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SUPPORTING INFORMATION

Supporting Information
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Iron-catalyzed oxidative arylmethylation of activated alkenes using a peroxide as the methyl source

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Supporting Information

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(A) Typical Experimental Procedure

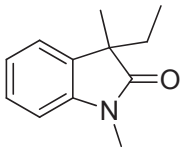
(a) Synthesis of Substrates 1:

Substrates **1** were prepared according to the known procedures.[S1].

(b) Typical Experimental Procedure for the Iron-catalyzed Oxidative Arylmethylation of Activated Alkenes Using Peroxides as the Methyl Resource:

To a Schlenk tube were added alkens **1** (0.3 mmol), $Fe(OAc)_2$ (5 mol%), DABCO (10 mol%), (peroxybis(propane-2,2-diyl))dibenzene (DCP, 2 equiv), and DMSO (2 mL). Then the tube was charged with argon, and was stirred at 120 ^oC (oil bath temperature) for the indicated time until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was cooled to room temperature, diluted in ethyl acetate, and washed with brine. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate = 20:1) to afford the desired products **3**.

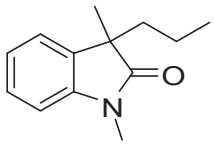
(B) Analytical data



3-Ethyl-1,3-dimethylindolin-2-one (3aa): [S2]

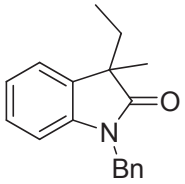
Yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.26 (t, J = 6.4 Hz, 1H), 7.17 (d, J = 7.2 Hz, 1H), 7.07 (t, J = 7.2 Hz, 1H), 6.84 (d, J = 8.0 Hz, 1H), 3.22 (s, 3H), 1.97-1.89 (m,

1H), 1.82-1.73 (m, 1H), 1.35 (s, 3H), 0.59 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.8, 143.5, 133.9, 127.6, 122.5, 122.4, 107.8, 48.9, 31.4, 26.0, 23.3, 8.8; IR (KBr, cm^{-1}): 1722, 1461; LRMS (EI, 70 eV) *m/z* (%): 189 (M^+, 21), 161 (100), 190 (6); HRMS *m/z* (ESI) calcd for $C_{12}H_{16}NO$ ($[M+H]^+$) 190.1154, found 190.1161.



1,3-Dimethyl-3-propylindolin-2-one (3ae):

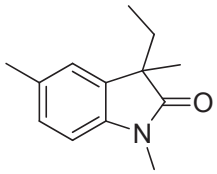
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.26 (t, J = 8.0 Hz, 1H), 7.17 (d, J = 7.2 Hz, 1H), 7.06 (t, J = 7.6 Hz, 1H), 6.84 (d, J = 8.0 Hz, 1H), 3.21 (s, 3H), 1.91-1.84 (m, 1H), 1.74-1.67 (m, 1H), 1.35 (s, 3H), 1.07-0.95 (m, 1H), 0.90-0.83 (m, 1H), 0.77 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.9, 143.3, 134.3, 127.6, 122.5, 122.4, 107.8, 48.5, 40.8, 26.1, 23.7, 17.8, 14.1; IR (KBr, cm^{-1}): 1719, 1466; LRMS (EI, 70 eV) *m/z* (%): 203 (M^+, 20), 161 (100), 204 (3); HRMS *m/z* (ESI) calcd for $C_{13}H_{18}NO$ ($[M+H]^+$) 204.1310, found 204.1315.



1-Benzyl-3-ethyl-3-methylindolin-2-one (3ba): [S3]

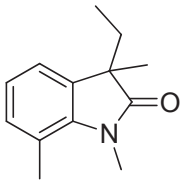
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.22 (m, 5H), 7.18-7.11 (m, 2H), 7.02 (t, J = 7.6 Hz, 1H), 6.72 (d, J = 7.6 Hz, 1H), 4.99 (t, J = 15.6 Hz, 1H), 4.85 (t, J = 15.6 Hz, 1H), 2.05-1.96 (m, 1H), 1.87-1.78 (m, 1H), 1.41 (s, 3H), 0.64 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.8, 142.5, 136.1, 133.8, 128.7, 127.5, 127.4, 127.2, 122.5, 122.4, 108.9. 48.9, 43.6, 31.4, 23.7, 9.0; IR (KBr, cm^{-1}): 1743, 1442;

LRMS (EI, 70 eV) *m/z* (%): 265 (M^+, 45), 91 (100), 266 (9); HRMS *m/z* (ESI) calcd for $C_{18}H_{20}NO$ ($[M+H]^+$) 266.1539, found 266.1551.



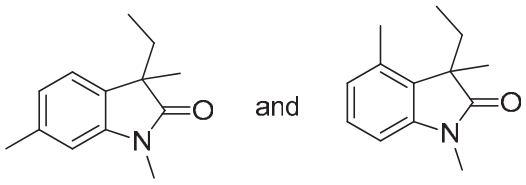
3-Ethyl-1,3,5-trimethylindolin-2-one (3da):[S4]

Yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.06 (d, *J* = 8.0 Hz, 1H), 6.98 (s, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 3.19 (s, 3H), 2.35 (s, 3H), 1.96-1.87 (m, 1H), 1.79-1.70 (m, 1H), 1.34 (s, 3H), 0.58 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.7, 141.1, 134.0, 131.9, 127.8, 123.4, 107.5, 49.0, 31.5, 26.1, 23.4, 21.2, 8.9; IR (KBr, cm^{-1}): 1738, 1442; LRMS (EI, 70 eV) *m/z* (%): 203 (M^+, 36), 174 (100), 204 (5); HRMS *m/z* (ESI) calcd for $C_{13}H_{18}NO$ ($[M+H]^+$) 204.1383, found 204.1381.



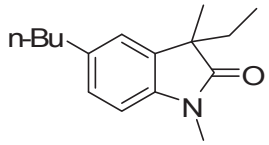
3-Ethyl-1,3,7-trimethylindolin-2-one (3ea):

Yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.00-6.93 (m, 3H), 3.50 (s, 3H), 2.59 (s, 3H), 1.95-1.88 (m, 1H), 1.77-1.68 (m, 1H), 1.32 (s, 3H), 0.57 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.5, 141.2, 134.6, 131.3, 122.3, 120.4, 119.4, 48.2, 31.8, 29.4, 23.8, 19.1, 8.9; IR (KBr, cm^{-1}): 1722, 1461; LRMS (EI, 70 eV) *m/z* (%): 203 (M^+, 32), 174 (100), 204 (5); HRMS *m/z* (ESI) calcd for $C_{13}H_{18}NO$ ($[M+H]^+$) 204.1383, found 204.1383.



3-Ethyl-1,3,6-trimethylindolin-2-one (3fa) and 3-Ethyl-1,3,4-trimethylindolin-2-one (3fa') (1.7:1):

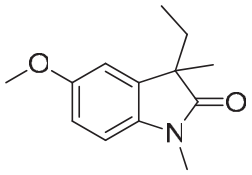
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.17 (t, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 7.2 Hz, 0.67H), 6.88 (d, *J* = 7.2 Hz, 0.67H), 6.83 (d, *J* = 7.6 Hz, 1H), 6.70 (s, 0.67H), 6.68 (s, 1H), 3.21 (s, 3H), 3.20 (s, 2H), 2.39 (s, 2H), 2.36 (s, 3H), 2.04-1.86 (m, 2.7H), 1.77-1.1.70 (m, 0.7H), 1.43 (s, 3H), 1.33 (s, 2H), 0.59 (t, *J* = 7.6 Hz, 2H), 0.49 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.1, 180.7, 143.5, 137.7, 134.2, 131.0, 130.3, 127.5, 125.0, 122.9, 122.3, 108.8, 105.6, 50.2, 48.7, 31.4, 29.4, 26.2, 23.4, 22.1, 21.8, 18.1, 14.1, 9.2, 8.9; IR (KBr, cm^{-1}): 1738, 1442; LRMS (EI, 70 eV) *m/z* (%): 203 (M^+, 36), 174 (100), 204 (5); HRMS *m/z* (ESI) calcd for $C_{13}H_{18}NO$ ($[M+H]^+$) 204.1383, found 204.1381.



