19 (s, 1H), 7.56 (dd, J_1 = 8.0 Hz, J_2 = 8.0 Hz, 1H), 7.45 (d, J = 8.4 Hz, 1H), 7.10 (d, J = 7.6 Hz, 1H), 5.85 (s, 1 H), 4.09 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 155.6, 148.3, 138.6, 137.4, 130.6, 128.5, 119.6, 119.5, 108.4, 56.1.



2f

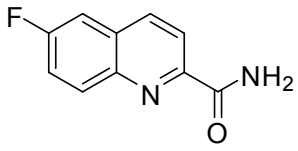
^{1}H NMR (400 MHz, THF-d_8) δ 8.02 (br s, 2H), 7.86 (br s, 1H), 7.58 (br s, 1H), 7.42 (br s, 2H),

6.77 (s, 1H), 6.40 (br s, 2H); ^{13}C NMR (100 MHz, THF-d_8) δ 166.9, 152.8, 151.0, 148.0, 129.7, 128.9, 124.4, 121.4, 119.3, 100.2. HRMS (EI) m/z: [M] Calcd for $C_{10}H_9N_3O$ 187.0746, Found 187.0747. m.p. 232-236 °C.



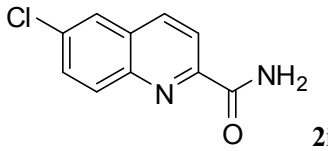
2g

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.91 (s, 1H), 8.22 (s, 1H), 8.08 (d, J = 8.8 Hz, 1H), 8.00 (s, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.75 (dd, J_1 = 8.0 Hz, J_2 = 6.4 Hz, 1H), 7.63 (dd, J_1 = J_2 = 7.6 Hz, 1H), 5.90 (s, 1 H), 1.45 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 166.8, 150.3, 147.3, 142.0, 131.0, 129.9, 127.9, 121.7, 119.6, 109.9, 40.4, 27.6. HRMS (EI) m/z: [M] Calcd for $C_{15}H_{17}N_3O_2$ 271.1321, Found 271.1322. m.p. 233-236 °C.



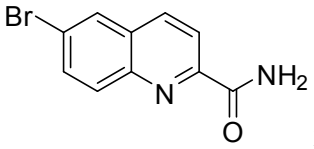
2h

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (dd, J_1= 8.8 Hz, J_2 = 23.2 Hz, 1H), 8.30 (d, J_1 = 14.4 Hz, 1H), 8.13 (dd, J_1 = 5.2 Hz, J_2 = 9.2 Hz, 1H), 8.02 (s, 1H), 7.49-7.58 (m, 2H), 5.87 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 161.5 ($J_{F\text{-}C}$ = 249.7 Hz), 148.9 (($J_{F\text{-}C}$ = 2.6 Hz), 143.7, 136.8 ($J_{F\text{-}C}$ = 5.6 Hz), 132.5 ($J_{F\text{-}C}$ = 9.3 Hz), 130.2 ($J_{F\text{-}C}$ = 10.3 Hz), 120.7 ($J_{F\text{-}C}$ = 25.9 Hz), 119.6, 110.8 ($J_{F\text{-}C}$ = 21.7 Hz). HRMS (EI) m/z: [M] Calcd for $C_{10}H_7FN_2O$ 190.0542, Found 190.0538. m.p. 196-199 °C.



2i[4]

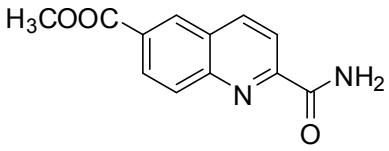
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, J = 8.4 Hz, 1H), 8.24 (d, J = 8.4 Hz, 1H), 8.06 (d, J = 9.2 Hz, 1H), 8.03 (s, 1H), 7.88 (d, J = 2.0 Hz, 1H), 7.71 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1H), 5.80 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 149.6, 145.0, 136.6, 134.1, 131.4, 131.2, 129.9, 126.4, 119.8.



2j[5]

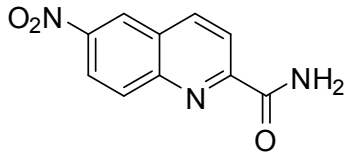
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.54 (d, J = 8.8 Hz, 1H), 8.40 (d, J = 2.0 Hz, 1H), 8.32 (s, 1 H), 8.20 (d, J = 8.4 Hz, 1H), 8.06 (d, J = 8.8 Hz, 1H), 7.99 (dd, J_1 = 2.0 Hz, J_2 = 8.4 Hz, 1H), 7.83 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.4, 151.4, 145.1, 137.5, 134.0, 131.9, 130.5, 130.4,

121.6, 120.0.



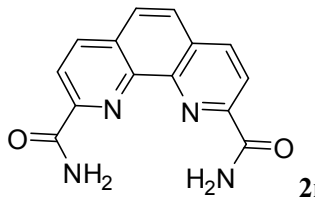
2k

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.78 (s, 1H), 8.77 (d, J = 8.8 Hz, 1H), 8.36 (s, 1H), 8.31 (dd, J_1 = 1.6 Hz, J_2 = 8.8 Hz, 1H), 8.22 (d, J = 8.4 Hz, 1H), 8.21 (d, J = 8.8 Hz, 1H), 7.87 (s, 1H), 3.97 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.3, 166.2, 152.9, 148.3, 139.8, 131.3, 130.4, 129.7, 129.1, 128.5, 119.9, 53.0. HRMS (EI) m/z: [M] Calcd for $C_{12}H_{10}N_2O_3$ 230.0691, Found 230.0683. m.p. 254-256 ^{o}C.



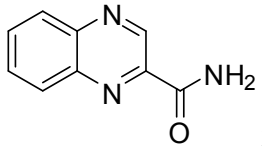
2l

^{1}H NMR (400 MHz, DMF-d_7) δ 9.20 (d, J = 2.4 Hz, 1H), 8.99 (d, J = 8.4 Hz, 1H), 8.63 (dd, J_1 = 2.4 Hz, J_2 = 9.2 Hz, 1H), 8.58 (s, 1H), 8.42 (d, J = 8.4 Hz, 1H), 8.35 (d, J = 9.2 Hz, 1H), 7.95 (s, 1H); ^{13}C NMR (100 MHz, DMF-d_7) δ 165.9, 153.9, 148.7, 146.4, 140.3, 131.5, 128.3, 125.1, 123.7, 120.4. HRMS (EI) m/z: [M] Calcd for $C_{10}H_7N_3O_3$ 217.0487, Found 217.0482. m.p. 285-298 ^{o}C.



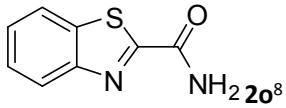
2m[6]

^{1}H NMR (400 MHz, DMF-d_7) δ 9.20 (s, 2H), 8.96 (d, J = 8.4 Hz, 2H), 8.72 (d, J = 8.4 Hz, 2H), 8.41 (s, 2H), 7.98 (s, 2H); ^{13}C NMR (100 MHz, DMF-d_7) δ 166.6, 150.7, 144.7, 138.3, 130.8, 128.2, 121.3.



2n[7]

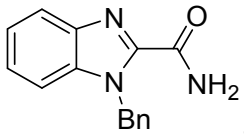
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (s, 1H), 8.21 (d, J = 7.6 Hz, 1H), 8.14 (d, J = 8.0 Hz, 1H), 7.84-7.91 (m, 3H), 6.07 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 144.0, 143.9, 143.1, 140.4, 131.8, 130.9, 129.8, 129.5.



2o[8]

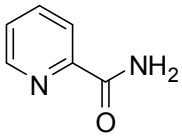
^{1}H NMR (400 MHz, DMF-d_7) δ 8.36 (s, 1H), 8.13 (d, J = 7.6 Hz, 1H), 8.00 (d, J = 8.0 Hz, 1H), 7.99 (s, 1H), 7.44-7.54 (m, 2H); ^{13}C NMR (100 MHz, DMF-d_7) δ 165.2, 161.7, 153.4, 137.0,

127.1, 127.0, 124.3, 123.0.



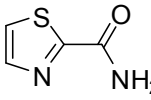
2p[9]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.82 (m, 1H), 7.70 (s, 1H), 7.39-7.43 (m, 1H), 7.32-7.36 (m, 2H), 7.21-7.30 (m, 5H), 6.00 (s, 2H), 5.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 142.4, 141.2, 136.7, 136.6, 128.8, 127.8, 127.0, 125.1, 123.8, 120.9, 111.3, 48.7.



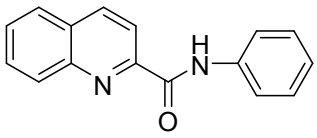
2q[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, J = 6.4 Hz, 1H), 8.21 (d, J = 8.0 Hz, 1H), 7.85-7.89 (m, 2H), 7.46 (ddd, J_1 = 1.2 Hz, J_2 = 4.8 Hz, J_3 = 6.0 Hz, 1H), 5.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 149.5, 148.3, 137.3, 126.5, 122.5.



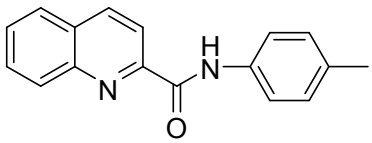
2r[10]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 3.2 Hz, 1H), 7.62 (d, J = 3.2 Hz, 1H), 7.18 (s, 1H), 5.87 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.95, 161.63, 143.79, 125.26. ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 161.4, 143.8, 125.3.



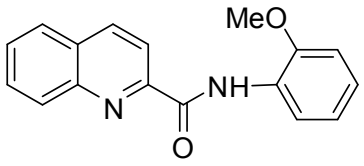
2s[11]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.24 (s, 1H), 8.35-8.42 (m, *2*H), 8.19 (d, J = 8.4 Hz, 1H), 7.79-7.92 (m, 4H), 7.65 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.42 (dd, J_1 = J_2 = 8.0 Hz, 2H), 7.17 (t, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 149.7, 146.3, 137.9, 137.8, 130.3, 129.7, 129.4, 129.1, 128.2, 127.8, 124.4, 119.8, 118.8.



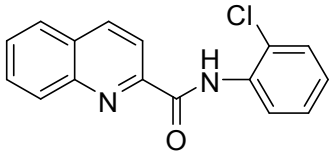
2t[12]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.18 (s, 1H), 8.35-8.41 (m, 2H), 8.18 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.80 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.65 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.22 (d, J = 8.4 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 149.8, 146.3, 137.8, 135.3, 134.0, 130.3, 129.7, 129.6, 129.4, 128.1, 127.8, 119.8, 119.7, 118.8, 21.0.



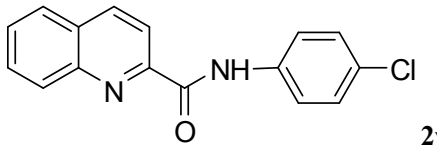
2u[12]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.82 (s, 1H), 8.65 (dd, J_1= 1.6 Hz, J_2 = 8.0 Hz, 1H), 8.34-8.41 (m, 2H), 8.21 (d, J = 8.4 Hz, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.79 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.64 (dd, J_1 = J_2 = 7.6 Hz, 1H), 7.03-7.13 (m, 2H), 6.96-6.98 (dd, J_1= 1.2 Hz, J_2 = 8.0 Hz, 1H), 4.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 150.2, 148.9, 146.4, 137.7, 130.1, 130.0, 129.4, 128.0, 127.8, 127.7, 124.0, 121.1, 119.8 , 118.8, 110.2, 56.0.



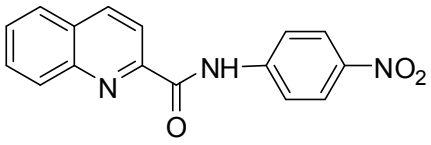
2v[12]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.98 (s, 1H), 8.70 (d, J = 8.4 Hz, 1H), 8.38 (br s, 2H), 8.21 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.81 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.66 (dd, J_1 = J_2 = 7.6 Hz, 1H), 7.46 (d, J = 8.0 Hz, 1H), 7.36 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.09 (ddd, J_1 = 1.2 Hz, J_2 = 7.6 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 149.5, 146.4, 137.9, 134.8, 130.4, 130.1, 129.5, 129.3, 128.3, 127.8, 127.7, 124.6, 123.6, 121.0, 118.7.



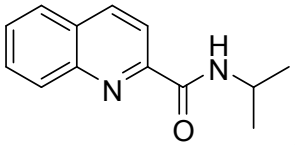
2w[12]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.25 (s, 1H), 8.38 (br s, 2H), 8.18 (d, J = 8.4 Hz, 1H), 7.92 (d, J = 7.6 Hz, 1H), 7.80-7.84 (m, 3H), 7.67 (dd, J_1 = J_2 = 8.0 Hz, 1H), 7.38 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 149.4, 146.3, 138.0, 136.4, 130.4, 129.7, 129.5, 129.3, 129.2, 128.3, 127.9, 121.0, 118.7.



2x[13]

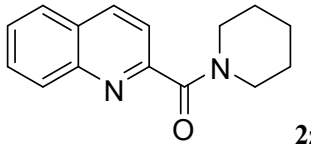
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.59 (s, 1H), 8.38-8.43 (m, 2H), 8.31 (d, J = 9.2 Hz, 2H), 8.21 (d, J = 8.4 Hz, 1H), 8.04 (d, J = 9.2 Hz, 2H), 7.95 (d, J = 8.4 Hz, 1H), 7.85(dd, J_1 = J_2 = 8.0 Hz, 1H), 7.70 (dd, J_1 = J_2 = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 148.7, 146.3, 143.7, 143.5, 138.3, 130.7, 129.7, 129.7, 128.7, 127.9, 125.3, 119.3, 118.7.



2y[14]

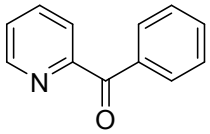
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29-8.33 (m, 2H), 8.11-8.13 (m, 2H), 7.88 (d, J = 8.4 Hz, 1H),

7.74-7.79 (m, 1H), 7.59-7.63 (m, 1H), 4.30-4.39 (m, 1H), 1.35 (d, J = 6.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6, 150.1, 146.5, 137.4, 130.0, 129.7, 129.3, 127.8, 127.8, 118.9, 41.6, 22.9.



2z[15]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, J = 8.4 Hz, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.85 (d, J = 8.0 Hz, 1H), 7.74-7.78 (m, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.58-7.62 (m, 1H), 3.81 (t, J = 5.2 Hz, 2H), 3.52 (t, J = 5.2 Hz, 2H), 1.73 (br s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 154.4, 146.8, 137.1, 130.0, 129.7, 127.9, 127.7, 127.4, 120.4, 48.4, 43.4, 26.5, 25.6, 24.6.



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, J = 4.4 Hz, 1H), 8.02-8.07 (m, 3H), 7.86, 7.90 (m, 1H), 7.58 (t, J = 7.6 Hz, 1H), 7.46-7.49 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 155.1, 148.6, 137.1, 136.3, 133.0, 131.0, 128.2, 126.2, 124.6.

Reference

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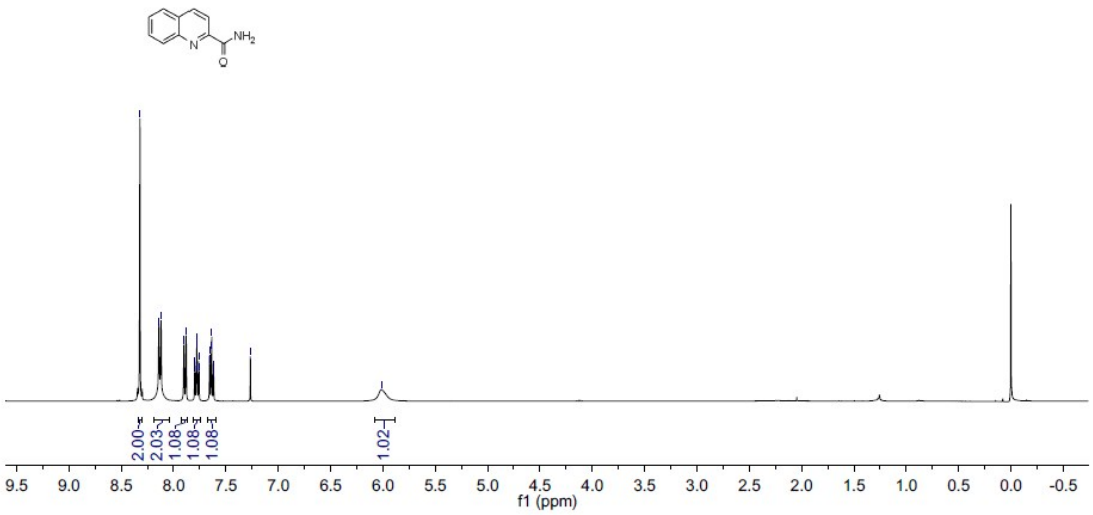
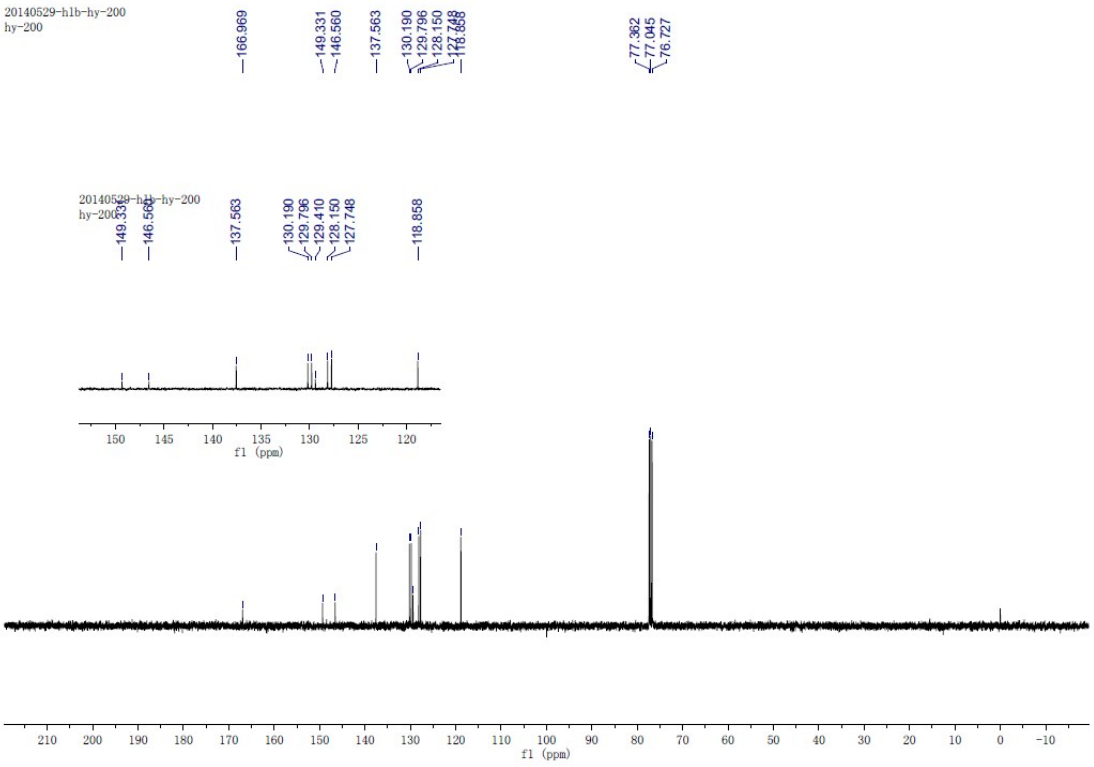
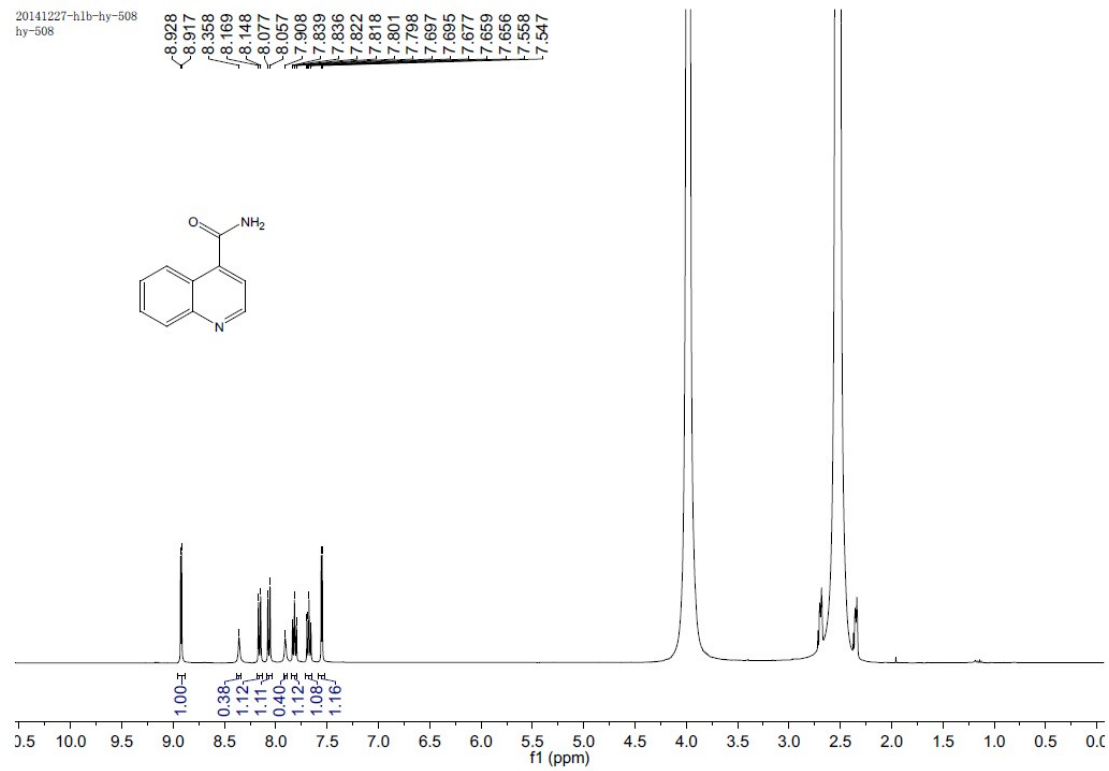
Copies of ^{1}H NMR and ^{13}C NMR spectroscopies