5-Butyl-3-ethyl-1,3-dimethylindolin-2-one (3ga):

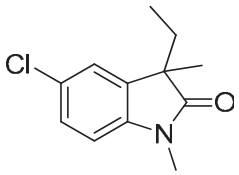
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.06 (d, *J* = 8.0 Hz, 1H), 6.98 (s, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 3.20 (s, 3H), 2.60 (t, *J* = 7.6 Hz, 3H), 1.96-1.87 (m, 1H), 1.80-1.71 (m, 1H), 1.63-1.55 (m, 2H), 1.41-1.31 (m, 2H), 1.34 (s, 3H), 0.93 (t, *J* = 7.6 Hz, 3H), 0.59 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.8, 141.3, 137.2, 133.9, 127.2, 122.7, 107.5, 49.0, 35.5, 34.1, 31.5, 26.1, 23.4, 22.4, 14.0, 8.9; IR (KBr,

cm^{-1}): 1746, 1456; LRMS (EI, 70 eV) *m/z* (%): 245 (M^+, 33), 202 (100), 246 (5); HRMS *m/z* (ESI) calcd for $C_{16}H_{24}NO$ ($[M+H]^+$) 246.1852, found 246.1842.



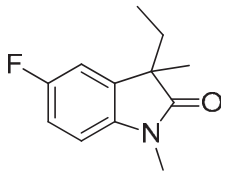
3-Ethyl-5-methoxy-1,3-dimethylindolin-2-one (3ha): [S5]

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.80-6.77 (m, 2H), 6.74 (t, *J* = 6.0 Hz, 1H), 3.81 (s, 3H), 3.19 (s, 3H), 1.97-1.89 (m, 1H), 1.79-1.67 (m, 1H), 1.34 (s, 3H), 0.59 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.4, 156.0, 137.1, 135.4, 111.5, 110.3, 108.0, 55.8, 49.4, 31.5, 26.2, 23.4, 8.9; IR (KBr, cm^{-1}): 1741, 1472; LRMS (EI, 70 eV) *m/z* (%): 219 (M^+, 58), 190 (100), 210 (10); HRMS *m/z* (ESI) calcd for $C_{13}H_{18}NO_2$ ($[M+H]^+$) 220.1332, found 220.1333.



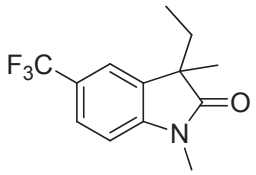
5-Chloro-3-ethyl-1,3-dimethylindolin-2-one (3ia): [S4]

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24 (d, *J* = 8.4 Hz, 1H), 7.14 (d, *J* = 2.0 Hz, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 3.20 (s, 3H), 1.98-1.80 (m, 1H), 1.78-1.71 (m, 1H), 1.35 (s, 3H), 0.59 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.2, 142.1, 135.7, 127.9, 127.6, 123.1, 108.8, 49.3, 31.4, 26.2, 23.3, 8.8; IR (KBr, cm^{-1}): 1728, 1459; LRMS (EI, 70 eV) *m/z* (%): 223 (M^+, 48), 194 (100), 224 (7); HRMS *m/z* (ESI) calcd for $C_{12}H_{15}ClNO$ ($[M+H]^+$) 224.0837, found 224.0843.



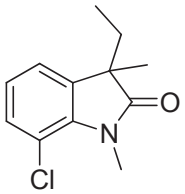
3-Ethyl-5-fluoro-1,3-dimethylindolin-2-one (3ja): [S4]

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.99-6.90 (m, 2H), 6.76 (d, *J* = 8.4 Hz, 1H), 3.21 (s, 3H), 1.99-1.88 (m, 1H), 1.79-1.70 (m, 1H), 1.35 (s, 3H), 0.59 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.4, 159.4 (d, *J* = 238.7 Hz, 1C), 139.4, 135.7 (d, *J* = 7.6 Hz, 1C), 113.7 (d, *J* = 23.3 Hz, 1C), 110.7 (d, *J* = 24.3 Hz, 1C), 108.2 (d, *J* = 8.1 Hz, 1C), 49.5, 31.4, 26.2, 23.3, 8.8; IR (KBr, cm^{-1}): 1728, 1465; LRMS (EI, 70 eV) m/z (%): 207 (M^+, 42), 178 (100), 208 (6); HRMS *m/z* (ESI) calcd for $C_{12}H_{15}FNO$ ($[M+H]^+$) 208.1132, found 208.1140.



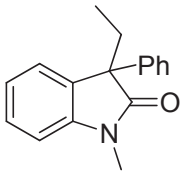
3-Ethyl-1,3-dimethyl-5-(trifluoromethyl)indolin-2-one (3ka):

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.4 Hz, 1H), 7.40 (s, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 3.25 (s, 3H), 2.01-1.92 (m, 1H), 1.85-1.76 (m, 1H), 1.38 (s, 3H), 0.60 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.6, 146.5, 134.5, 125.5 (m, 1C), 124.1 (m, 1C), 119.5 (m, 1C), 107.6, 49.0, 31.4, 26.3, 23.2, 8.8; IR (KBr, cm^{-1}): 1733, 1472; LRMS (EI, 70 eV) m/z (%): 257 (M^+, 73), 228 (100), 258 (11); HRMS *m/z* (ESI) calcd for $C_{13}H_{15}F_3NO$ ($[M+H]^+$) 258.1100, found 258.1100.



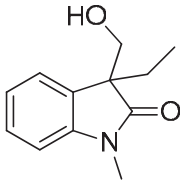
7-Chloro-3-ethyl-1,3-dimethylindolin-2-one (3la):

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.18 (d, *J* = 8.0 Hz, 1H), 7.04-6.95 (m, 2H), 3.58 (s, 3H), 1.99-1.90 (m, 1H), 1.78-1.69 (m, 1H), 1.34 (s, 3H), 0.58 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.9, 138.3, 135.8, 128.9, 122.2, 120.0, 114.3, 47.8, 30.8, 28.4, 22.7, 7.8; IR (KBr, cm^{-1}): 1728, 1459; LRMS (EI, 70 eV) *m/z* (%): 223 (M^+, 48), 194 (100), 224 (7); HRMS *m/z* (ESI) calcd for $C_{12}H_{15}ClNO$ ($[M+H]^+$) 224.0837, found 224.0828.



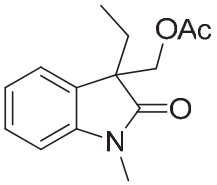
3-Ethyl-1-methyl-3-phenylindolin-2-one (3ma): [S6]

Yellow solid; mp 77.9-78.6 ^{o}C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38-7.20 (m, 7H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 3.22 (s, 3H), 2.47-2.39 (m, 1H), 2.28-2.19 (m, 1H), 0.68 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.6, 144.1, 140.3, 132.1, 128.5, 128.1, 127.2, 127.0, 124.8, 122.6, 108.2, 57.3, 30.9, 26.3, 9.1; IR (KBr, cm^{-1}): 1740, 1473; LRMS (EI, 70 eV) *m/z* (%): 251 (M^+, 75), 222 (100), 252 (14); HRMS *m/z* (ESI) calcd for $C_{17}H_{18}NO$ ($[M+H]^+$) 252.1383, found 252.1390.



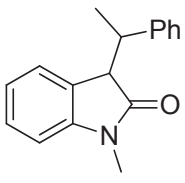
3-Ethyl-3-(hydroxymethyl)-1-methylindolin-2-one (3na):

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32 (t, *J* = 7.6 Hz, 1H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.88 (t, *J* = 7.6 Hz, 1H), 3.89 (t, *J* = 10.8 Hz, 1H), 3.75 (t, *J* = 10.8 Hz, 1H), 3.28 (s, 3H), 2.11-2.02 (m, 1H), 1.88-1.79 (m, 1H), 1.26 (s, 1H), 0.62 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.4, 144.4, 129.8, 128.2, 122.9, 122.7, 108.2, 66.9, 55.3, 42.7, 26.1, 8.3; IR (KBr, cm^{-1}): 3620, 1728, 1461; LRMS (EI, 70 eV) *m/z* (%): 205 (M^+, 28), 160 (100), 206 (4); HRMS *m/z* (ESI) calcd for $C_{12}H_{16}NO_2$ ($[M+H]^+$) 206.1176, found 206.1161.



(3-Ethyl-1-methyl-2-oxoindolin-3-yl)methyl acetate (3oa):

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33-7.28 (m, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.87 (t, *J* = 7.6 Hz, 1H), 4.53 (d, *J* = 10.8 Hz, 1H), 4.19 (t, *J* = 10.8 Hz, 1H), 3.24 (s, 3H), 1.96-1.82 (m, 5H), 0.60 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 177.5, 170.4, 144.2, 129.3, 128.3, 123.2, 122.5, 107.9, 67.2, 53.1, 26.7, 26.1, 20.5, 8.0; IR (KBr, cm^{-1}): 1743, 1712, 1433; LRMS (EI, 70 eV) *m/z* (%): 247 (M^+, 33), 175 (100), 248 (5); HRMS *m/z* (ESI) calcd for $C_{14}H_{18}NO_3$ ($[M+H]^+$) 248.1281, found 248.1296.



dr = 8.3:1

1-Methyl-3-(1-phenylethyl)indolin-2-one (3pa): [S4]

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35-7.25 (m, 4H), 7.12 (d, *J* = 7.2 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.87 (d, *J* = 8.8 Hz, 1H), 3.41 (s, 3H), 2.97-2.89 (m, 1H), 1.15 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 172.3, 141.1, 139.9, 128.9, 128.8, 128.5, 128.3, 127.8, 127.1, 123.0, 114.6, 48.9, 42.1, 29.9, 15.5; IR (KBr, cm^{-1}): 1731, 1445; LRMS (EI, 70 eV) *m/z* (%): 251 (100), 252 (22); HRMS *m/z* (ESI) calcd for $C_{17}H_{18}NO$ ($[M+H]^+$) 252.1383, found 252.1393.

(C) References

[S1] (a) Mu; X.; Wu; T.; Wang, H.-Y.; Guo, Y.-L.; Liu, G.-S. *J. Am. Chem. Soc*. **2012**, *134*, 878. (b) Pinto, A.; Jia, Y.; Neuville, L.; Zhu, J. *Chem. Eur. J*. **2007**, *13*, 961. (c) Jones, K.; Thompson, M.; Wright, C. *J. Chem. Soc*., *Chem. Commun*. **1986**, 715.

[S2] Wu, T.; Zhang, H.; Liu, G.-S. *Tetrahedron*. **2012**, *68*, 5229.

[S3] Alluri, S.; Feng, H.; Livings, M.; Samp, L.; Biswas, D.; Lam, T. W.; Lobkovsky, E.; Ganguly, A. K. *Tetrahedron Lett*. **2011***, 52,* 3945.

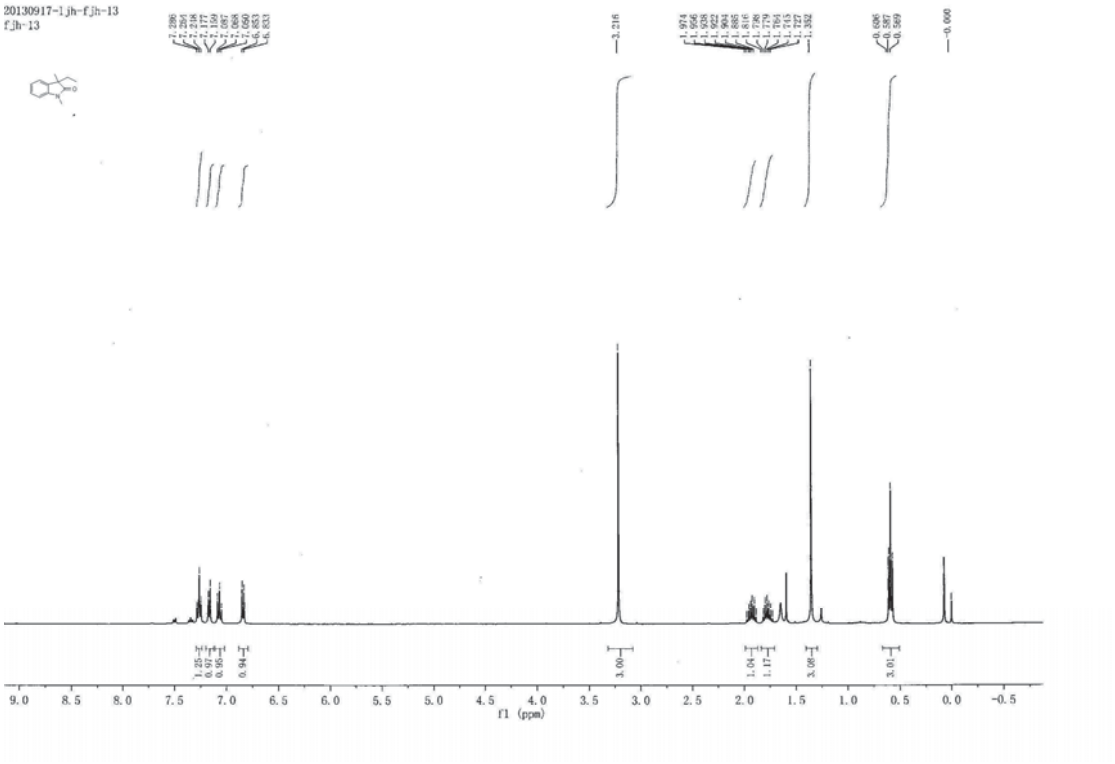
[S4] Xie, J.; Xu, P.; Li, H.-M.; Xue, Q.-C.; Jin, H.-M,; Cheng, Y.-X.; Zhu, C.-J. *Chem. Commun*. **2013**, *49*, 5672.

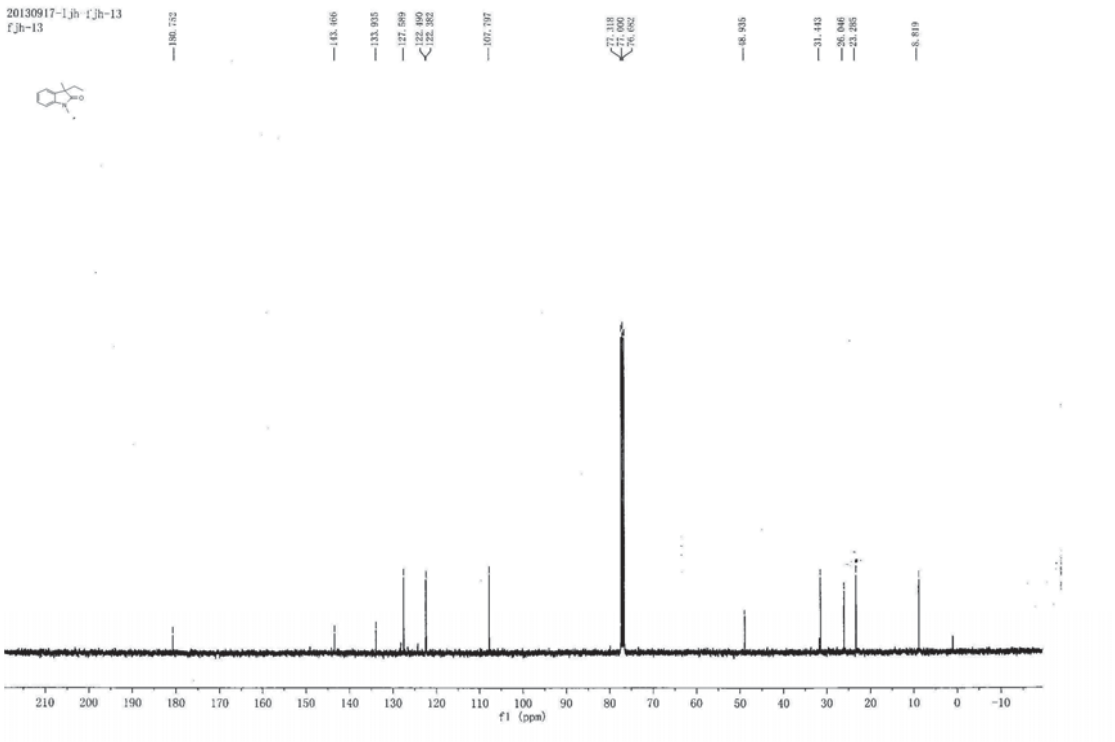
[S5] Chen, B.; Ji, Q.-E. *Yaoxue bao* **1990**, *25*, 247.

[S6] Beyer, A.; Buendia, J.; Bolm, C. *Org. Lett*. **2012,** *14*, 3948.