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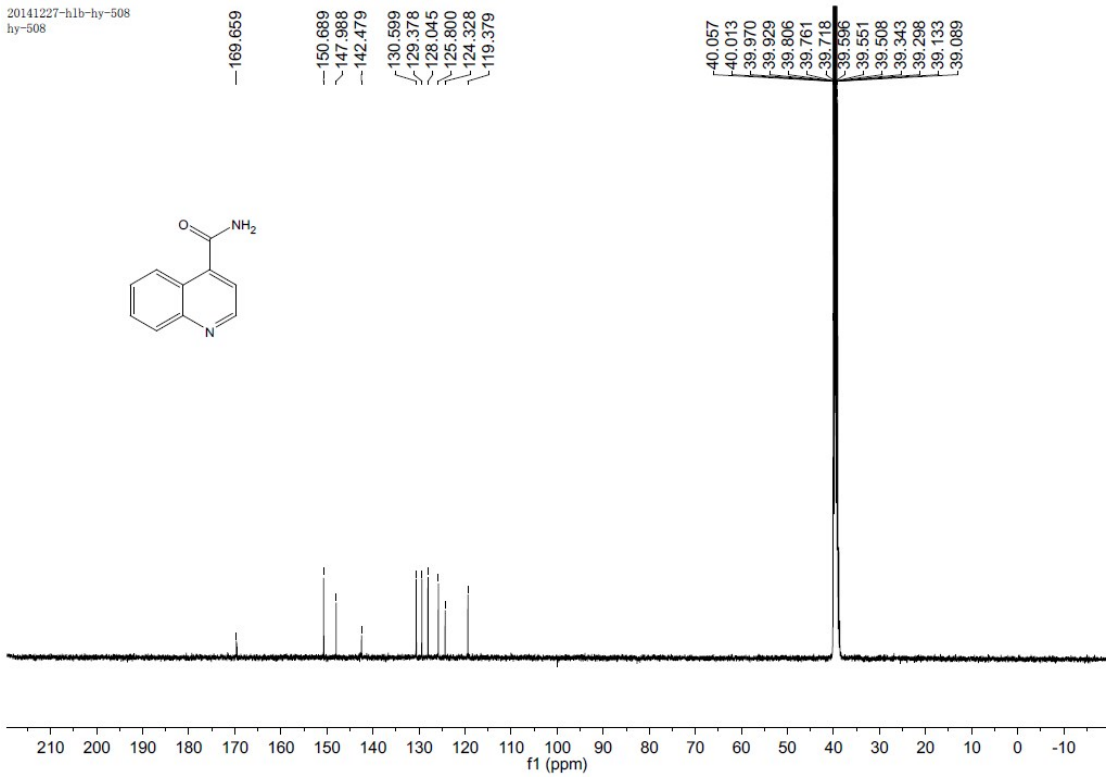
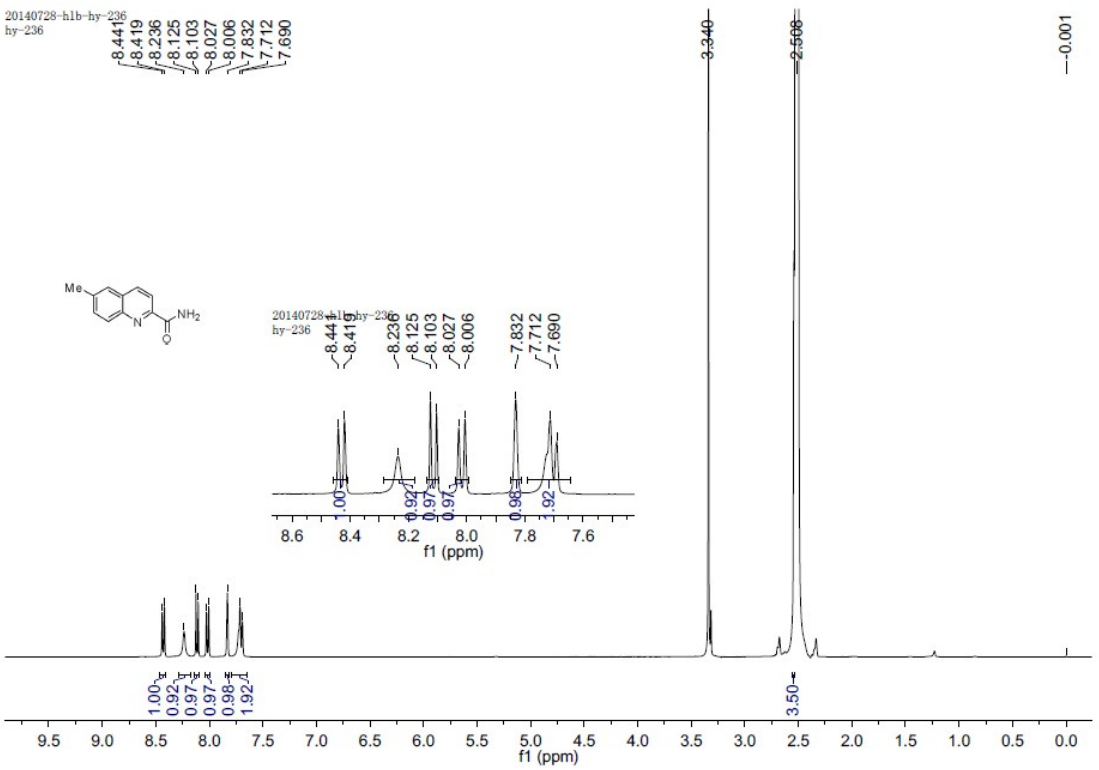
hy-508
8.928
8.917
8.358
8.169
8.148
8.077
8.057
7.908
7.839
7.836
7.822
7.818
7.801
7.798
7.697
7.695
7.677
7.659
7.656
7.558
7.547
O
NH2
N
1.00
0.38
1.12
1.11
0.40
1.12
1.08
1.16
f1 (ppm)


20141227-h1b-hy-508
hy-508
169.659
150.689
147.988
142.479
130.599
129.378
128.045
125.800
124.328
119.379
40.057
40.013
39.970
39.929
39.806
39.761
39.718
39.596
39.551
39.508
39.343
39.298
39.133
39.089
O
NH2
N
f1 (ppm)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hy-236
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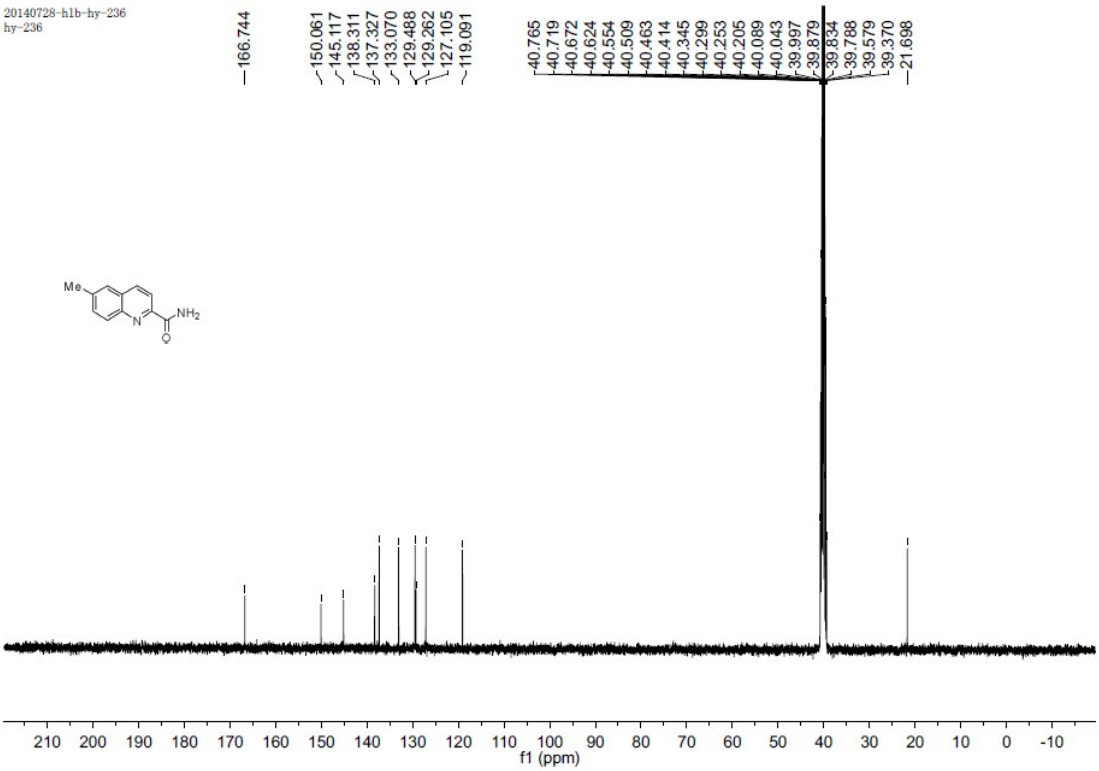
8.419
8.236
8.125
8.103
8.027
8.006
7.832
7.712
7.690
3.340
2.508
0.001
Me
NH2
O
N
1.00
0.92
0.97
0.97
0.98
1.92
3.50
f1 (ppm)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hy-236
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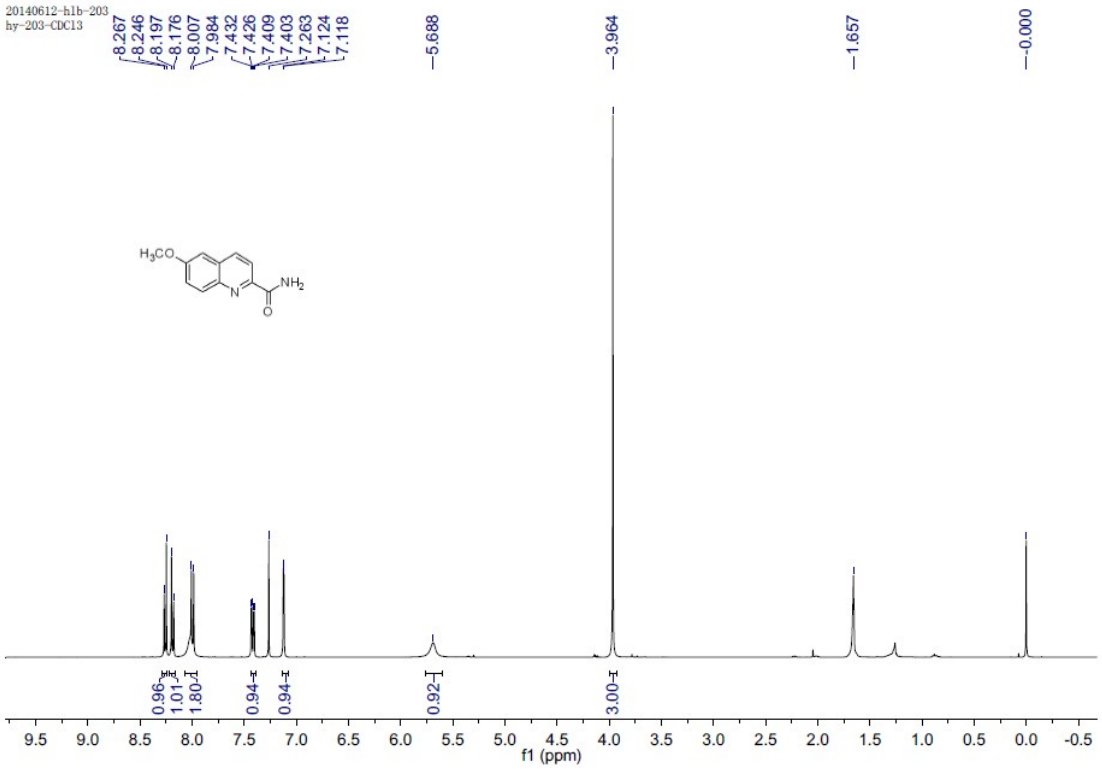
150.061
145.117
138.311
137.327
133.070
129.488
129.262
127.105
119.091
40.765
40.719
40.672
40.624
40.554
40.509
40.463
40.414
40.345
40.299
40.253
40.205
40.089
40.043
39.997
39.879
39.834
39.788
39.579
39.370
21.698
Me
NH2
O
N
f1 (ppm)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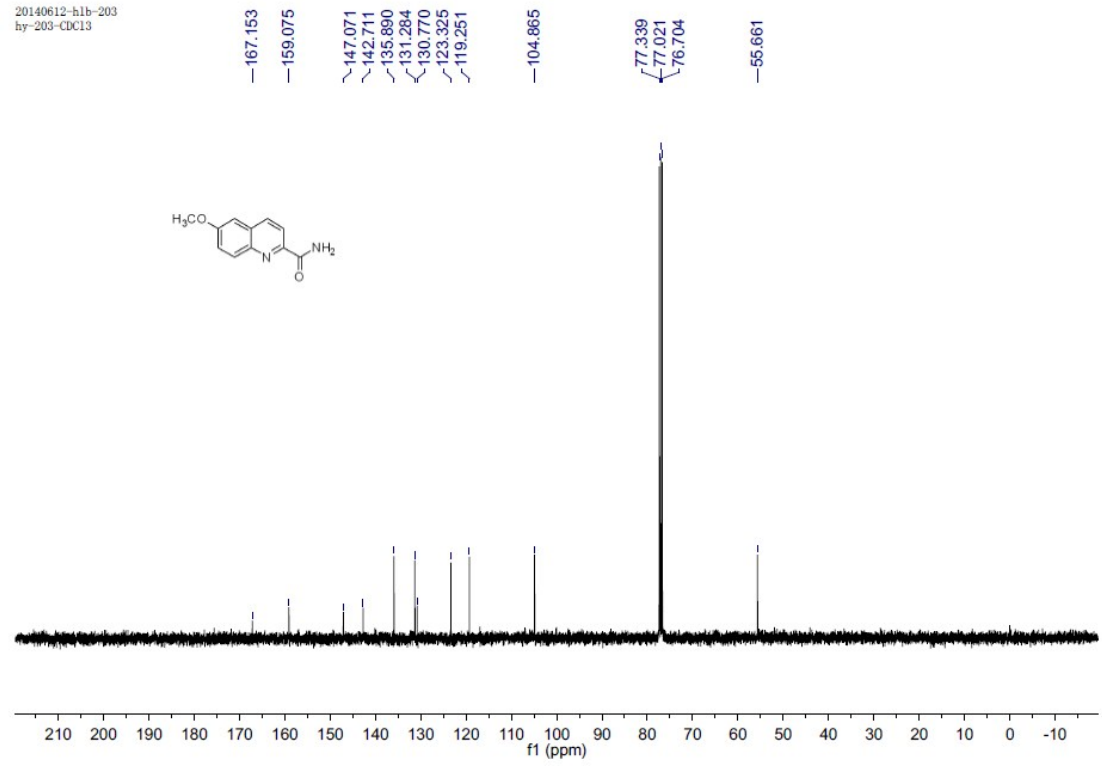
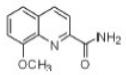
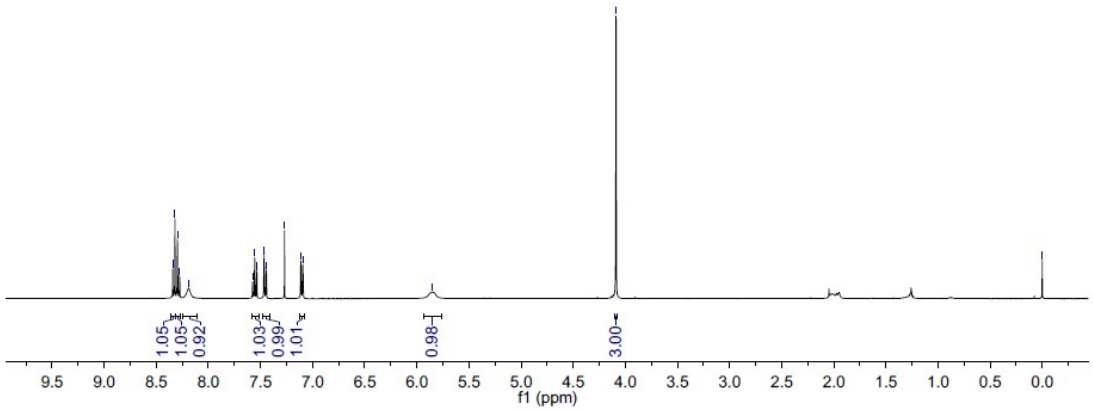
8.246
8.197
8.176
8.007
7.984
7.432
7.426
7.409
7.403
7.263
7.124
7.118
5.688
3.964
1.657
0.000
H3CO
NH2
O
N
0.96
1.01
1.80
0.94
0.94
0.92
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