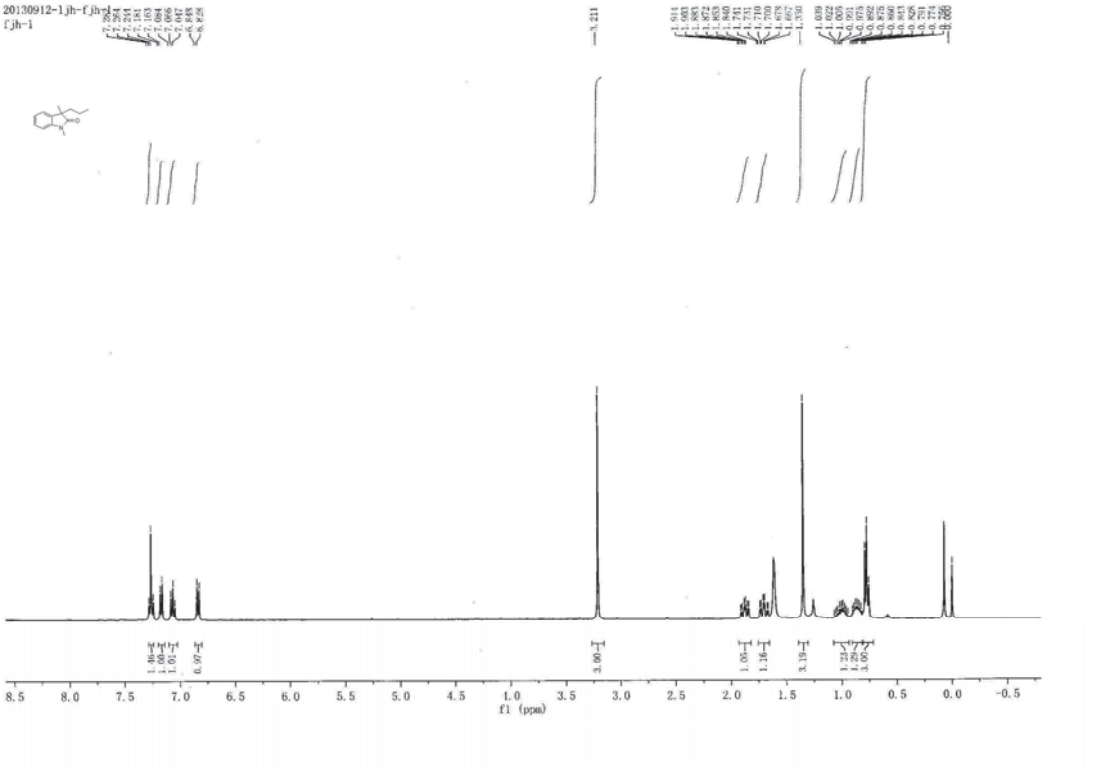
(D) Spectra

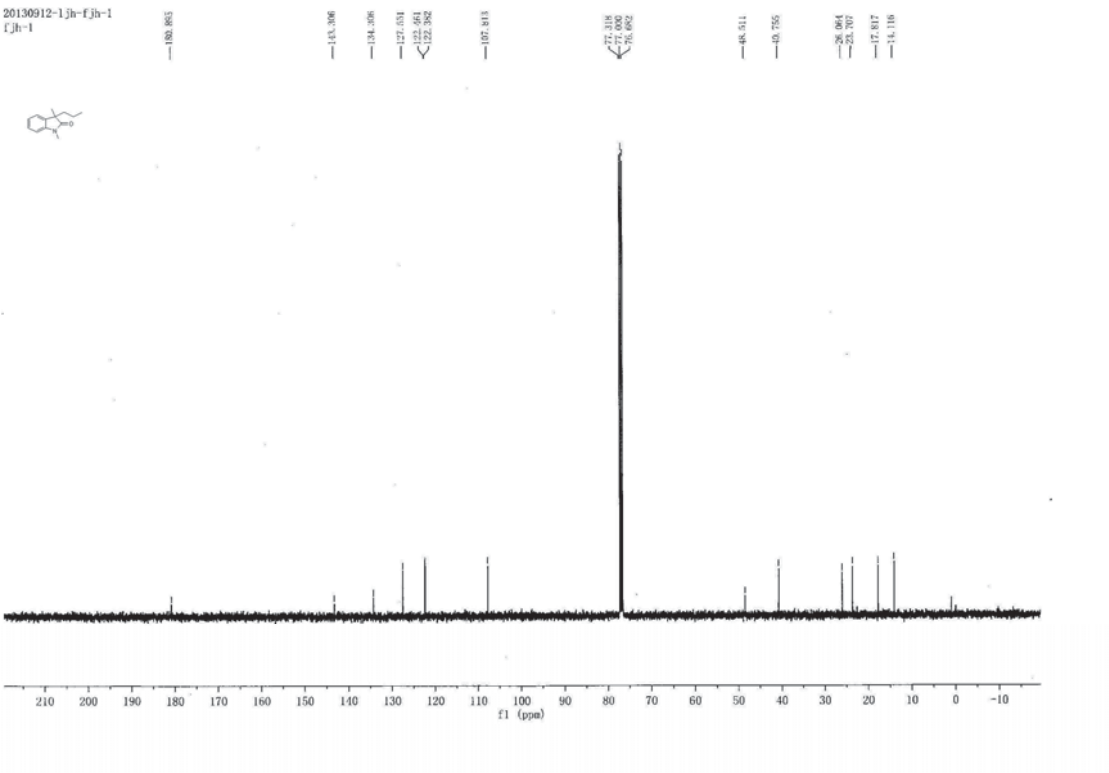
3-Ethyl-1,3-dimethylindolin-2-one (3aa)



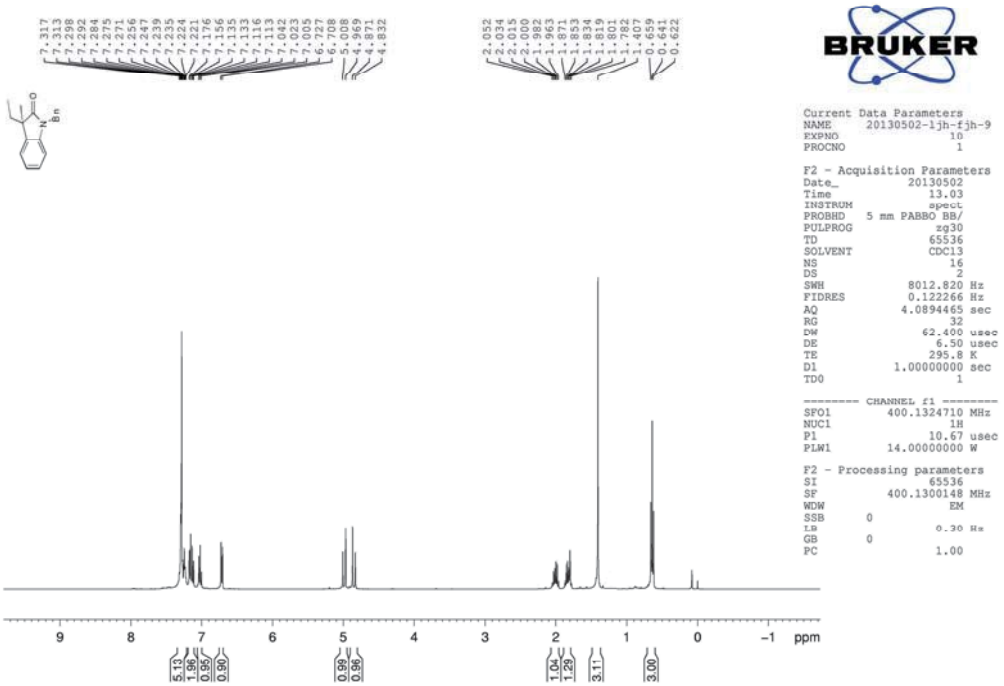


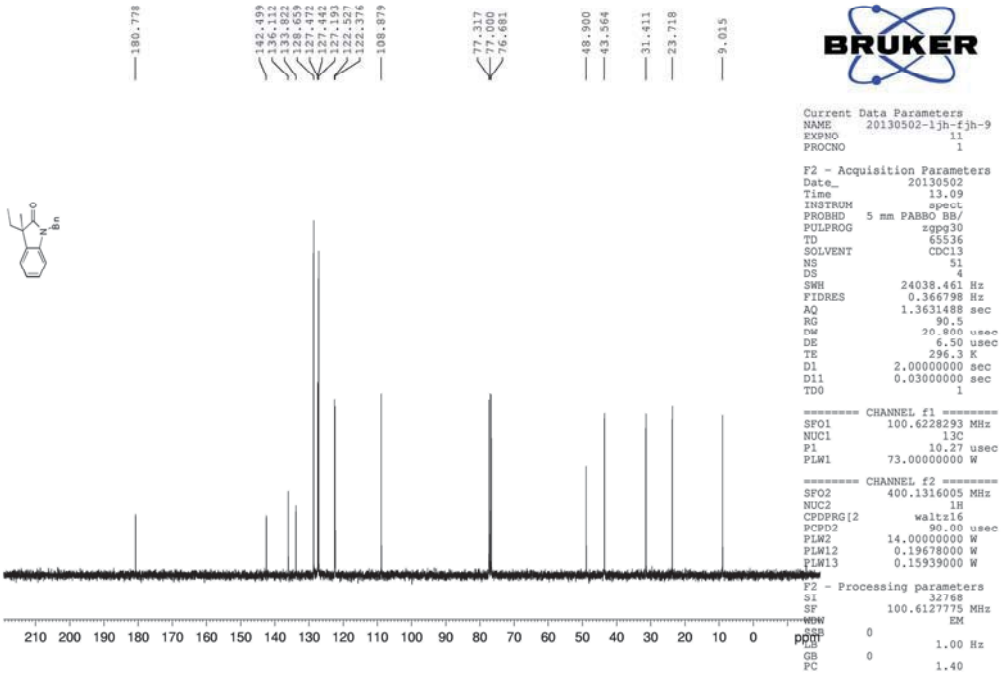
1,3-Dimethyl-3-propylindolin-2-one (3ae)



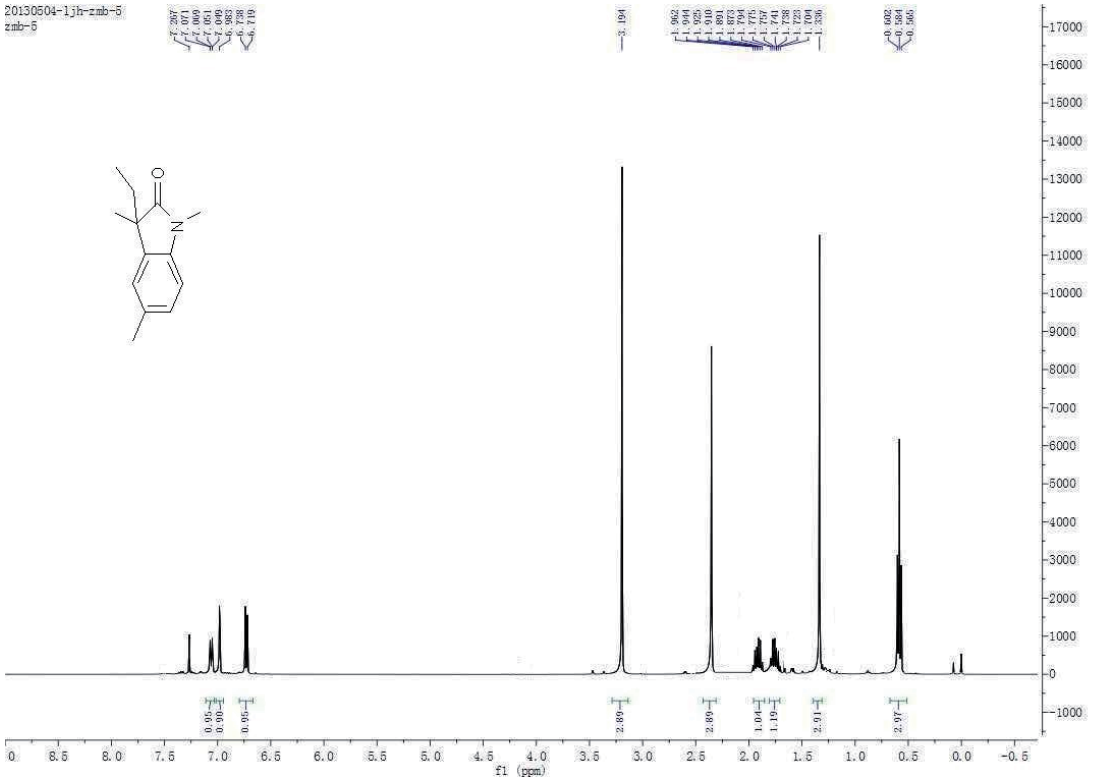


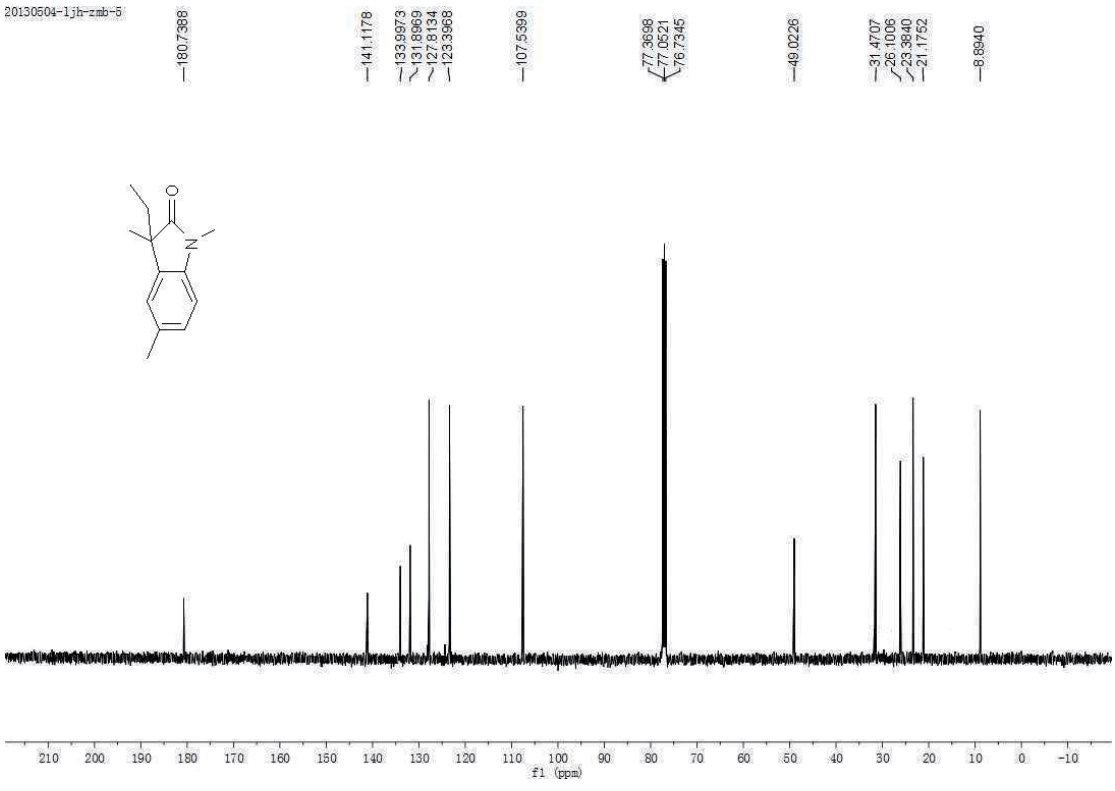
1-Benzyl-3-ethyl-3-methylindolin-2-one (3ba)