20140612-hlb-203
hy-203-CDCl3
167.153
159.075
147.071
142.711
135.890
131.284
130.770
123.325
119.251
104.865
77.339
77.021
76.704
55.661
H3CO
NH2
O
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

20140629-hlb-hy-218
hy-218-CDCl3

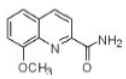
8.342
8.320
8.297
8.276
8.189
7.576
7.556
7.536
7.465
7.444
7.269
7.112
7.093
5.848
4.089
0.000

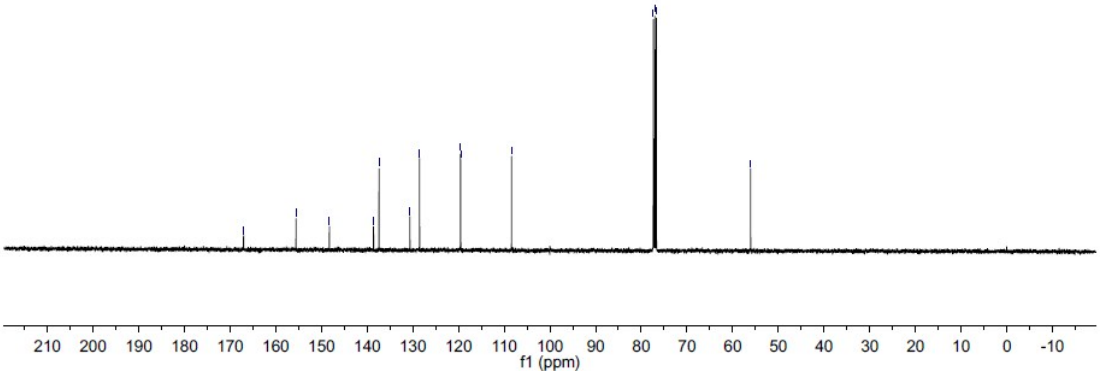


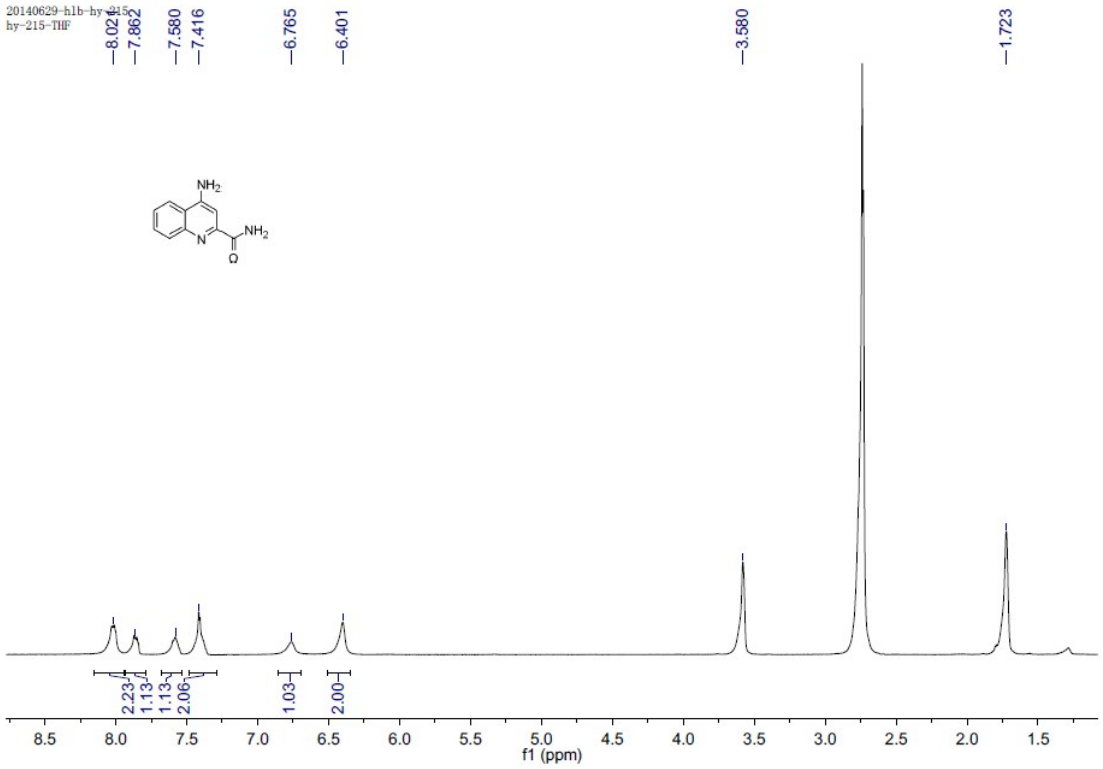


20140629-hlb-hy-218
hy-218-CDCl3

167.037
155.566
148.264
138.589
137.394
130.625
128.521
119.563
119.505
108.383
77.361
77.043
76.726
56.061



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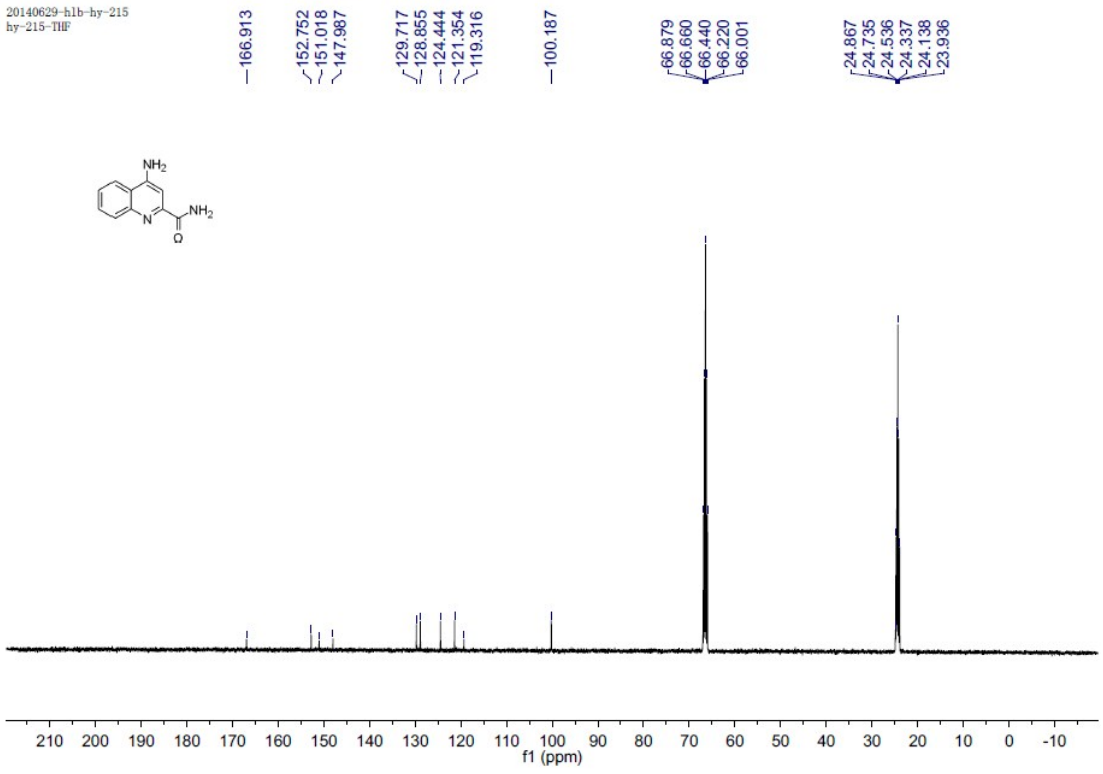
7.416
6.765
6.401
3.580
1.723
NH2
NH2
O
N
2.23
1.13
1.13
2.06
1.03
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)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hy-215-THF
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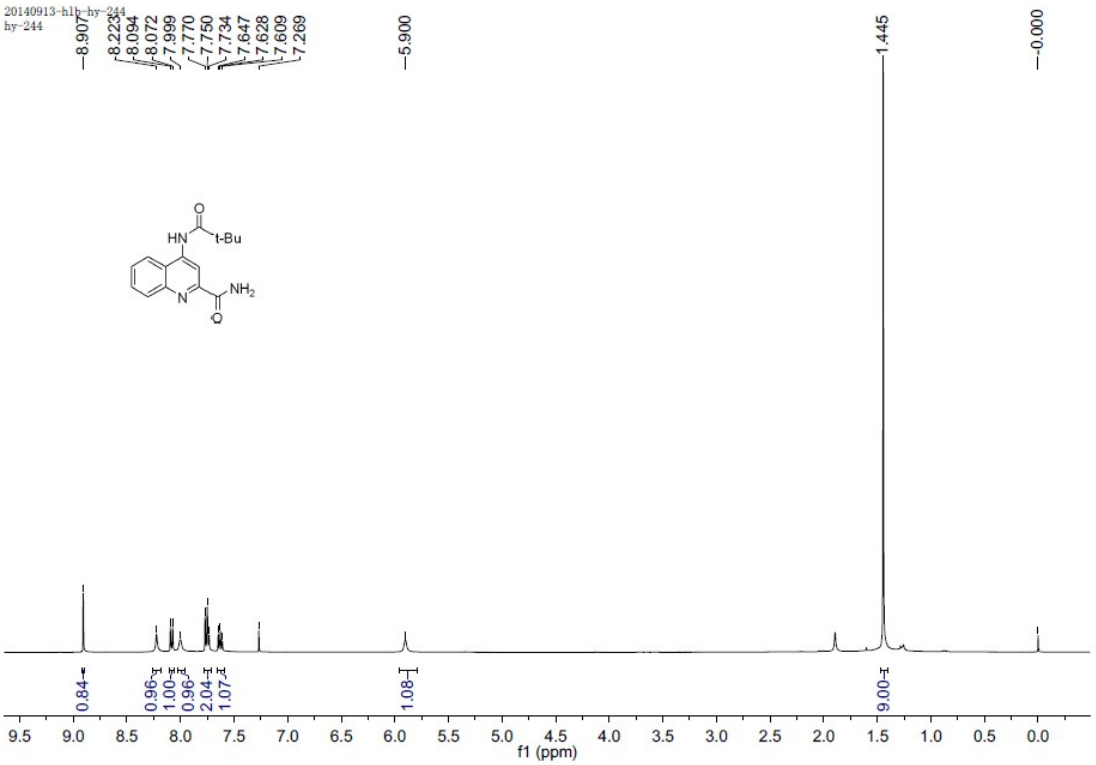
151.018
147.987
129.717
128.855
124.444
121.354
119.316
100.187
66.879
66.660
66.440
66.220
66.001
24.867
24.735
24.536
24.337
24.138
23.936
NH2
NH2
O
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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hy-244
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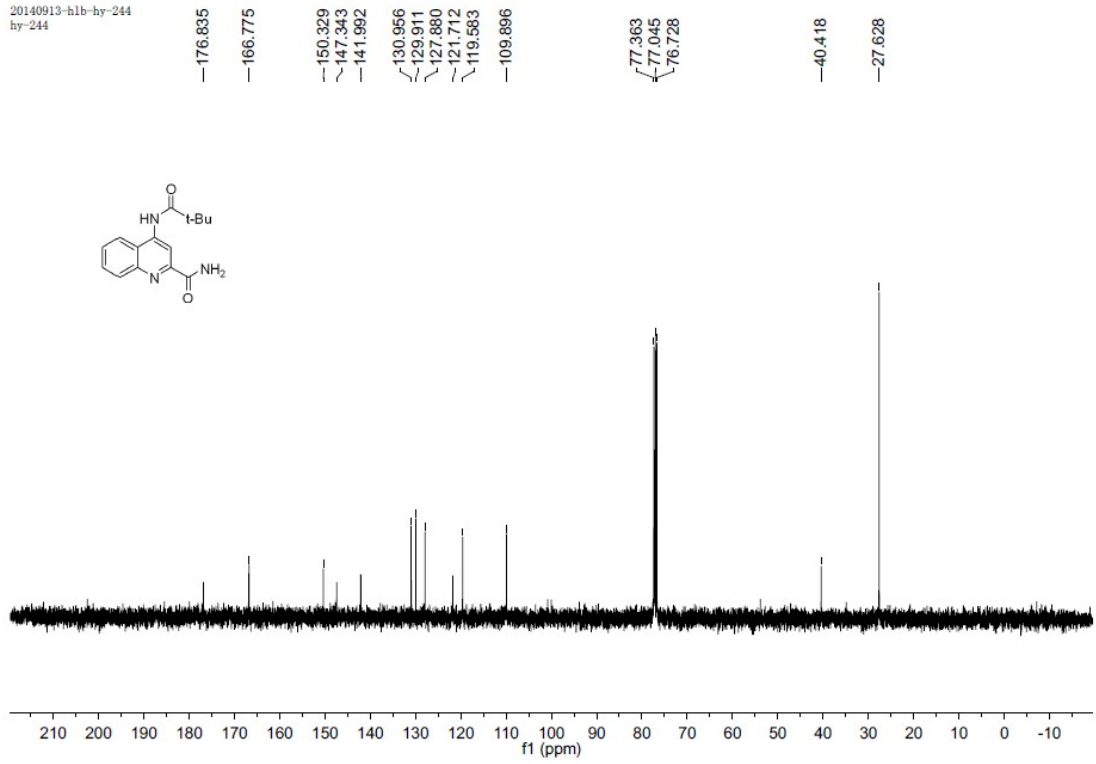
8.223
8.094
8.072
7.999
7.770
7.750
7.734
7.647
7.628
7.609
7.269
5.900
1.445
0.000
HN
t-Bu
NH2
O
0.84
0.96
1.00
0.96
2.04
1.07
1.08
9.00
f1 (ppm)


20140913-h1b-hy-244
hy-244
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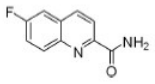
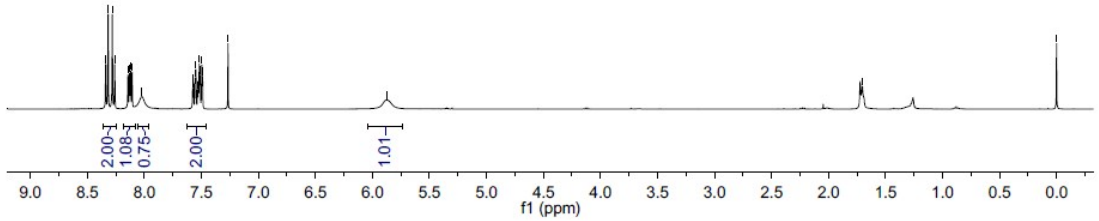
166.775
150.329
147.343
141.992
130.956
129.911
127.880
121.712
119.583
109.896
77.363
77.045
76.728
40.418
27.628
HN
t-Bu
NH2
O
f1 (ppm)





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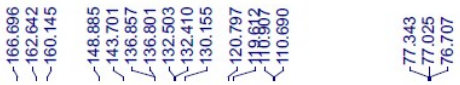
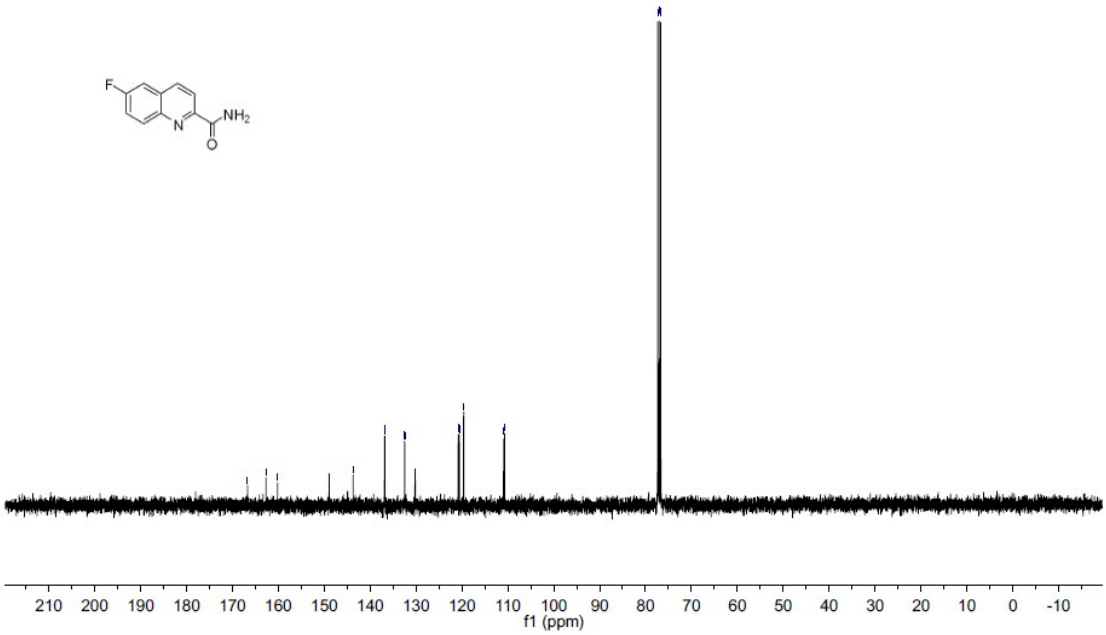
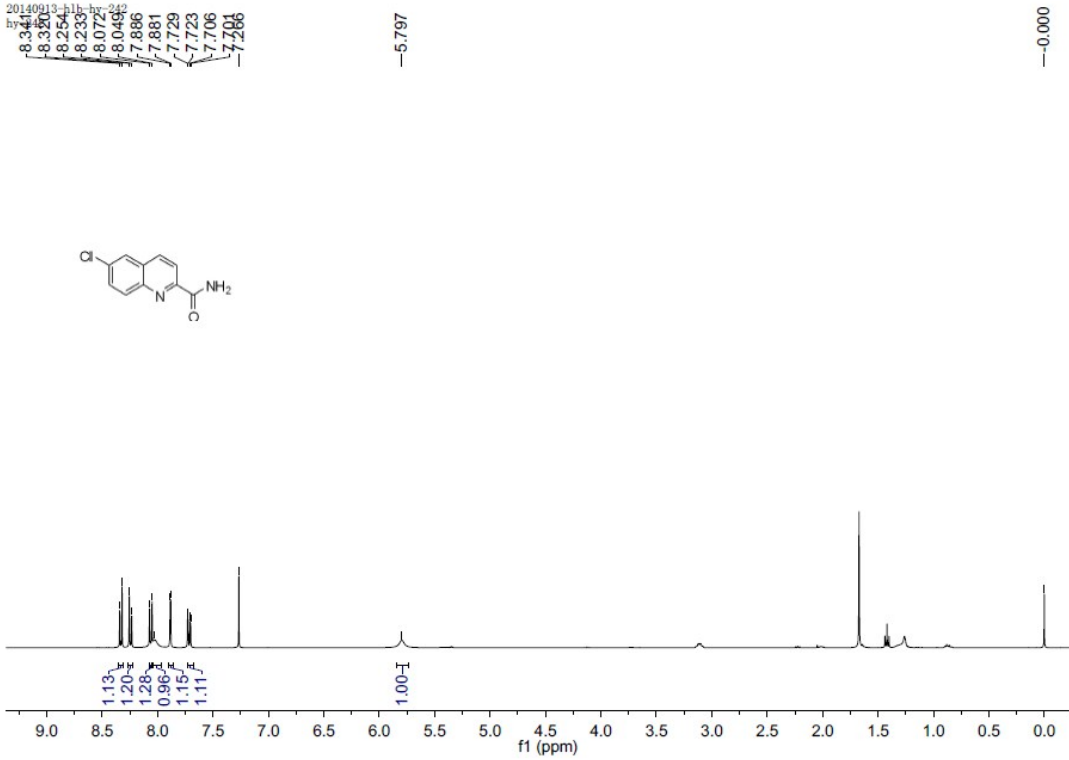
hy-204-CDCl3