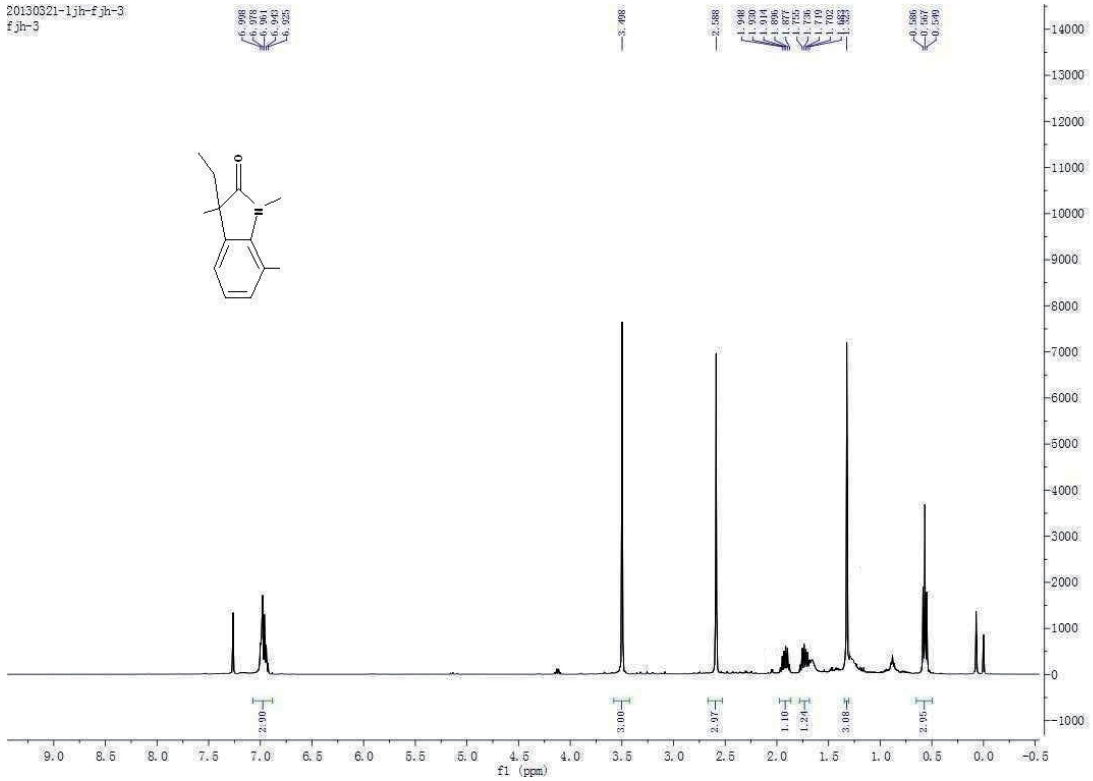


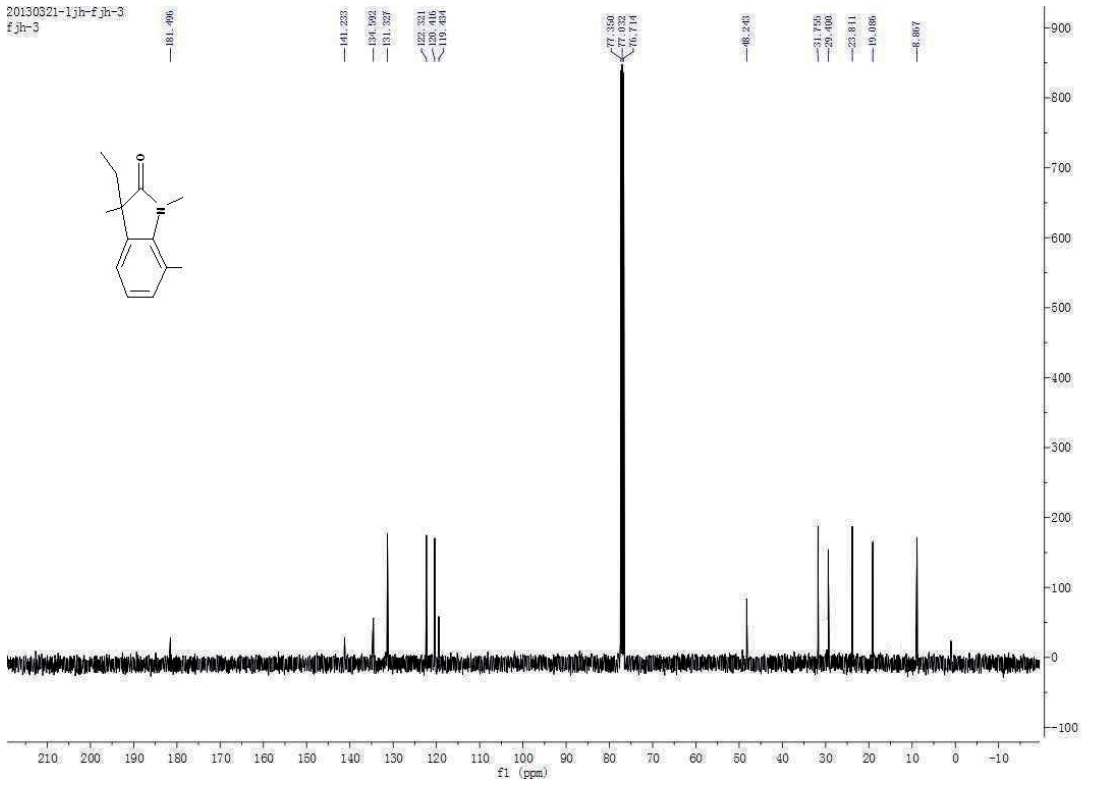
3-Ethyl-1,3,5-trimethylindolin-2-one (3da)



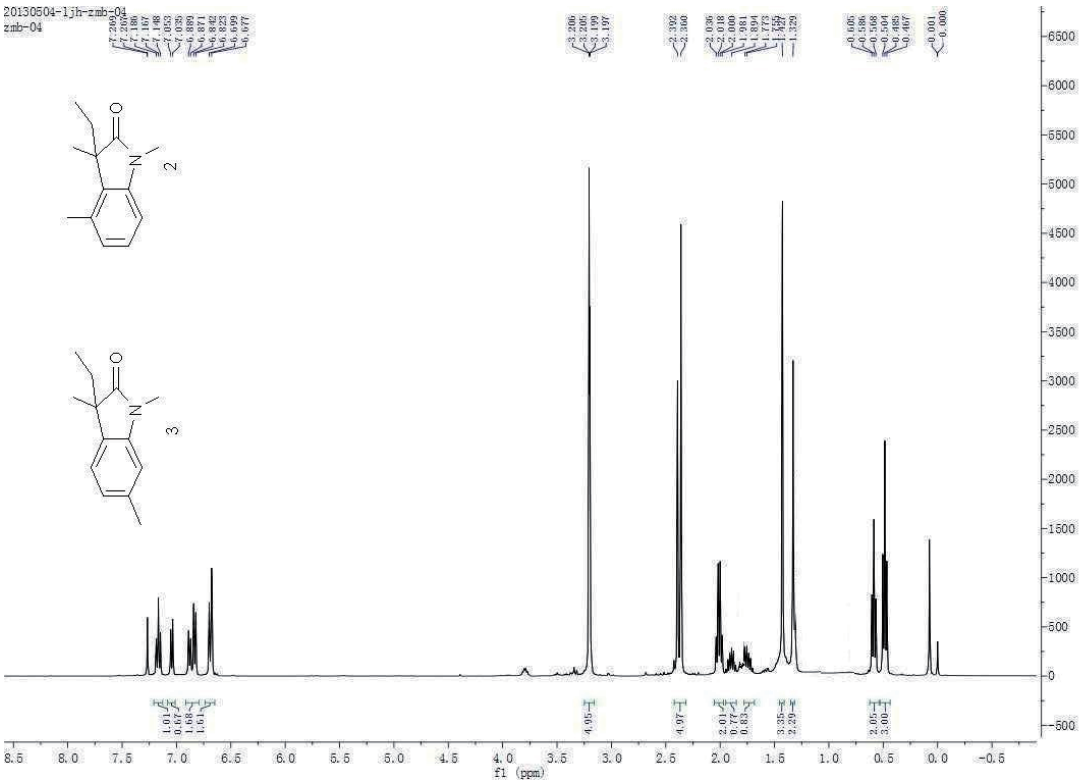


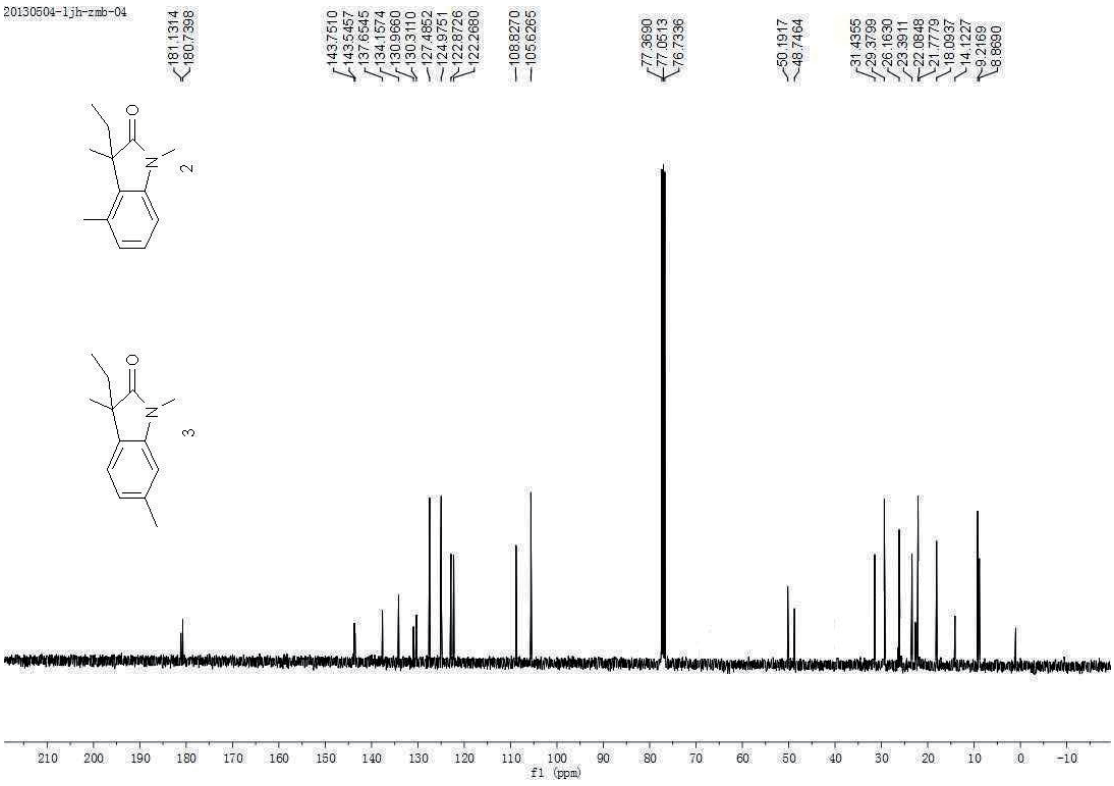
3-Ethyl-1,3,7-trimethylindolin-2-one (3ea)