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hy-242
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8.233
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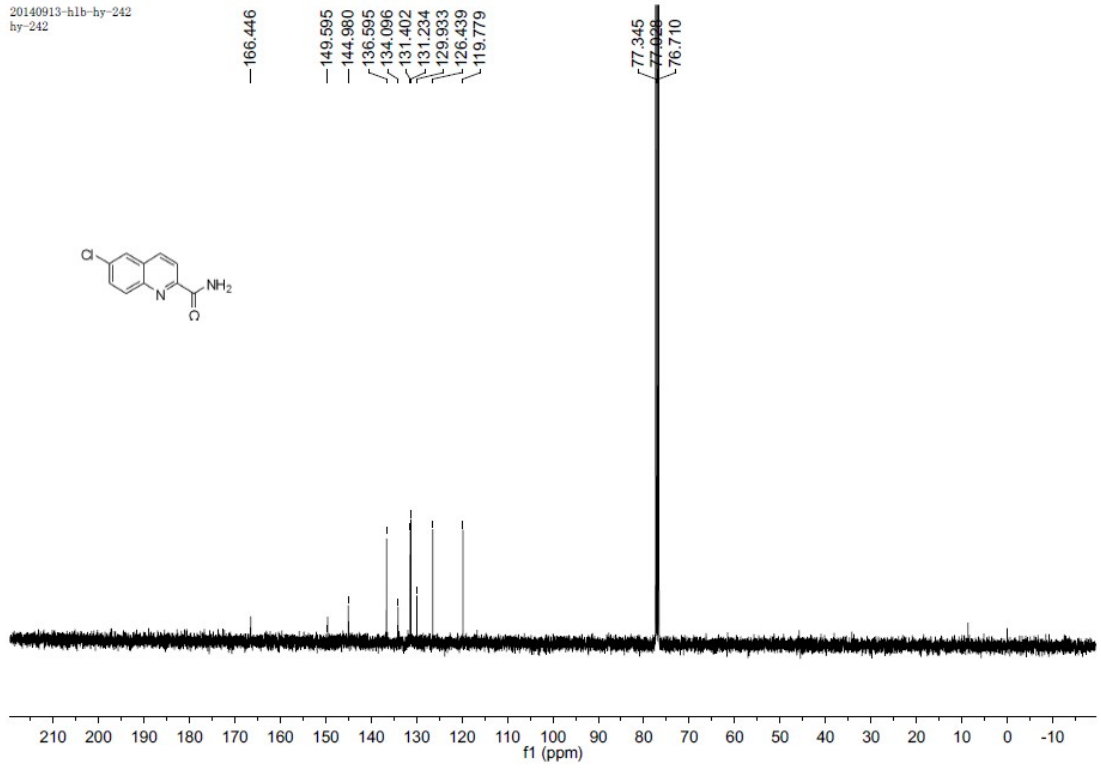
8.049
7.886
7.881
7.729
7.723
7.706
7.701
7.266
5.797
0.000
Cl
N
NH2
O
1.13
1.20
1.28
0.96
1.15
1.11
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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hy-242
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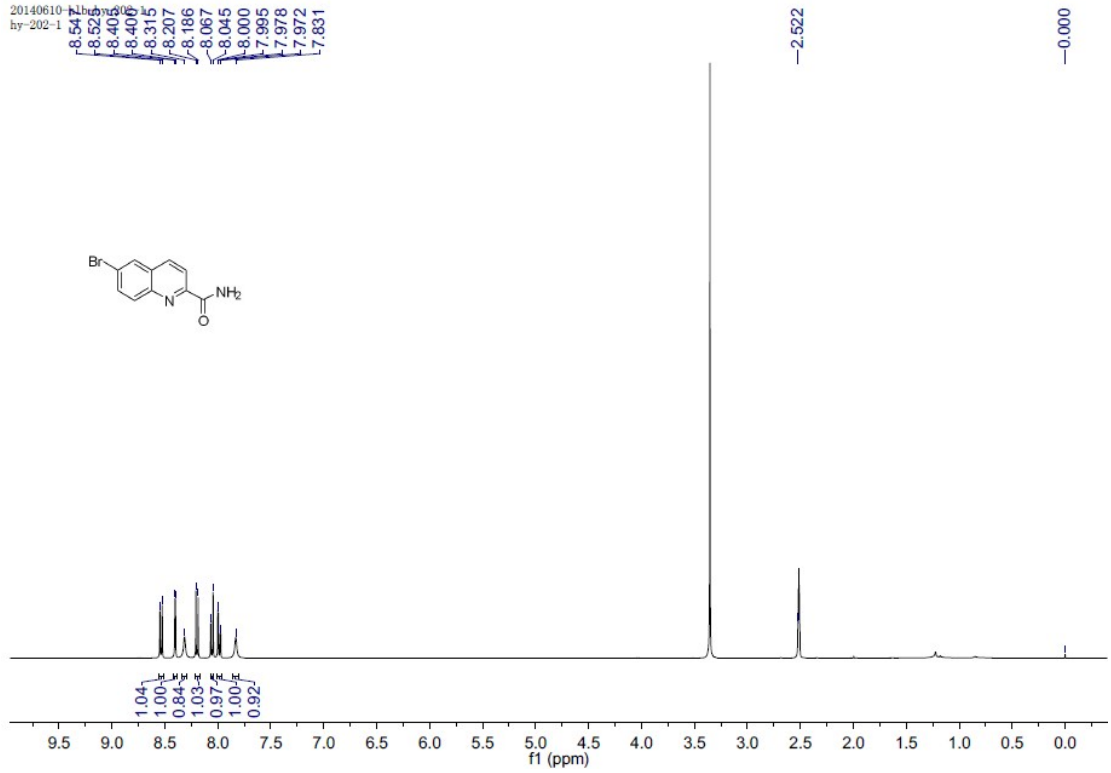
136.595
134.096
131.402
131.234
129.933
126.439
119.779
77.345
77.028
76.710
Cl
N
NH2
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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hy-202-1
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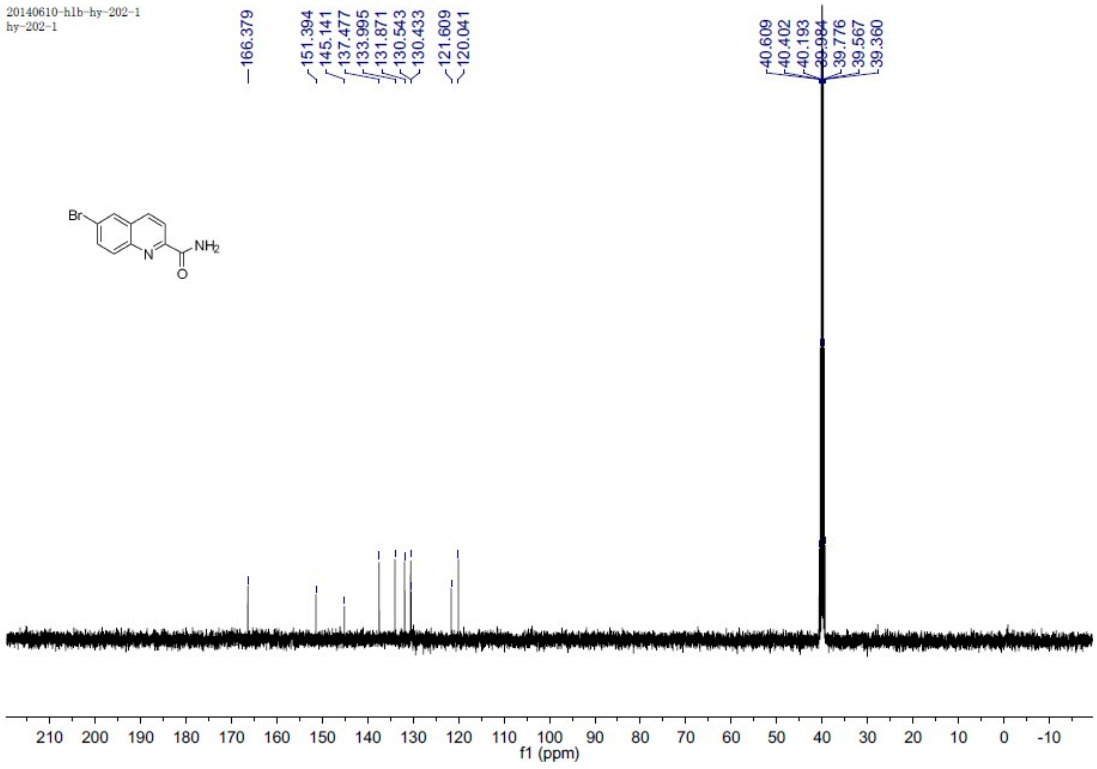
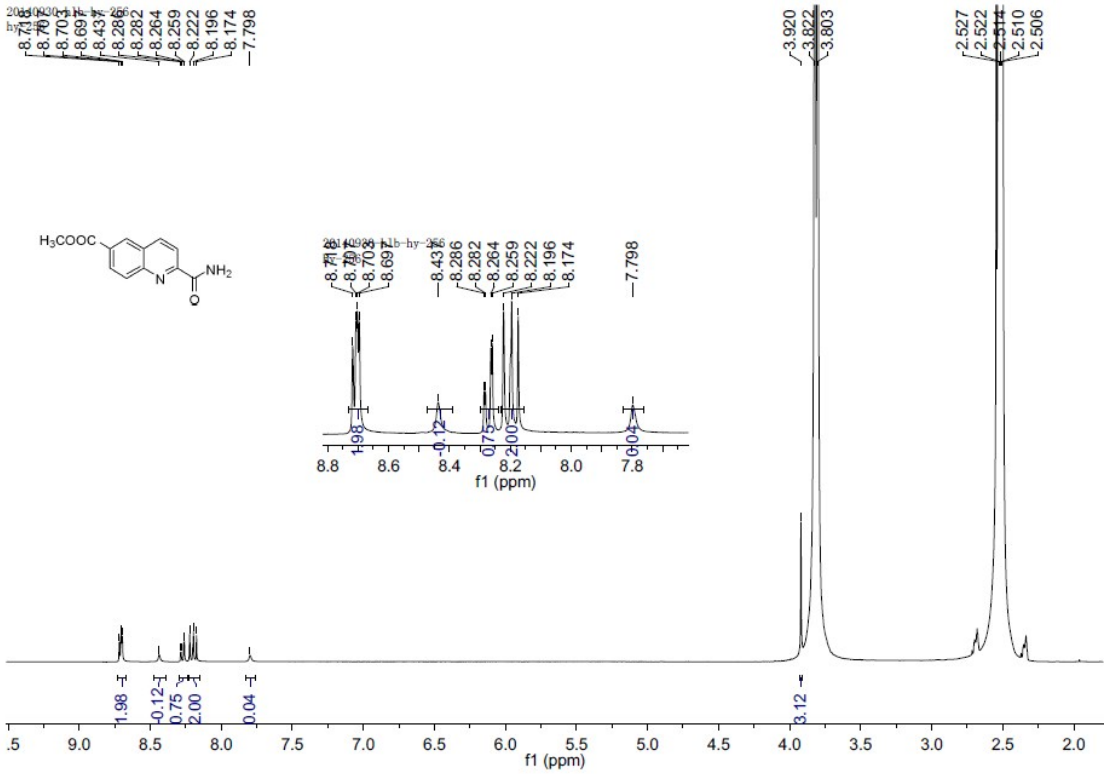
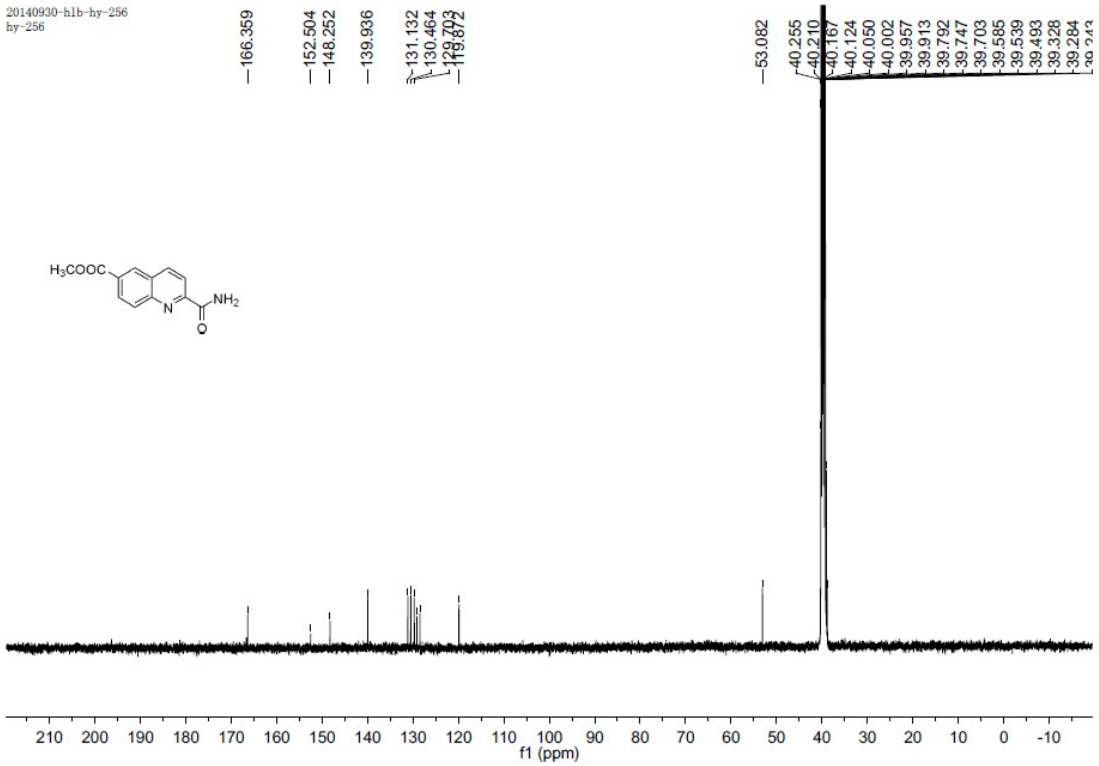
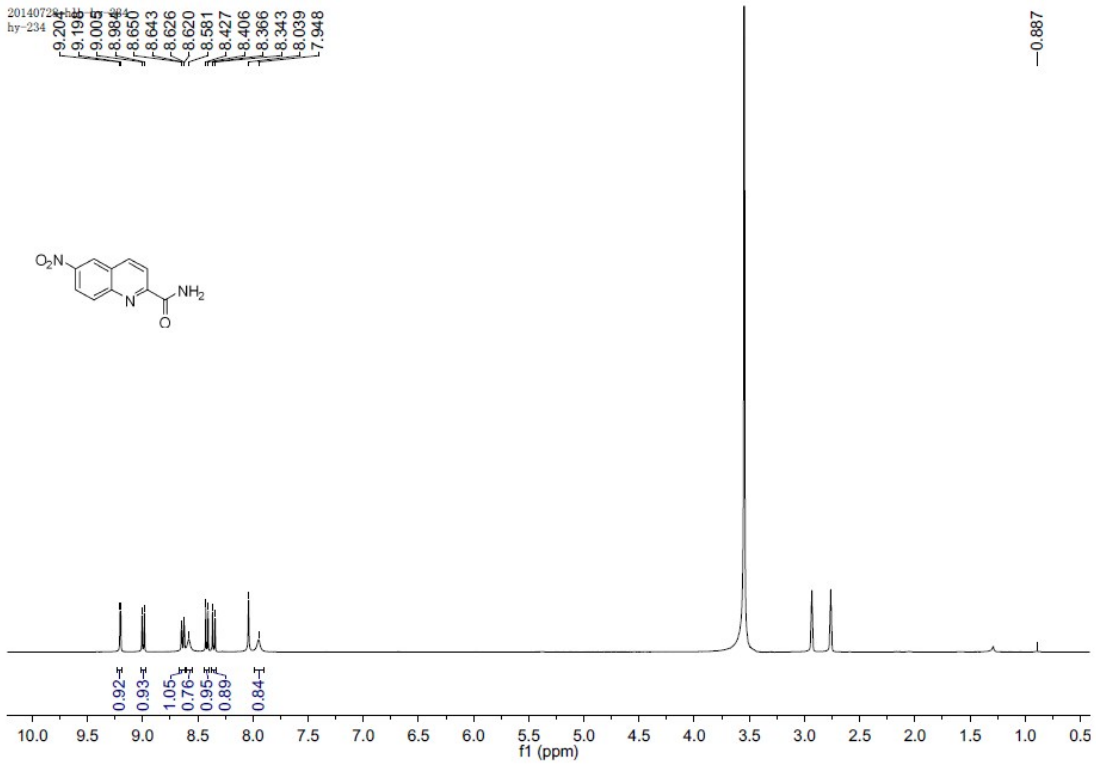
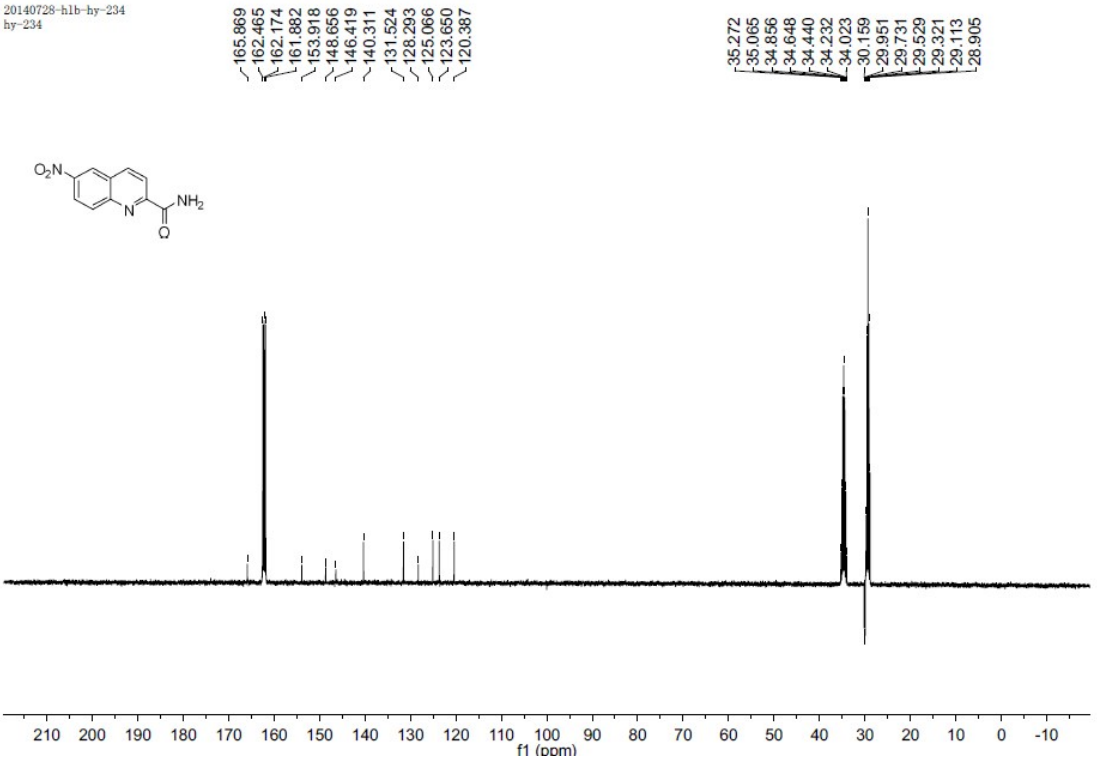
8.406
8.315
8.207
8.186
8.067
8.045
8.000
7.995
7.978
7.972
7.831
2.522
0.000
Br
N
NH2
O
1.04
1.00
0.84
1.03
0.97
1.00
0.92
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