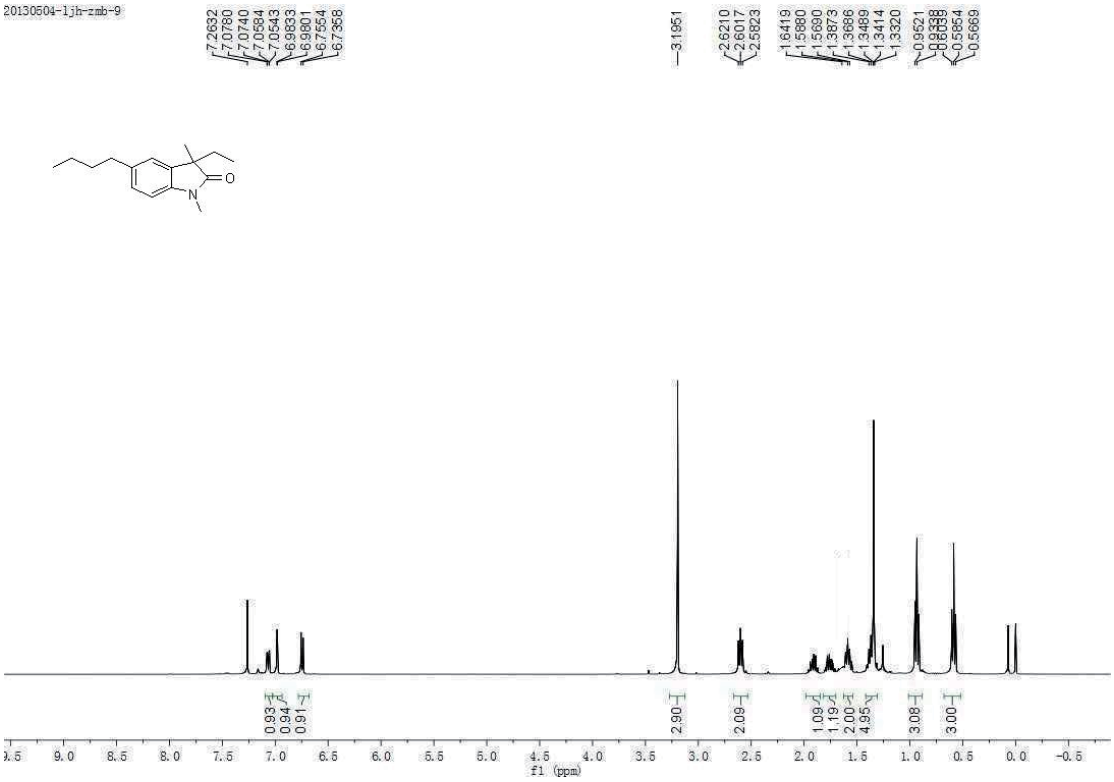


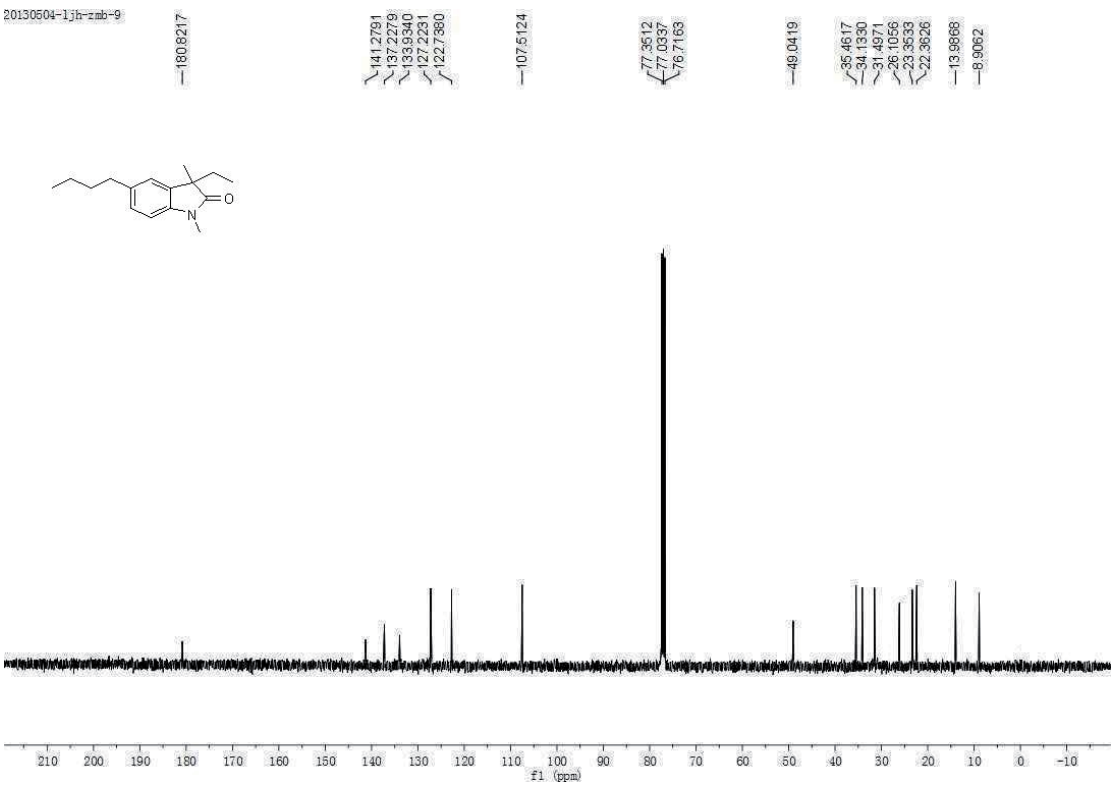
3-Ethyl-1,3,6-trimethylindolin-2-one (3fa) and 3-Ethyl-1,3,4-trimethylindolin-2-one (3fa')