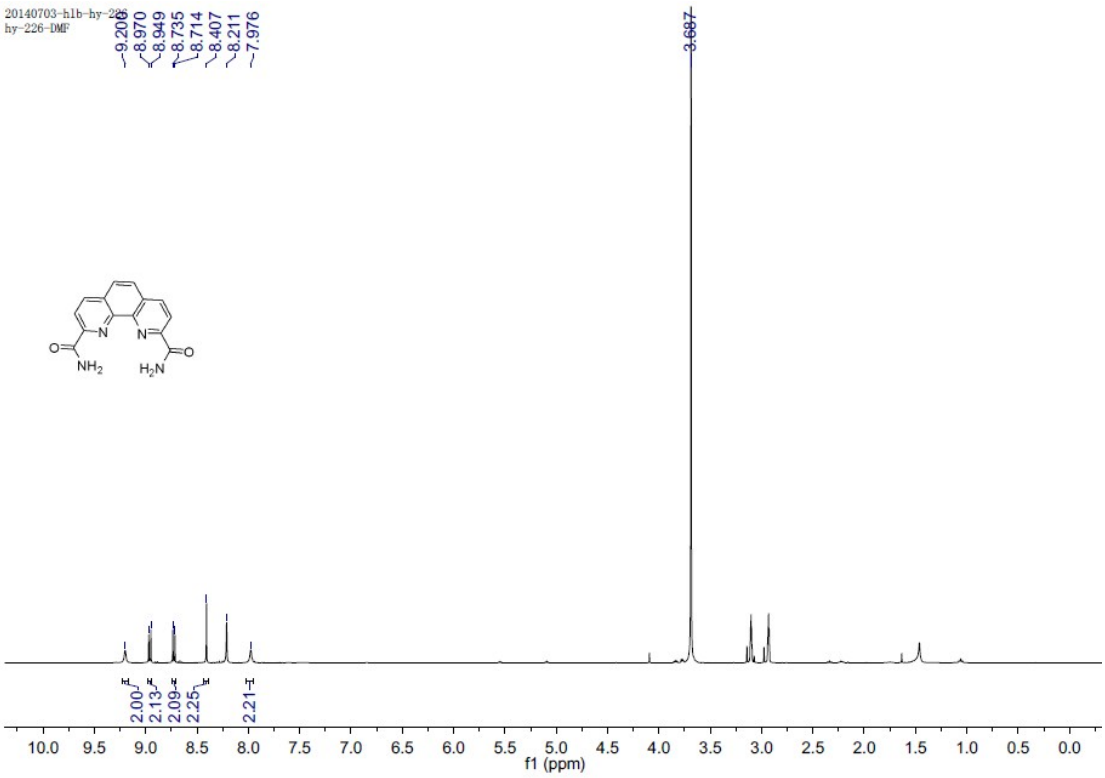

20140610-hlb-hy-202-1
hy-202-1
166.379
151.394
145.141
137.477
133.995
131.871
130.543
130.433
121.609
120.041
40.609
40.402
40.193
39.984
39.776
39.567
39.360
Br
N
NH2
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

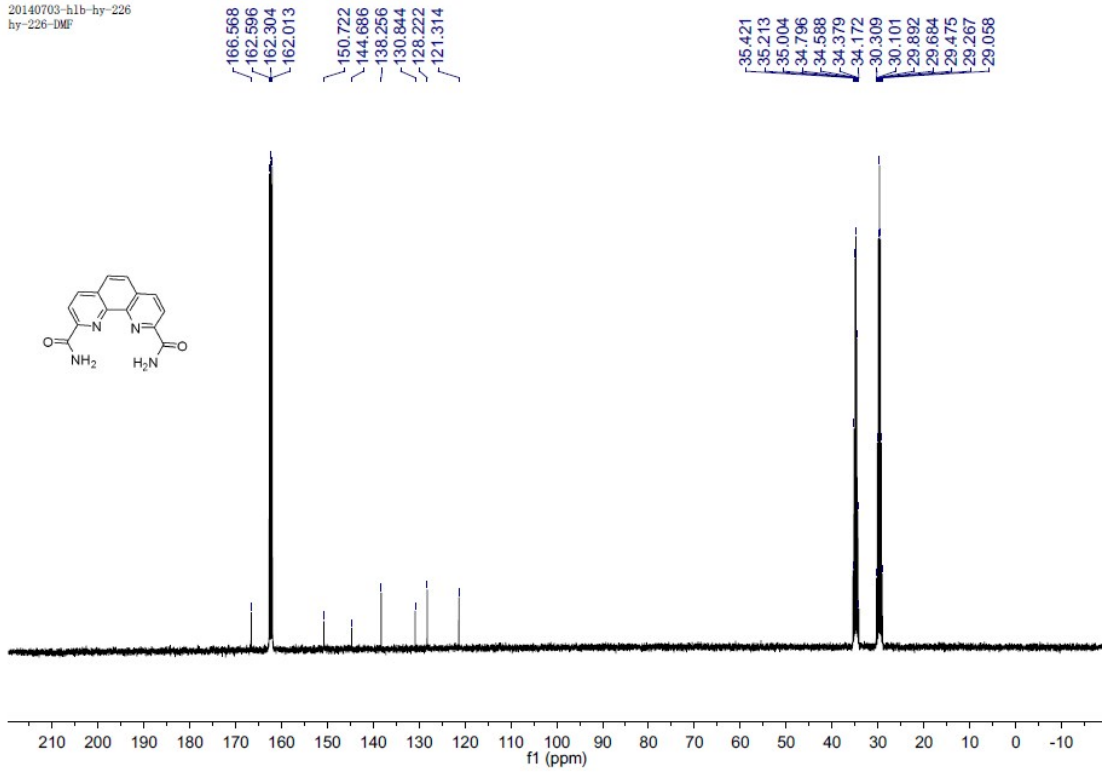

8.711
8.707
8.703
8.699
8.437
8.286
8.282
8.264
8.259
8.222
8.196
8.174
7.798
3.920
3.822
3.803
2.527
2.522
2.514
2.510
2.506
H3COOC
NH2
O
N
1.98
0.12
0.75
2.00
0.04
3.12
f1 (ppm)


20140930-hlb-hy-256
hy-256
166.359
152.504
148.252
139.936
131.132
130.464
129.703
119.872
53.082
40.255
40.210
40.167
40.124
40.050
40.002
39.957
39.913
39.792
39.747
39.703
39.585
39.539
39.493
39.328
39.284
H3COOC
NH2
O
N
f1 (ppm)


9.204
9.198
9.005
8.984
8.650
8.643
8.626
8.620
8.581
8.427
8.406
8.366
8.343
8.039
7.948
0.887
0.92
0.93
1.05
0.76
0.95
0.89
0.84
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
f1 (ppm)

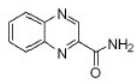

20140728-hlb-hy-234
hy-234
165.869
162.465
162.174
161.882
153.918
148.656
146.419
140.311
131.524
128.293
125.066
123.650
120.387
35.272
35.065
34.856
34.648
34.440
34.232
34.023
30.159
29.951
29.731
29.529
29.321
29.113
28.905
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

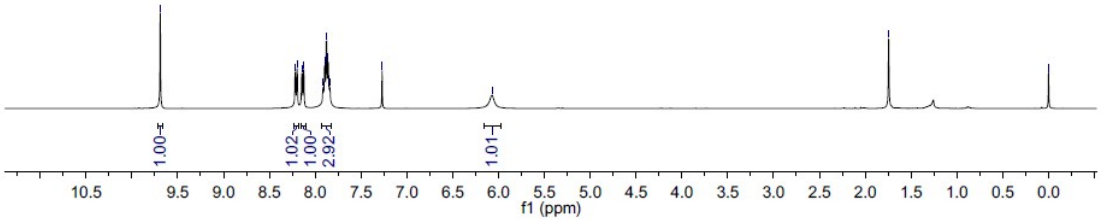

20140703-hlb-hy-226
hy-226-DMF
9.206
8.970
8.949
8.735
8.714
8.407
8.211
7.976
3.687
2.00
2.13
2.09
2.25
2.21
f1 (ppm)


20140703-hlb-hy-226
hy-226-DMF
166.568
162.596
162.304
162.013
150.722
144.686
138.256
130.844
128.222
121.314
35.421
35.213
35.004
34.796
34.588
34.379
34.172
30.309
30.101
29.892
29.684
29.475
29.267
29.058
f1 (ppm)

20140616-hlb-hy-212
hy-212

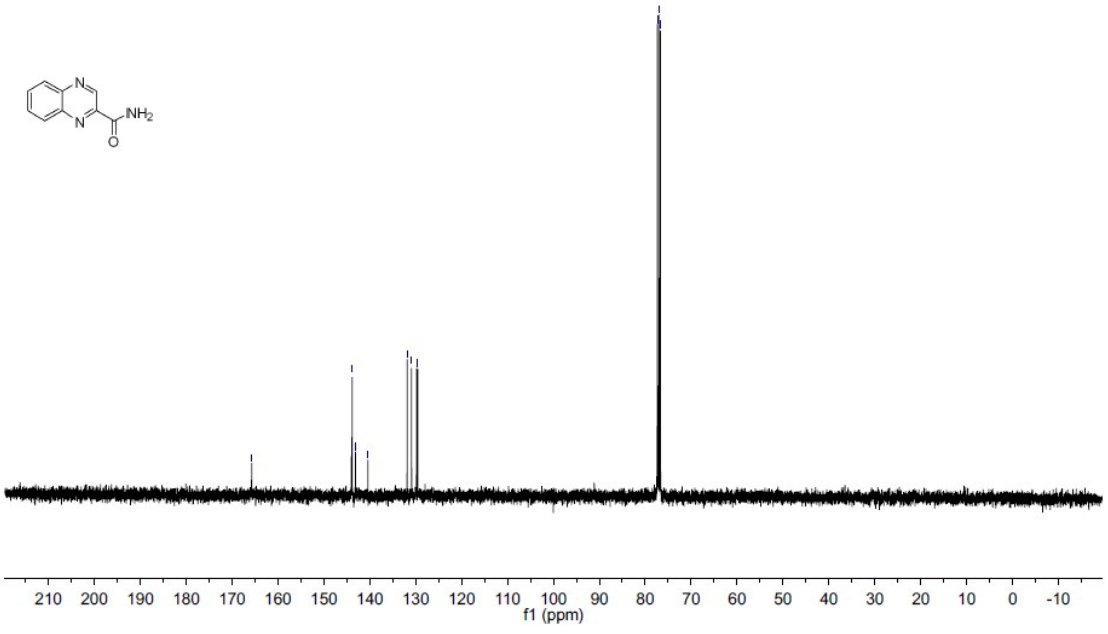
9.690
8.216
8.197
8.146
8.126
7.913
7.896
7.879
7.875
7.858
7.842
7.270
6.071
1.744
0.000

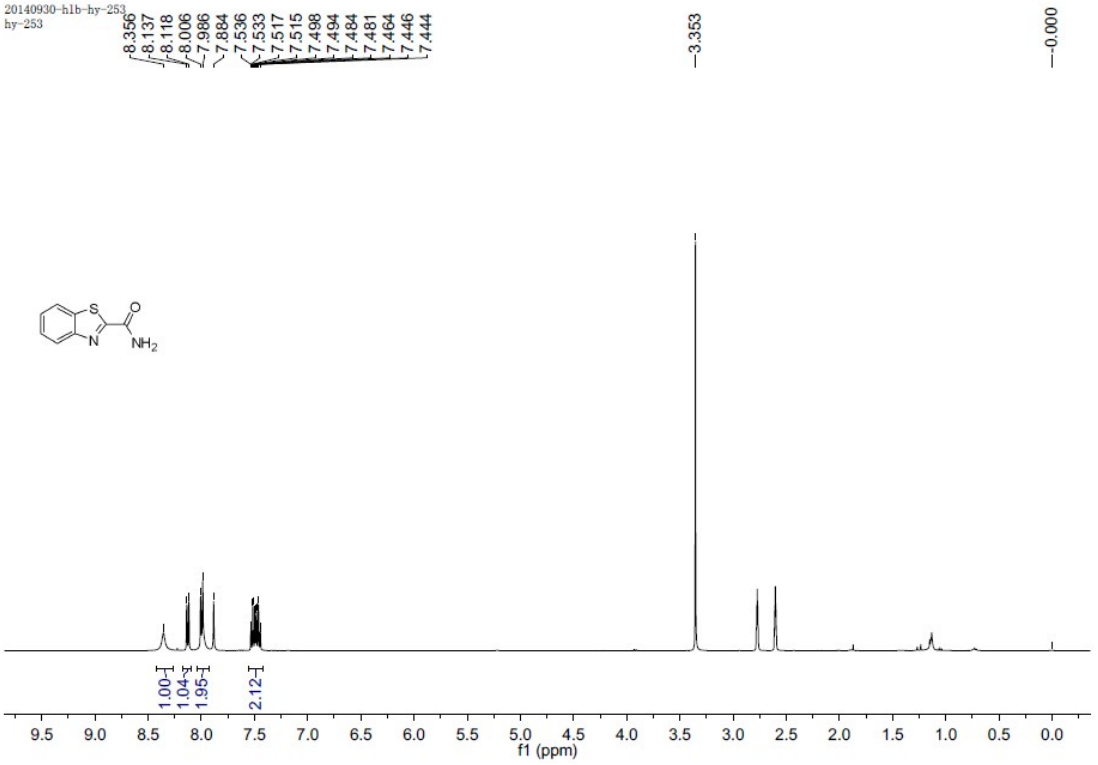


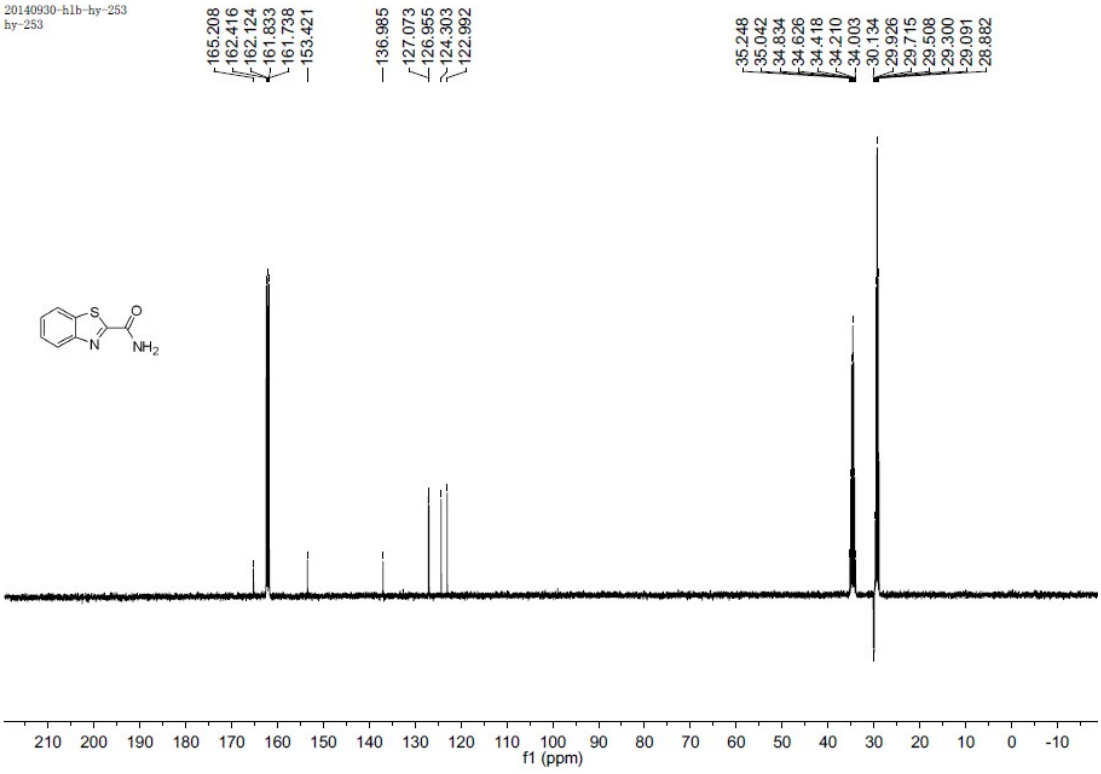


20140616-hlb-hy-212
hy-212

165.711
143.993
143.846
143.078
140.390
131.835
130.893
129.778
129.539
77.348
77.030
76.712



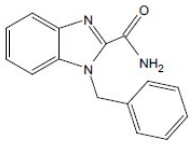

20140930-h1b-hy-253
hy-253
8.356
8.137
8.118
8.006
7.986
7.884
7.536
7.533
7.517
7.515
7.498
7.494
7.484
7.481
7.464
7.446
7.444
3.353
0.000
1.00
1.04
1.95
2.12
f1 (ppm)

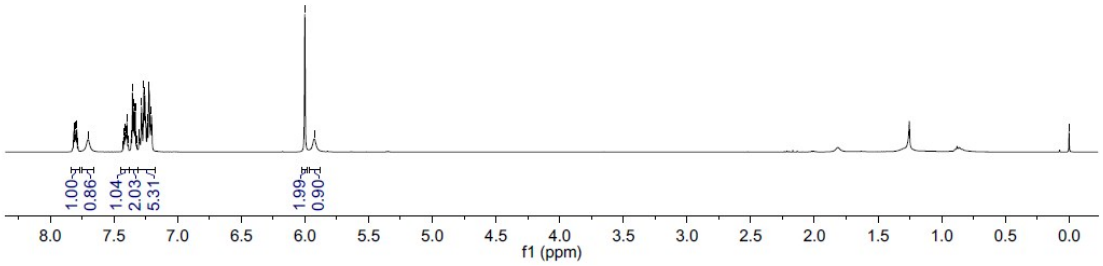

20140930-h1b-hy-253
hy-253
165.208
162.416
162.124
161.833
161.738
153.421
136.985
127.073
126.955
124.303
122.992
35.248
35.042
34.834
34.626
34.418
34.210
34.003
30.134
29.926
29.715
29.508
29.300
29.091
28.882
f1 (ppm)

20141227-hlb-hy-507
hy-507

7.814
7.799
7.791
7.398
7.354
7.347
7.339
7.331
7.283
7.264
7.256
7.237
7.225
7.222
6.005
5.926

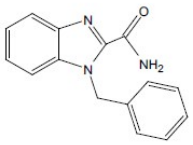
0.000

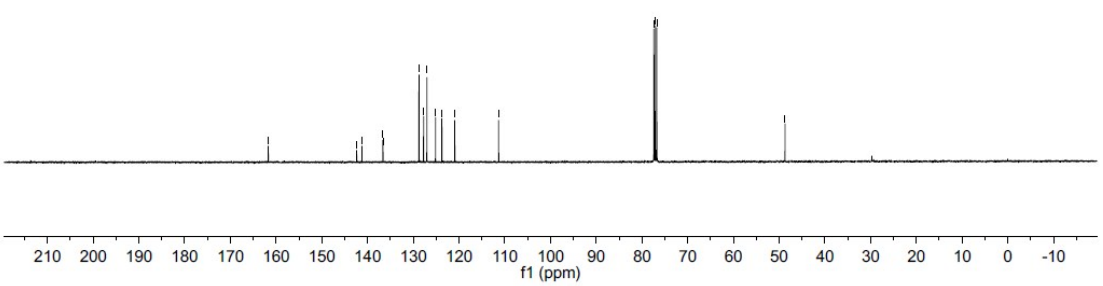


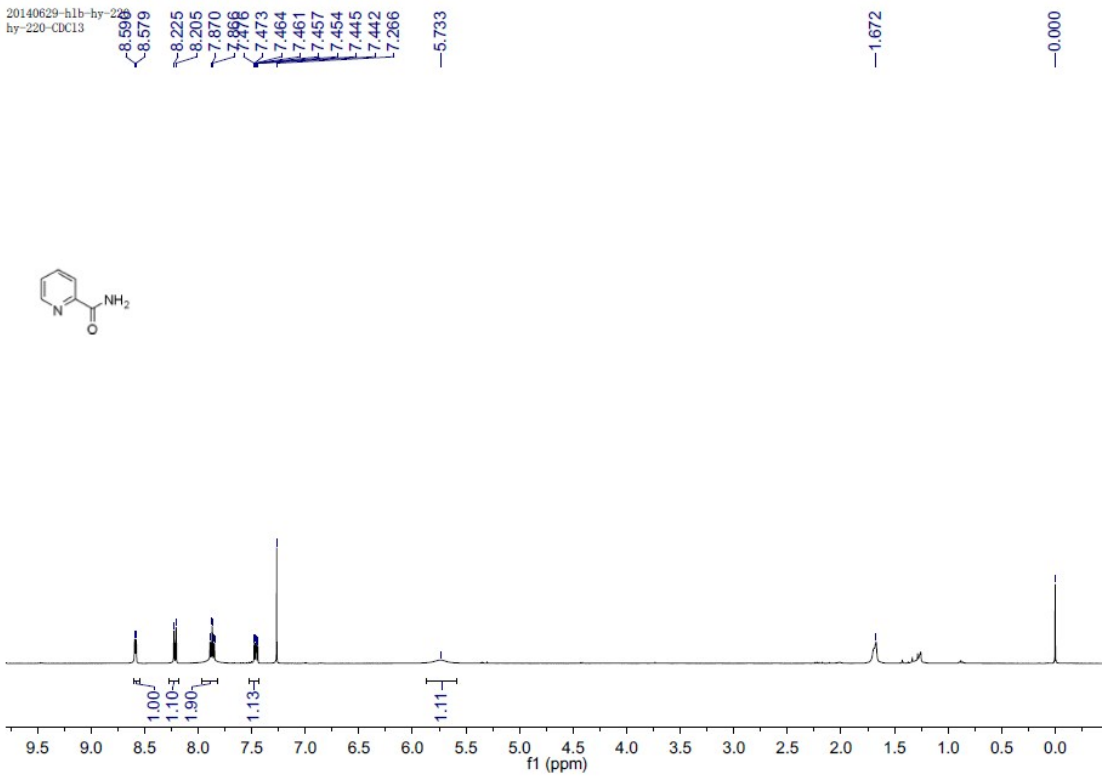


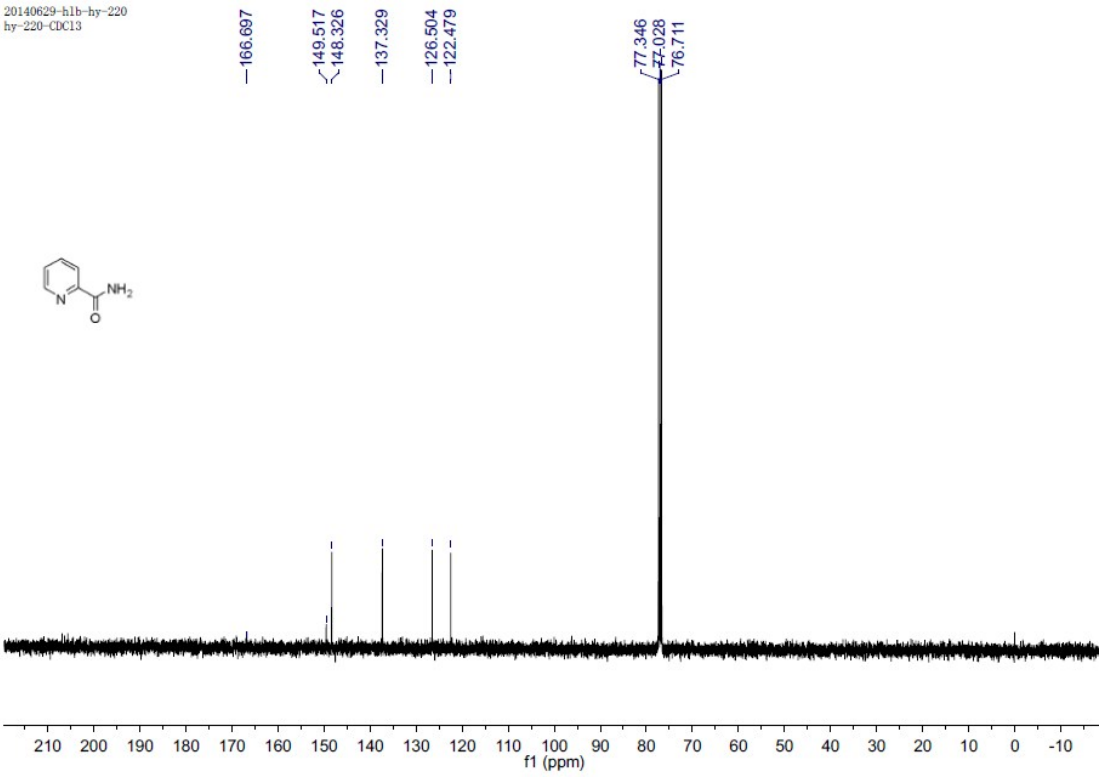
20141227-hlb-hy-507
hy-507

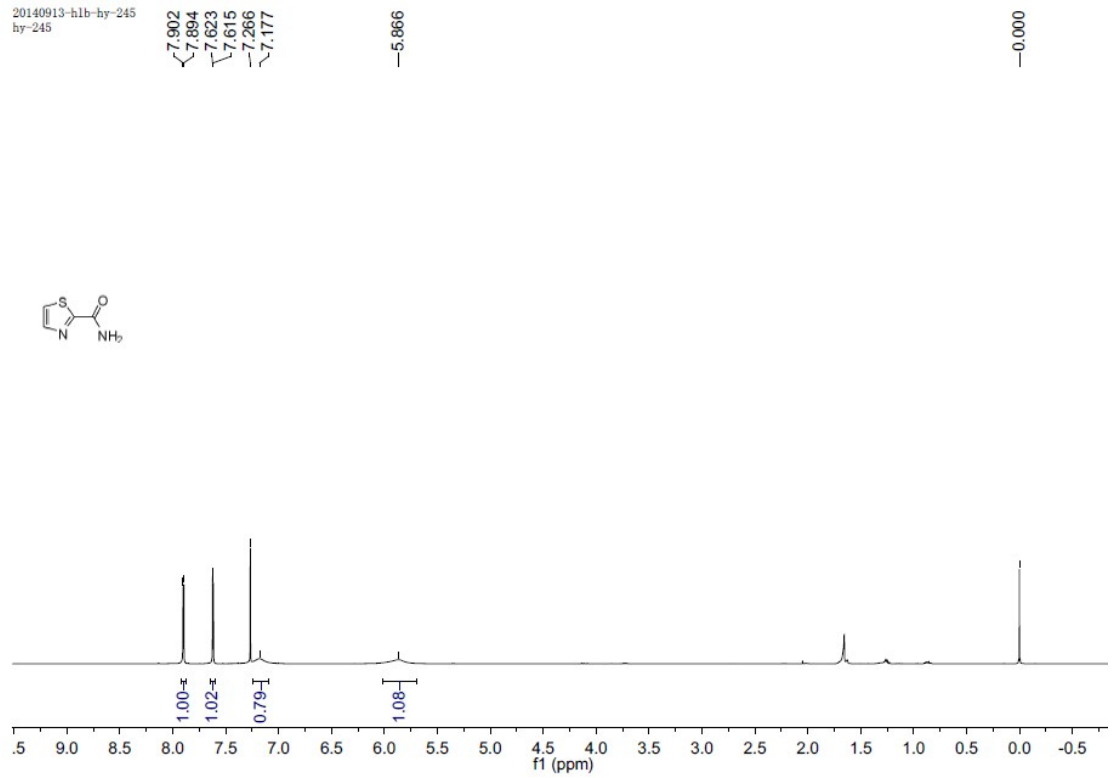
161.703
142.372
141.226
136.653
136.597
128.752
127.745
127.025
125.135
123.748
120.930
111.326
77.377
77.060
76.742
48.719

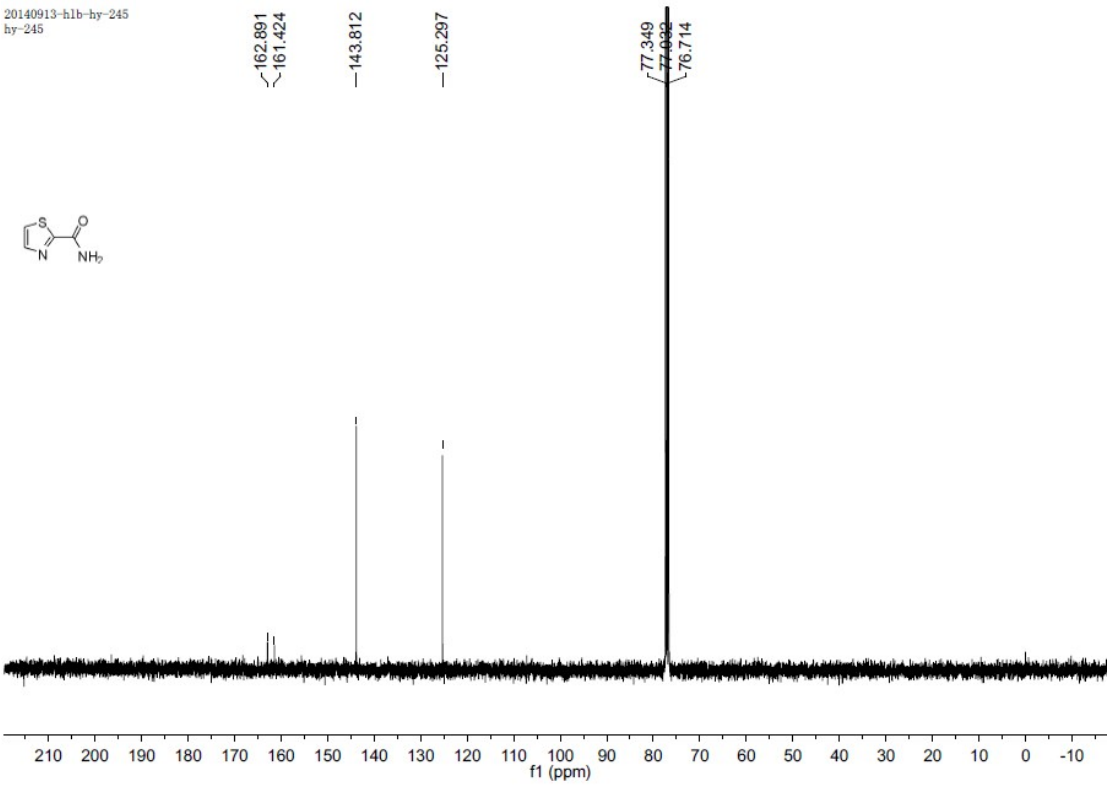





20140629-hlb-hy-220
hy-220-CDCl3
8.596
8.579
8.225
8.205
7.870
7.473
7.464
7.461
7.457
7.454
7.445
7.442
7.266
5.733
1.672
0.000
NH2
O
1.00
1.10
1.90
1.13
1.11
f1 (ppm)


20140629-hlb-hy-220
hy-220-CDCl3
166.697
149.517
148.326
137.329
126.504
122.479
77.346
77.028
76.711
NH2
O
f1 (ppm)

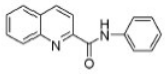

20140913-hlb-hy-245
hy-245
7.902
7.894
7.623
7.615
7.266
7.177
5.866
0.000
1.00
1.02
0.79
1.08
f1 (ppm)

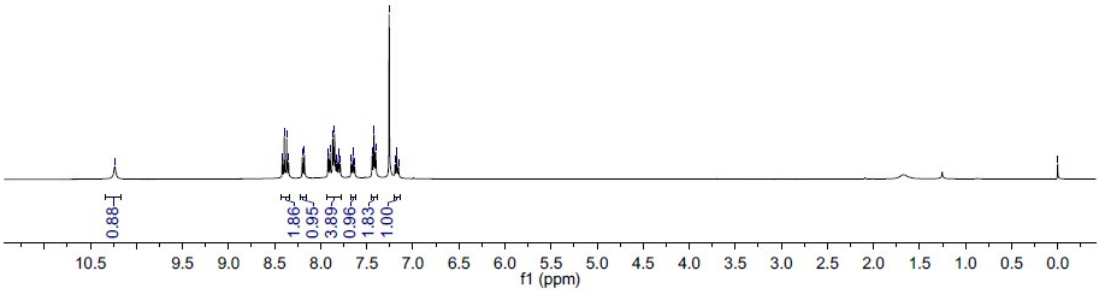

20140913-hlb-hy-245
hy-245
162.891
161.424
143.812
125.297
77.349
77.032
76.714
f1 (ppm)

20140703-hlb-hy-223
hy-223-CDCl3

10.236
8.416
8.395
8.370
8.349
8.199
8.178
7.920
7.899
7.869
7.849
7.827
7.807
7.788
7.669
7.649
7.631
7.441
7.422
7.402
7.256
7.192
7.173
7.155

0.000

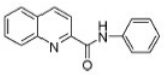


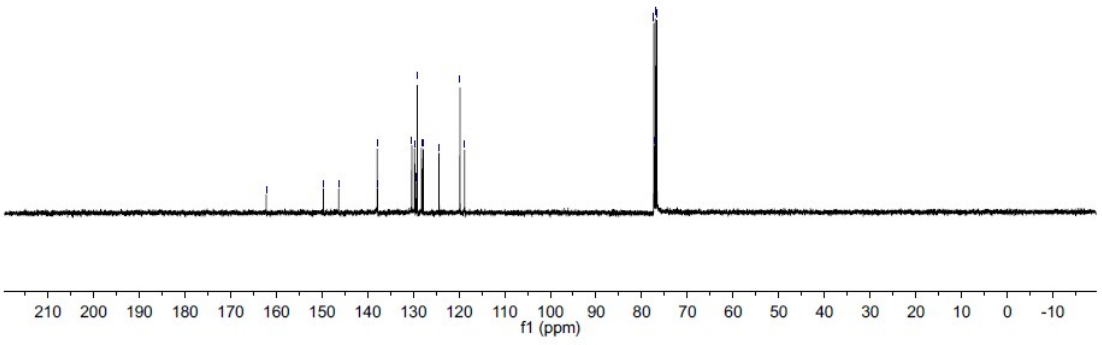


20140703-hlb-hy-223，2-甲基喹啉+苯胺产物，氢谱和碳谱都用，CDCl3
HY-223-cdcl3

162.179
149.677
146.313
137.867
137.801
130.332
129.682
129.442
129.119
128.167
127.829
124.364
119.787
118.766

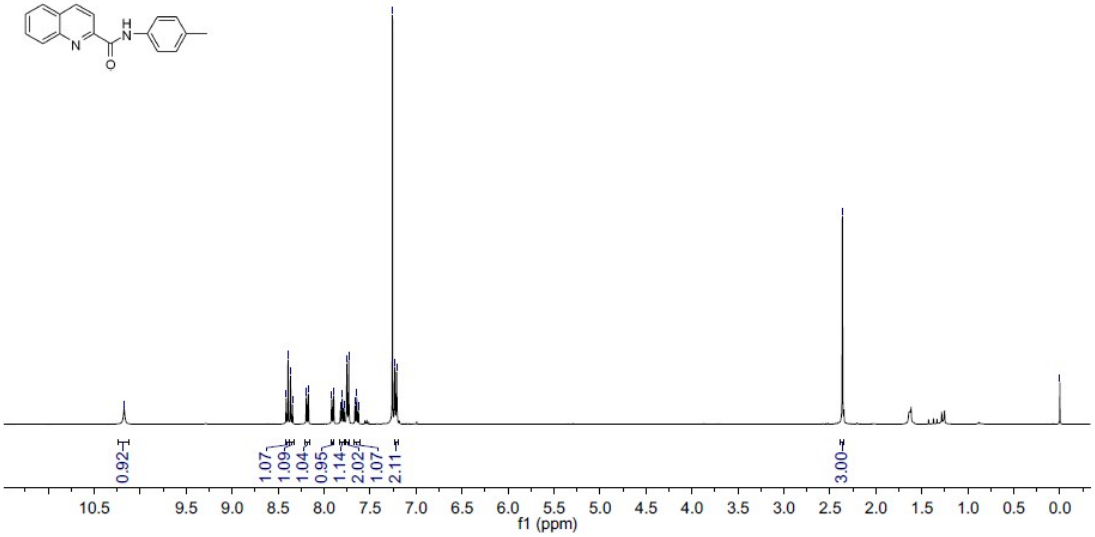
77.350
77.235
77.032
76.714





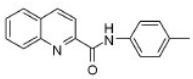
20140630-hlb-hy-222
hy-222-CDCl3

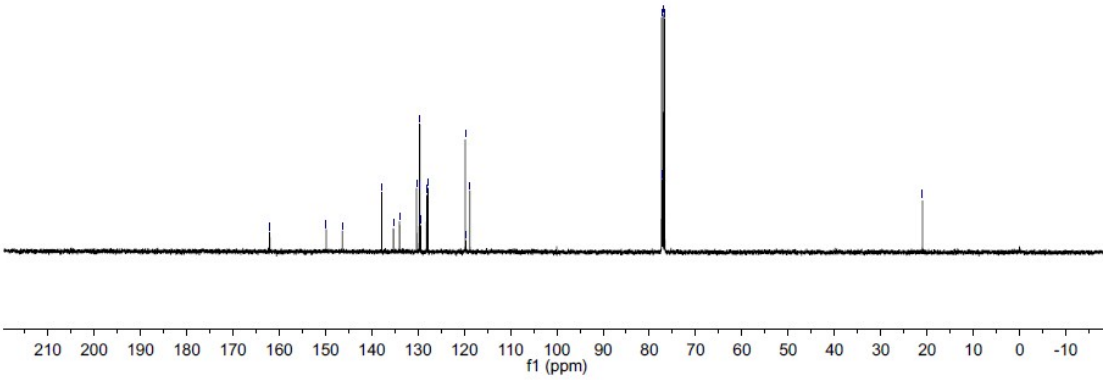
10.175
8.413
8.392
8.366
8.345
8.193
8.172
7.917
7.897
7.824
7.803
7.783
7.753
7.732
7.665
7.645
7.627
7.257
7.232
7.211
2.364
0.000

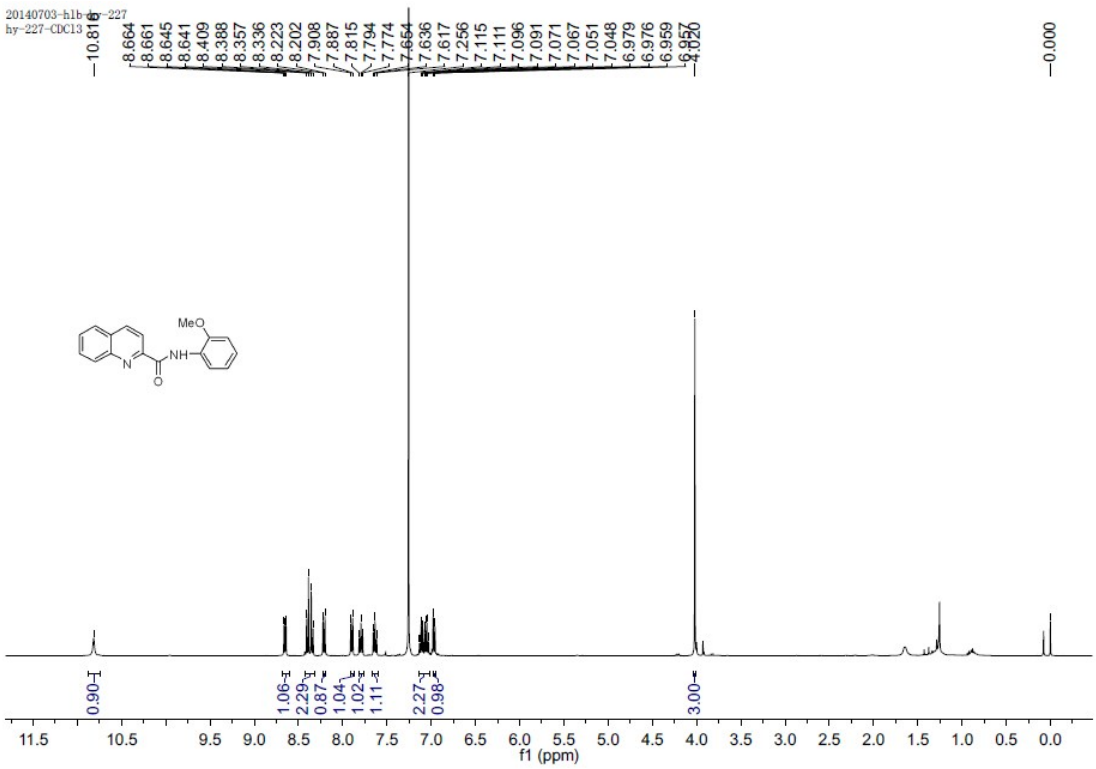


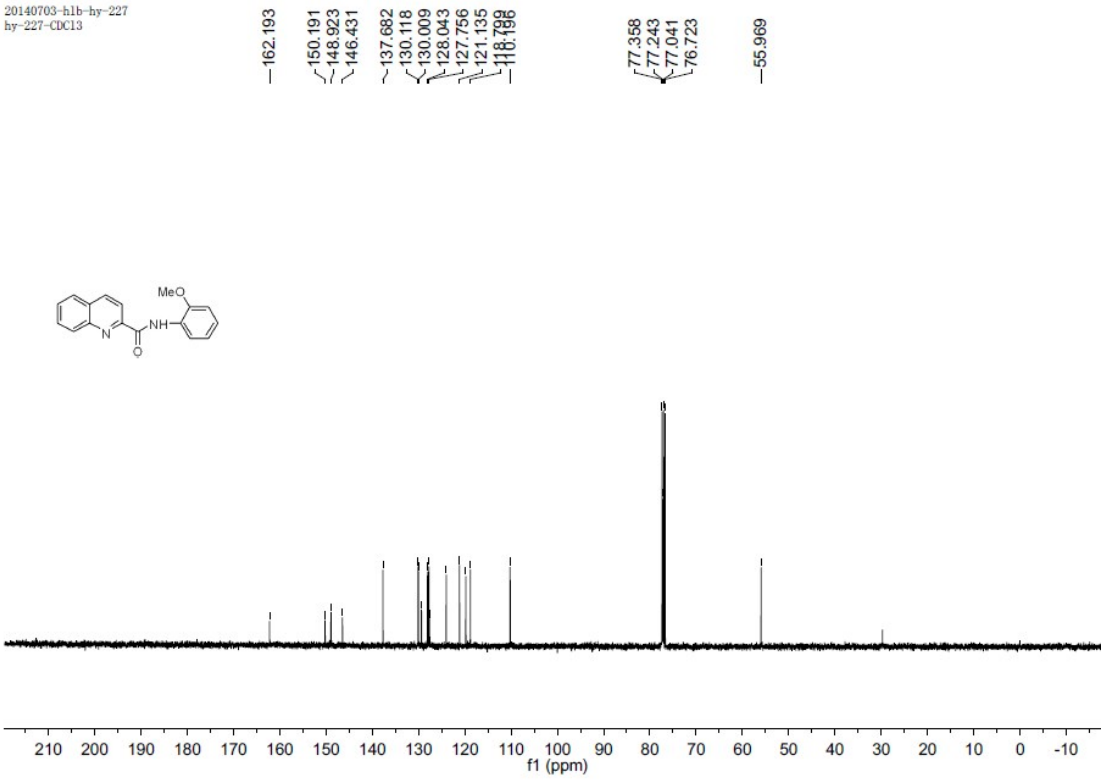
20140630-hlb-hy-222
hy-222-CDCl3

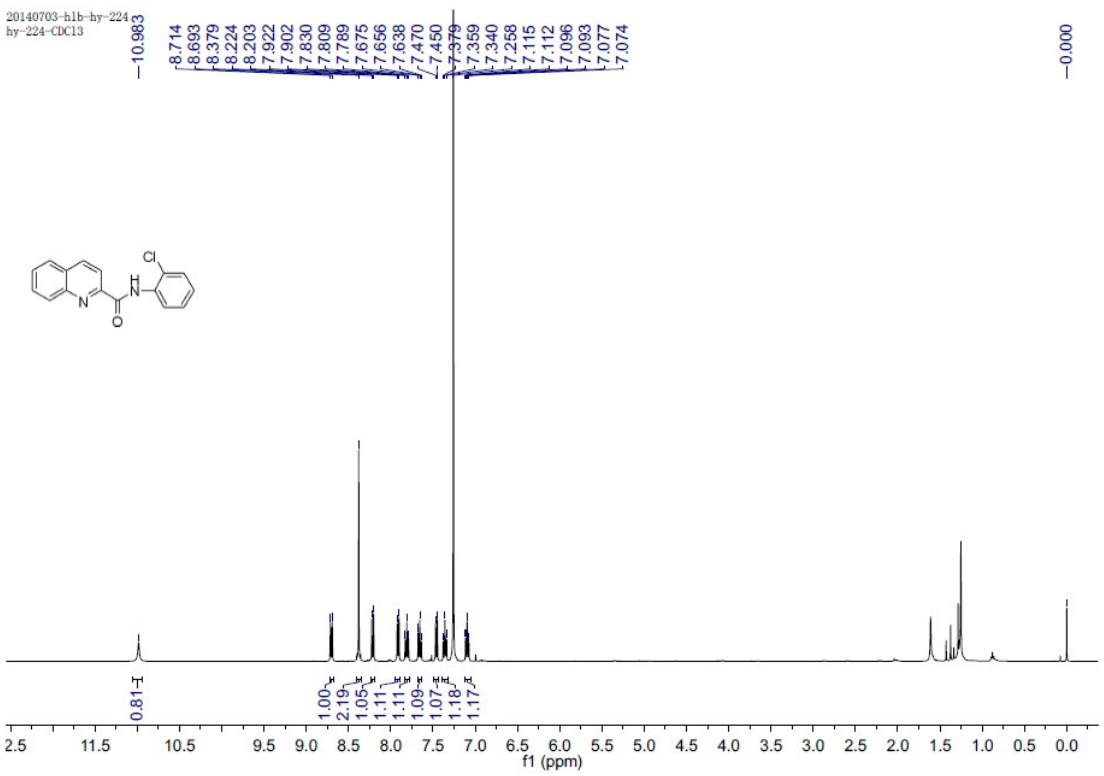
162.041
149.826
146.334
137.828
135.293
133.975
130.297
129.679
129.626
129.412
128.103
127.832
119.769
119.679
118.780
77.351
77.237
77.034
76.716
20.969

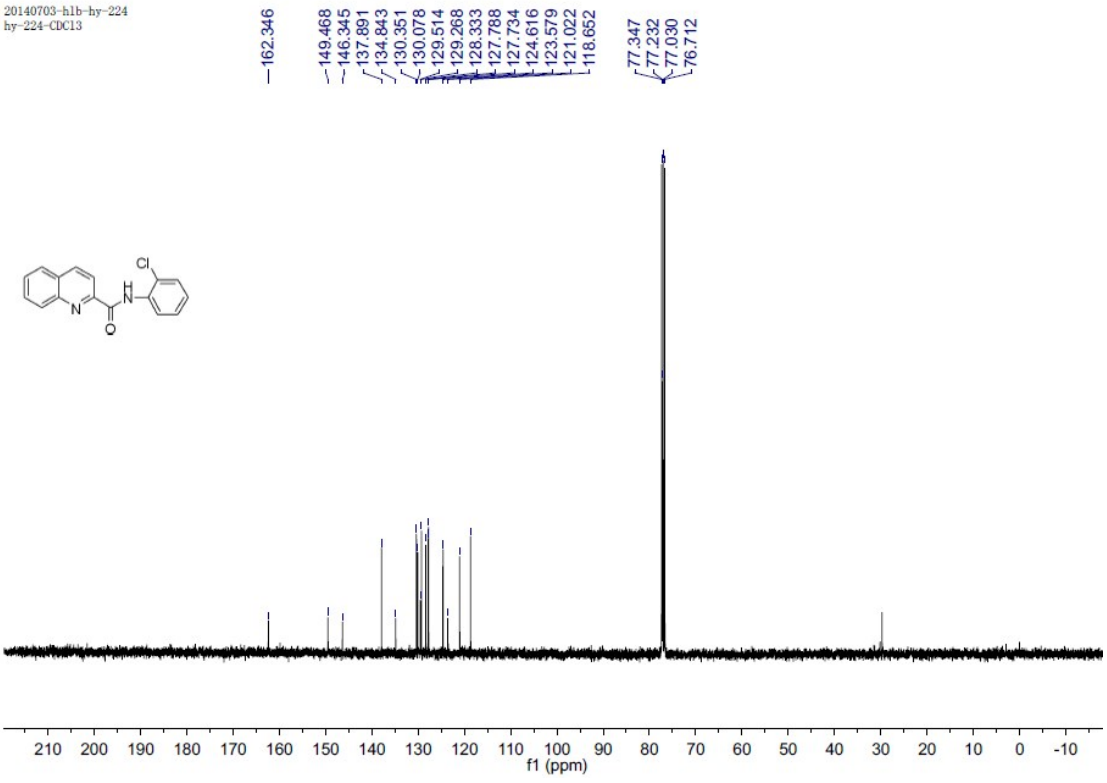


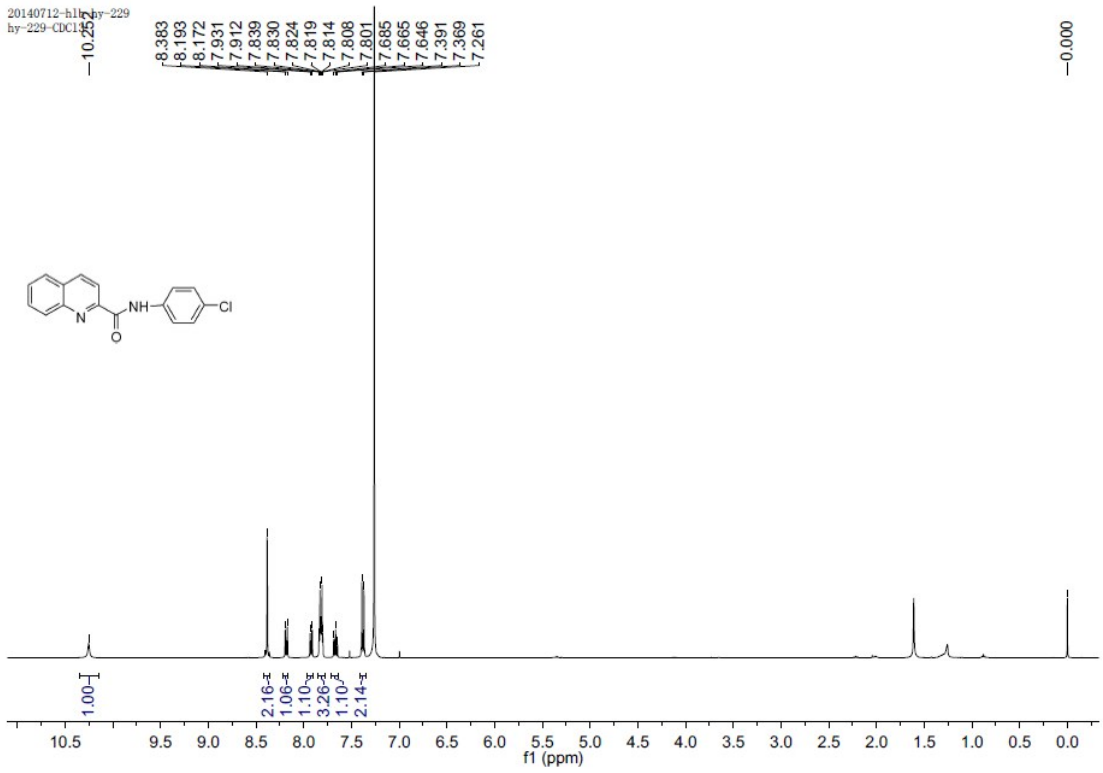


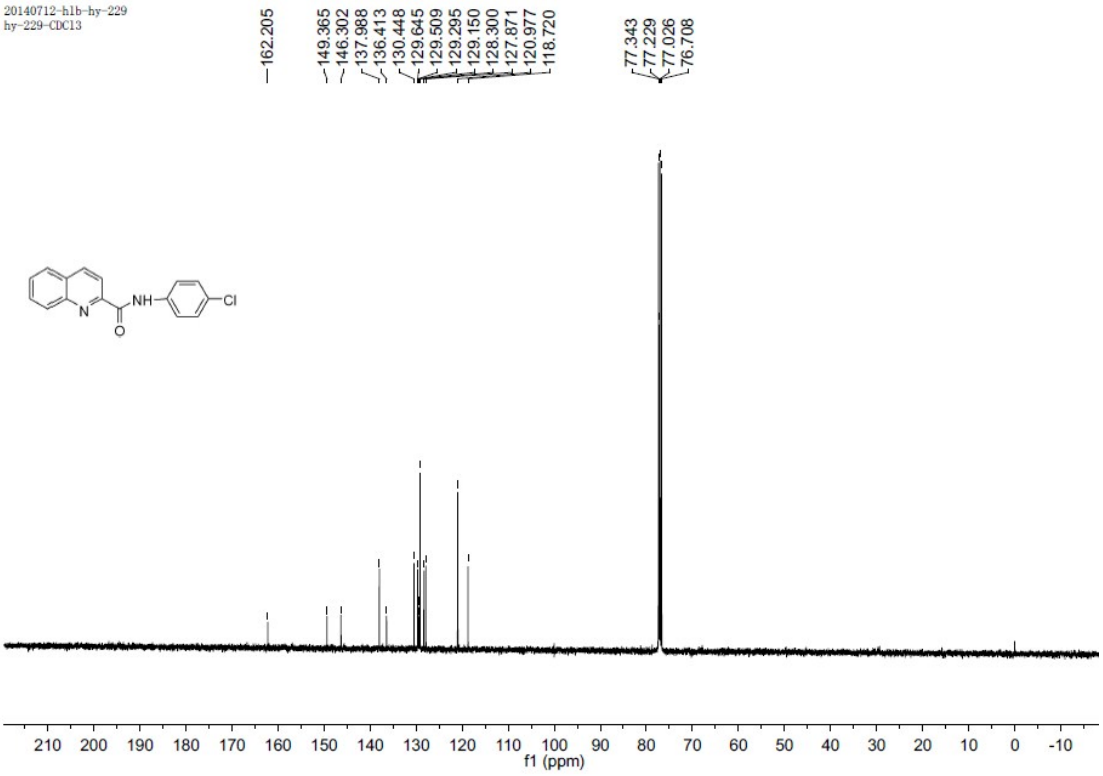

20140703-h1b-hy-227
hy-227-CDCl3
10.816
8.664
8.661
8.645
8.641
8.409
8.388
8.357
8.336
8.223
8.202
7.908
7.887
7.815
7.794
7.774
7.664
7.636
7.617
7.256
7.115
7.111
7.096
7.091
7.071
7.067
7.051
7.048
6.979
6.976
6.959
6.957
4.020
0.000
MeO
NH
O
N
0.90
1.06
2.29
0.87
1.04
1.02
1.11
2.27
0.98
3.00
11.5
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)

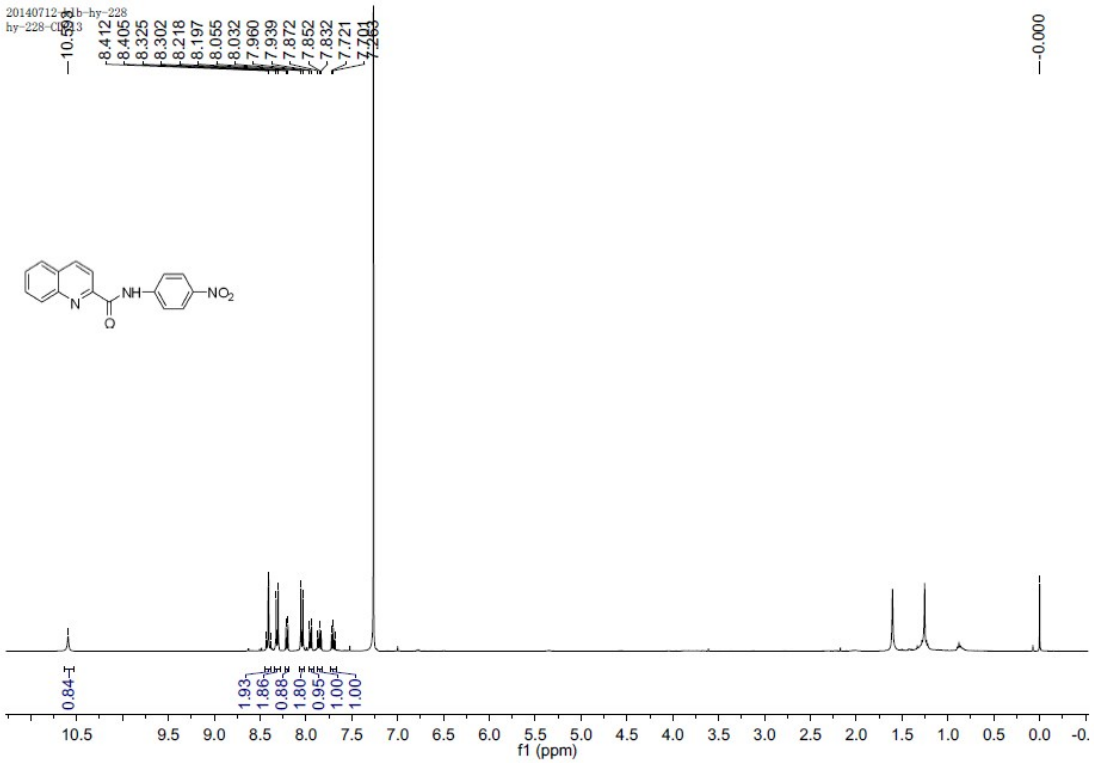

20140703-h1b-hy-227
hy-227-CDCl3
162.193
150.191
148.923
146.431
137.682
130.118
130.009
128.043
127.756
121.135
118.799
118.196
77.358
77.243
77.041
76.723
55.969
MeO
NH
O
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

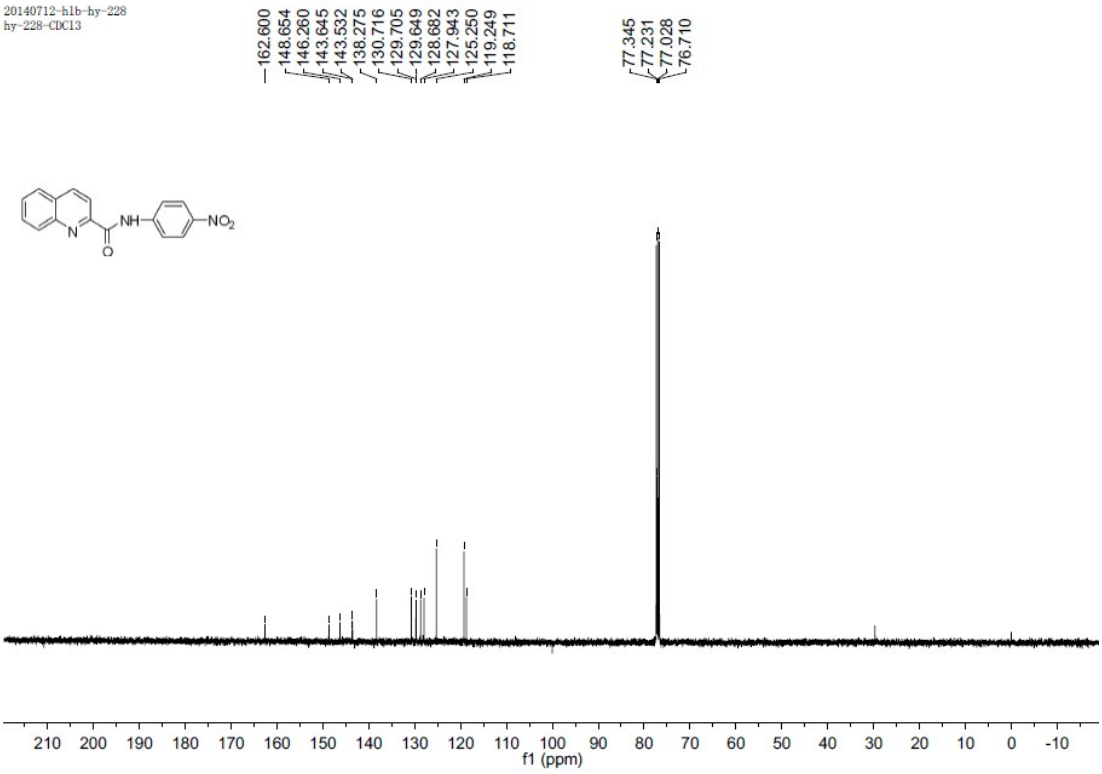

20140703-hlb-hy-224
hy-224-CDCl3
10.983
8.714
8.693
8.379
8.224
8.203
7.922
7.902
7.830
7.809
7.789
7.675
7.656
7.638
7.470
7.450
7.379
7.359
7.340
7.258
7.115
7.112
7.096
7.093
7.077
7.074
0.000
Cl
N
H
O
0.81
1.00
2.19
1.05
1.11
1.11
1.09
1.07
1.18
1.17
2.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 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)

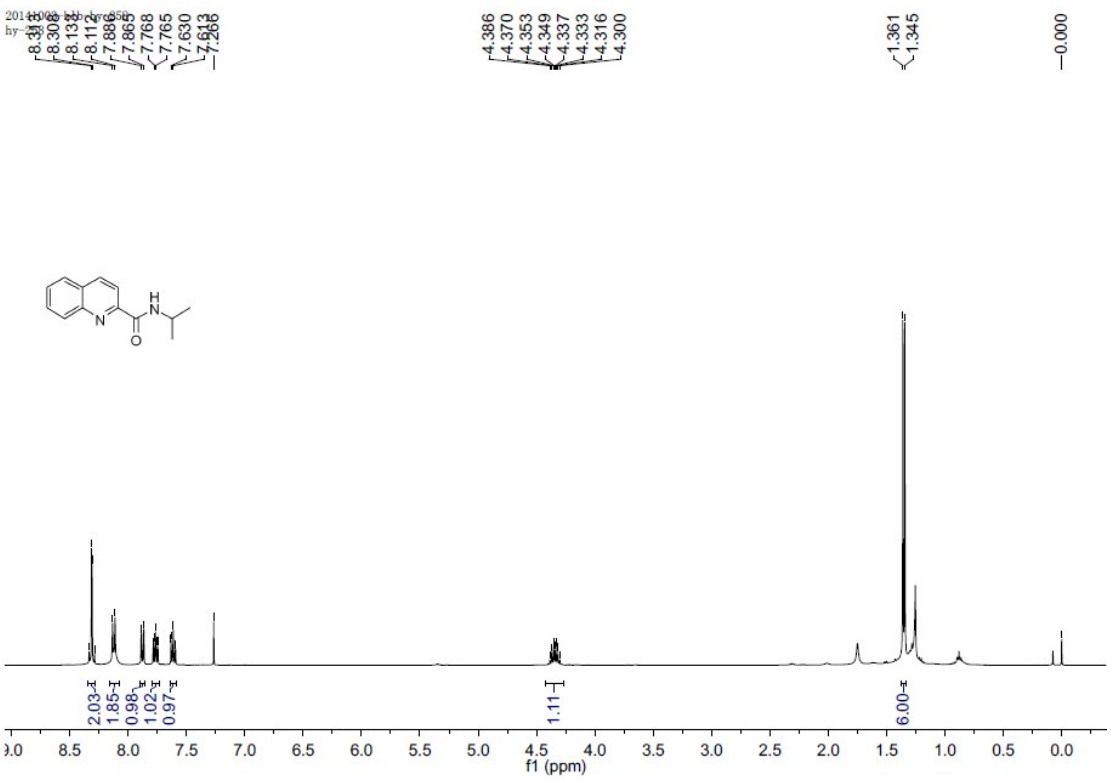

20140703-hlb-hy-224
hy-224-CDCl3
162.346
149.468
146.345
137.891
134.843
130.351
130.078
129.514
129.268
128.333
127.788
127.734
124.616
123.579
121.022
118.652
77.347
77.232
77.030
76.712
Cl
N
H
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

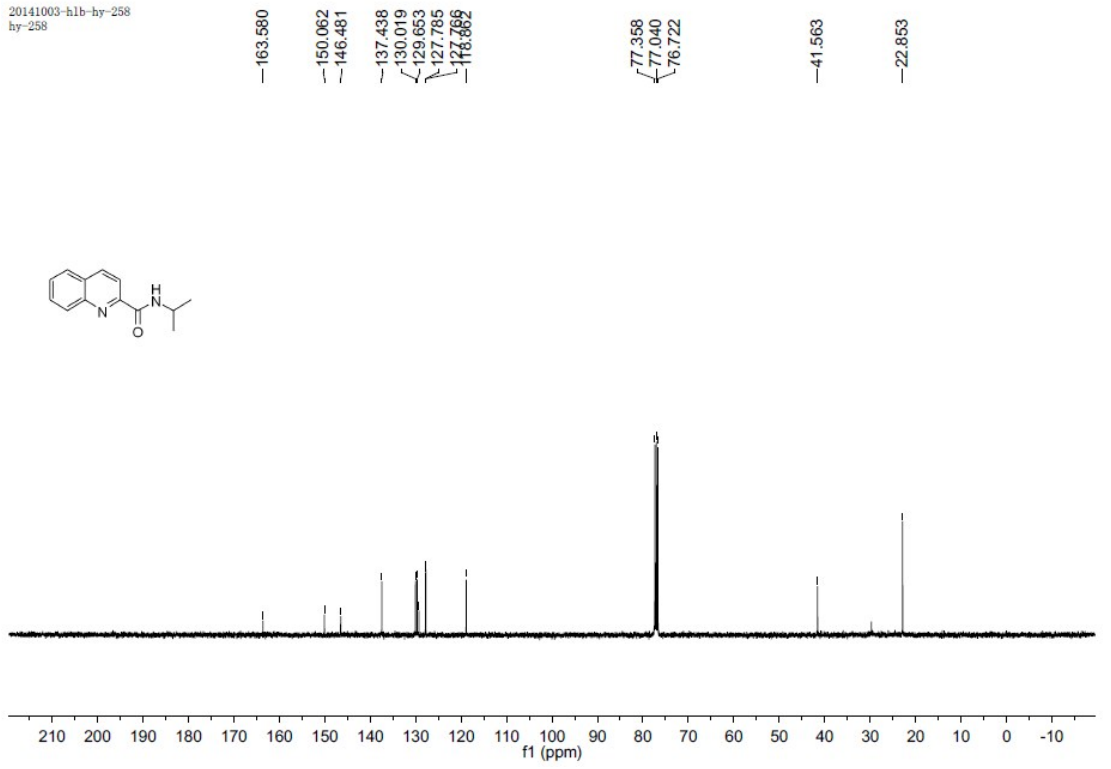

20140712-hlb-hy-229
hy-229-CDCl3
10.252
8.383
8.193
8.172
7.931
7.912
7.839
7.830
7.824
7.819
7.814
7.808
7.801
7.685
7.665
7.646
7.391
7.369
7.261
0.000
1.00
2.16
1.06
1.10
3.26
1.10
2.14
f1 (ppm)

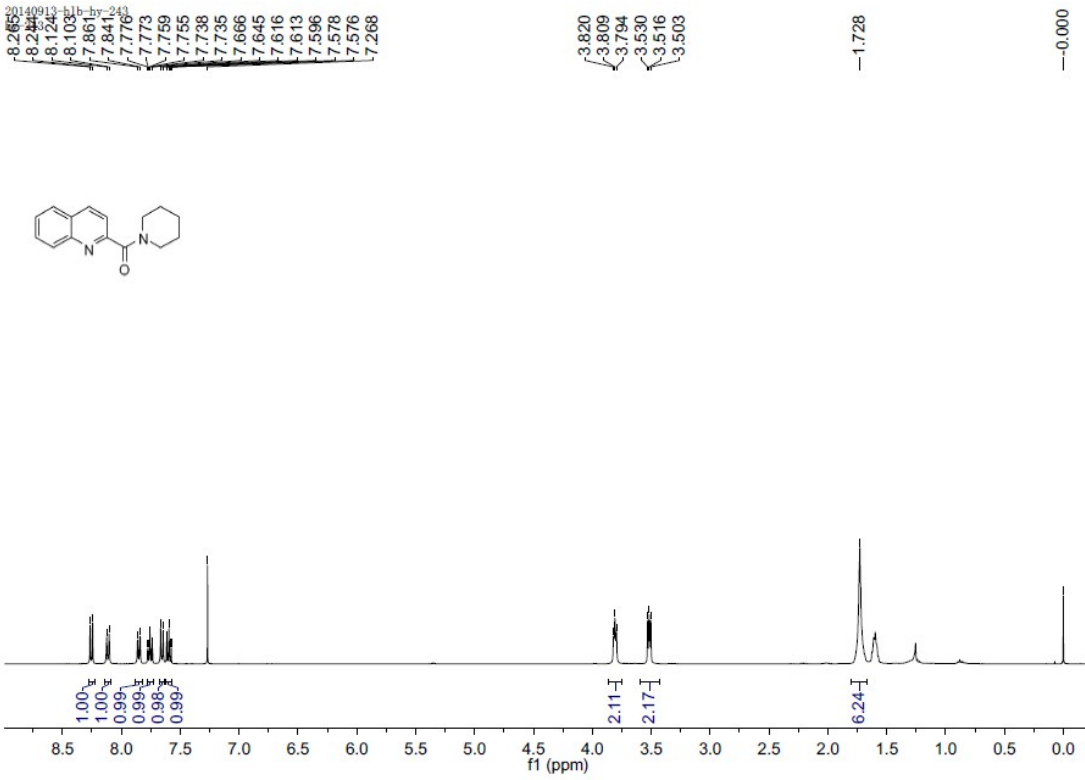

20140712-hlb-hy-229
hy-229-CDCl3
162.205
149.365
146.302
137.988
136.413
130.448
129.645
129.509
129.295
129.150
128.300
127.871
120.977
118.720
77.343
77.229
77.026
76.708
f1 (ppm)

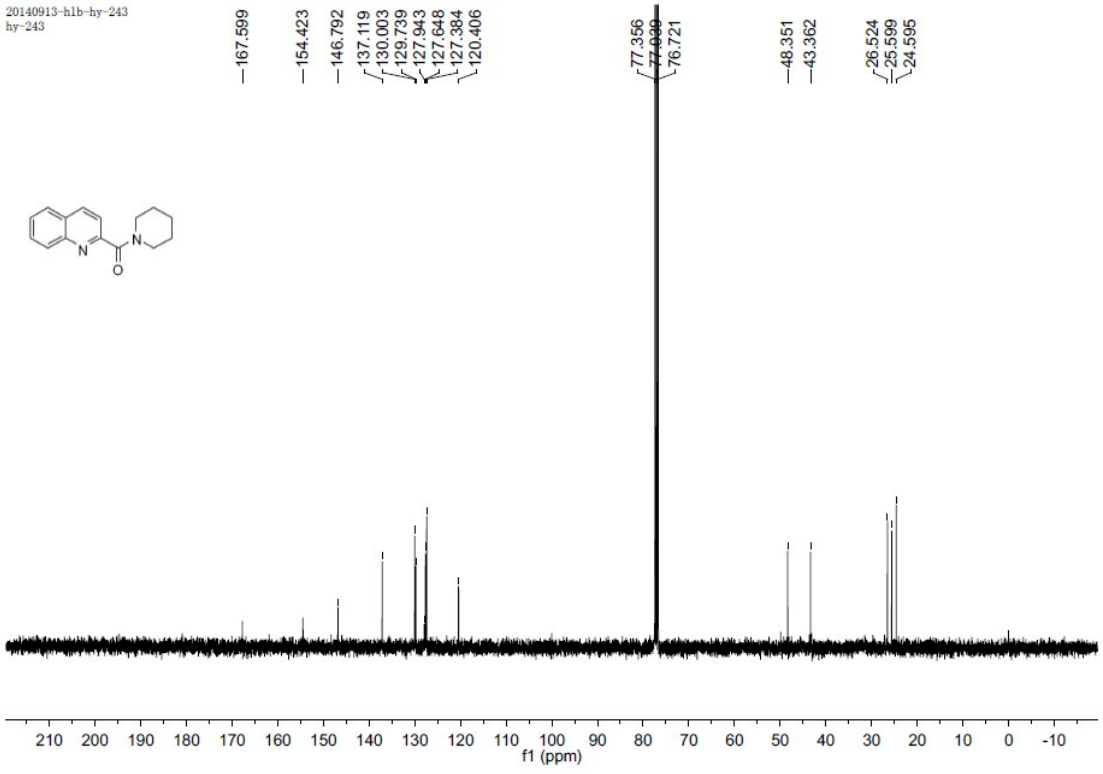

20140712-h1b-hy-228
hy-228-CDCl3
10.593
8.412
8.405
8.325
8.302
8.218
8.197
8.055
8.032
7.960
7.939
7.872
7.852
7.832
7.721
7.701
7.263
0.000
NH
NO2
O
0.84
1.93
1.86
0.88
1.80
0.95
1.00
1.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


20140712-h1b-hy-228
hy-228-CDCl3
162.600
148.654
146.260
143.645
143.532
138.275
130.716
129.705
129.649
128.682
127.943
125.250
119.249
118.711
77.345
77.231
77.028
76.710
NH
NO2
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.386
8.370
8.353
8.349
8.337
8.333
8.316
8.300
7.768
7.765
7.630
1.361
1.345
0.000
2.03
1.85
0.98
1.02
0.97
1.11
6.00
f1 (ppm)


20141003-hlb-hy-258
hy-258
163.580
150.062
146.481
137.438
130.019
129.653
127.785
77.358
77.040
76.722
41.563
22.853
f1 (ppm)

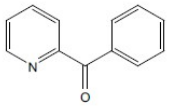

20140913-hlb-hy-243
8.265
8.244
8.241
8.124
8.103
7.861
7.841
7.776
7.773
7.759
7.755
7.738
7.735
7.666
7.645
7.616
7.613
7.596
7.578
7.576
7.268
3.820
3.809
3.794
3.530
3.516
3.503
1.728
0.000
1.00
1.00
0.99
0.99
0.98
0.99
2.11
2.17
6.24
f1 (ppm)

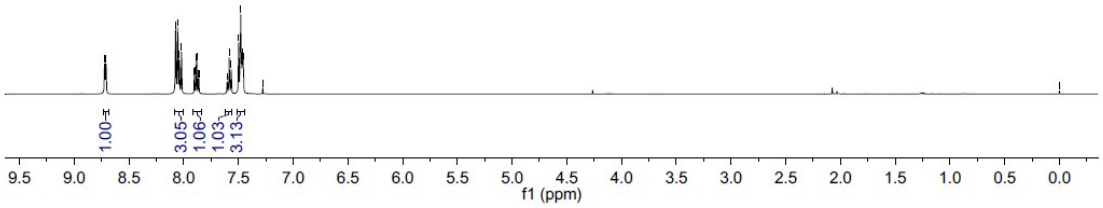

20140913-hlb-hy-243
hy-243
167.599
154.423
146.792
137.119
130.003
129.739
127.943
127.648
127.384
120.406
77.356
77.039
76.721
48.351
43.362
26.524
25.599
24.595
f1 (ppm)

20141223-hlb-hy-505
hy-505

8.721
8.710
8.074
8.056
8.040
8.020
7.903
7.899
7.884
7.880
7.865
7.861
7.603
7.584
7.566
7.498
7.479
7.465
7.460
7.457
7.278

0.000





20141223-hlb-hy-505
hy-505

193.915
155.048
148.573
137.100
136.257
132.959
130.994
128.188
126.220
124.642
77.486
77.168
76.849