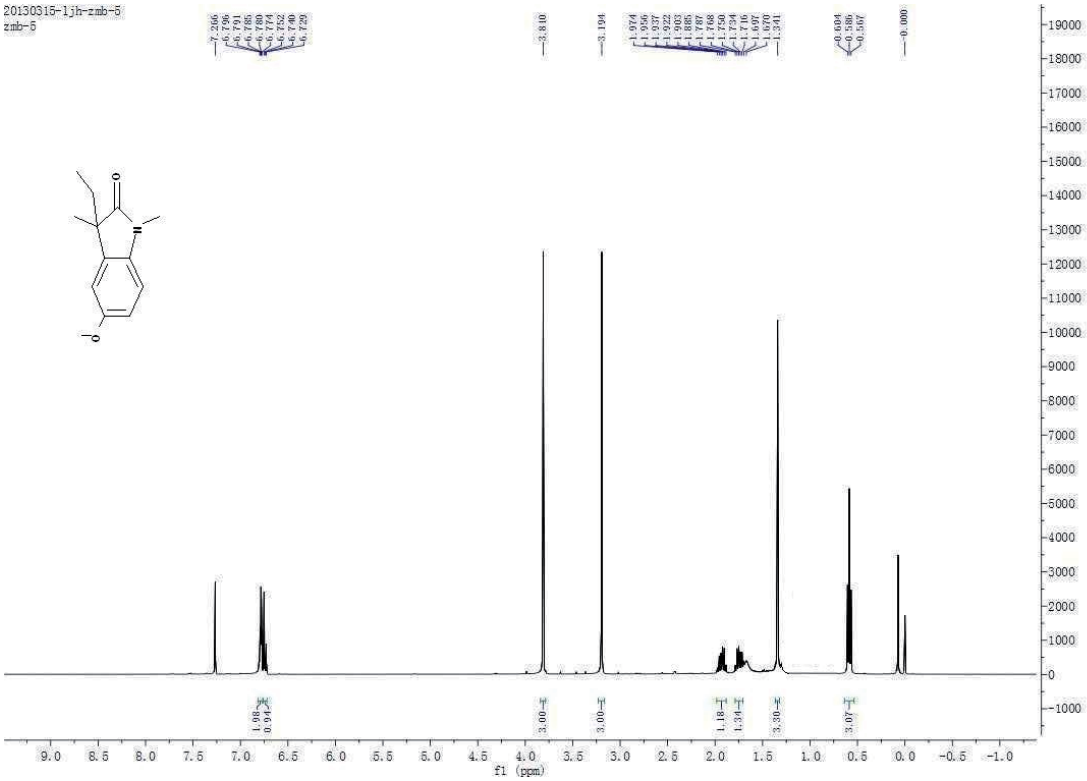


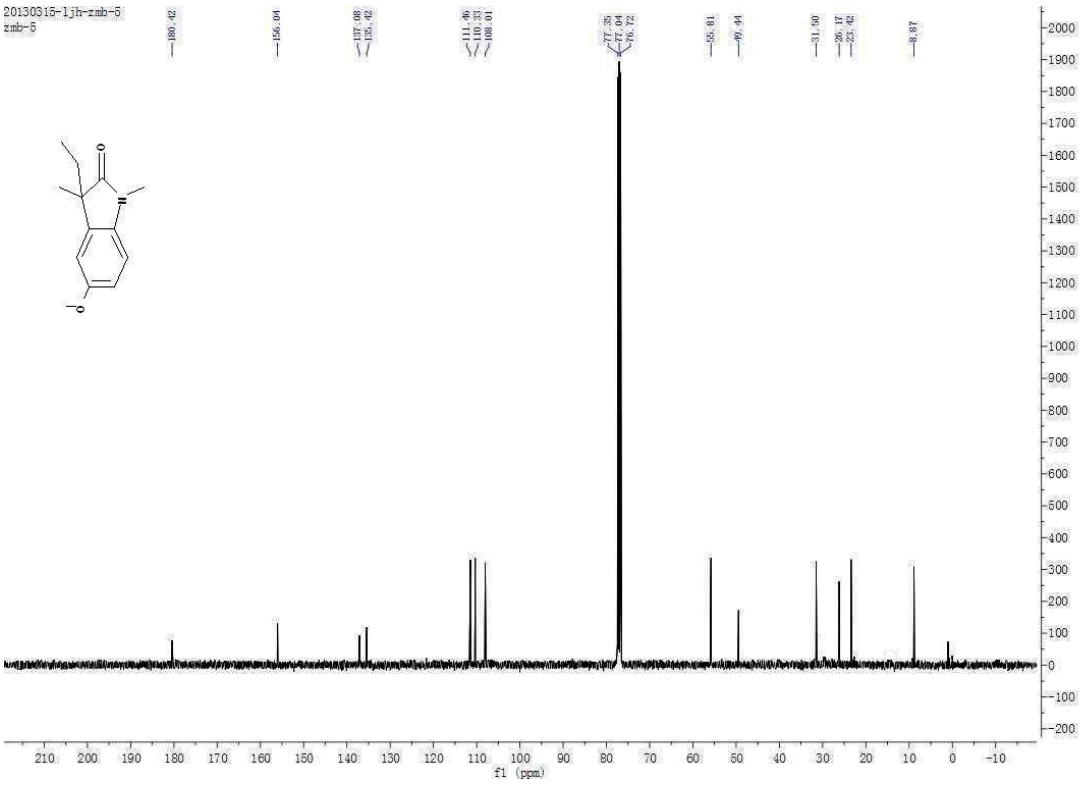
5-Butyl-3-ethyl-1,3-dimethylindolin-2-one (3ga)



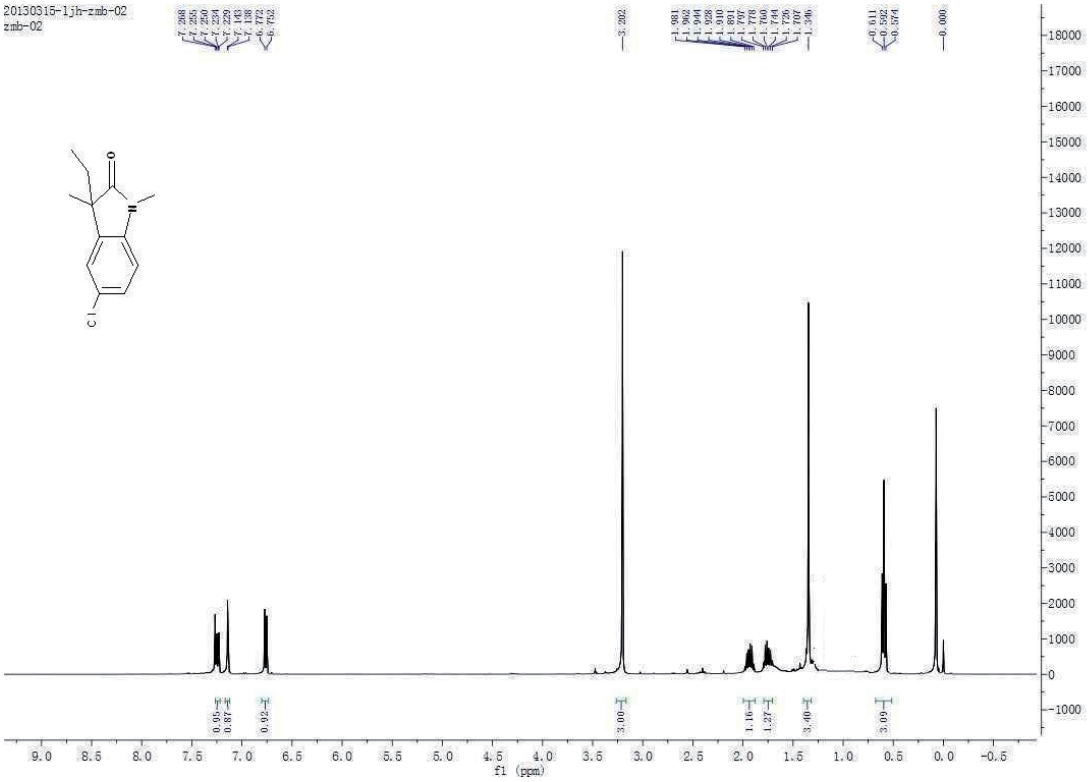


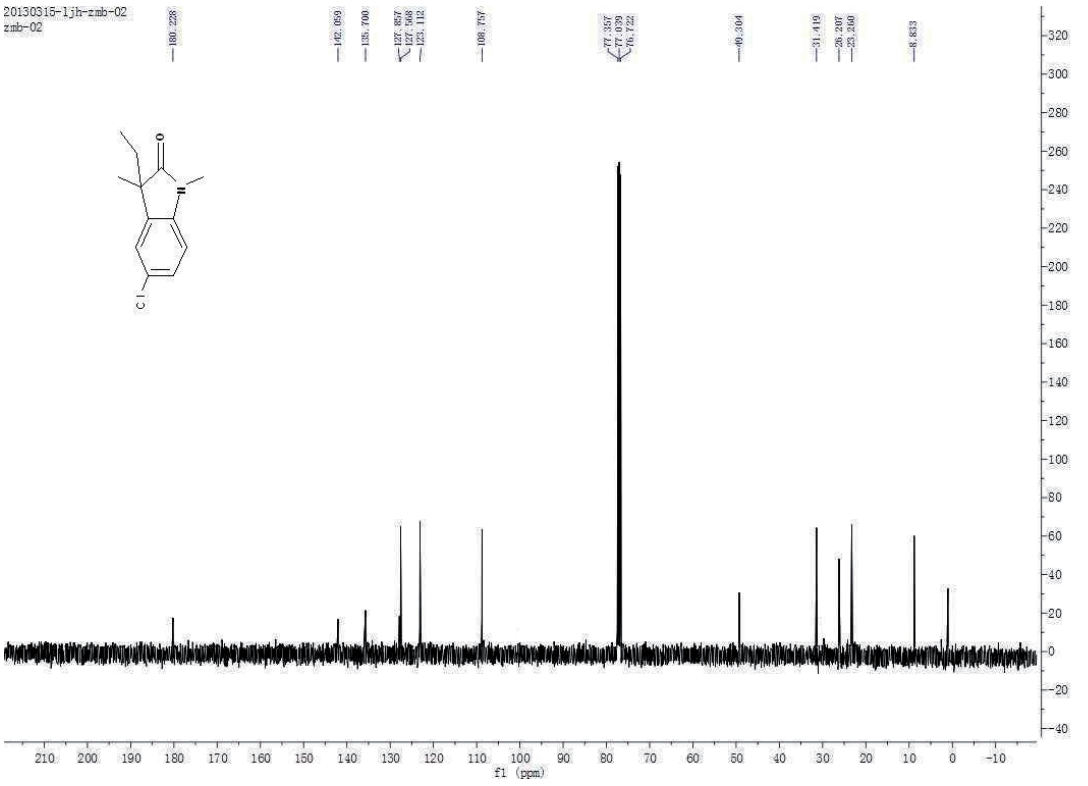
3-Ethyl-5-methoxy-1,3-dimethylindolin-2-one (3ha)



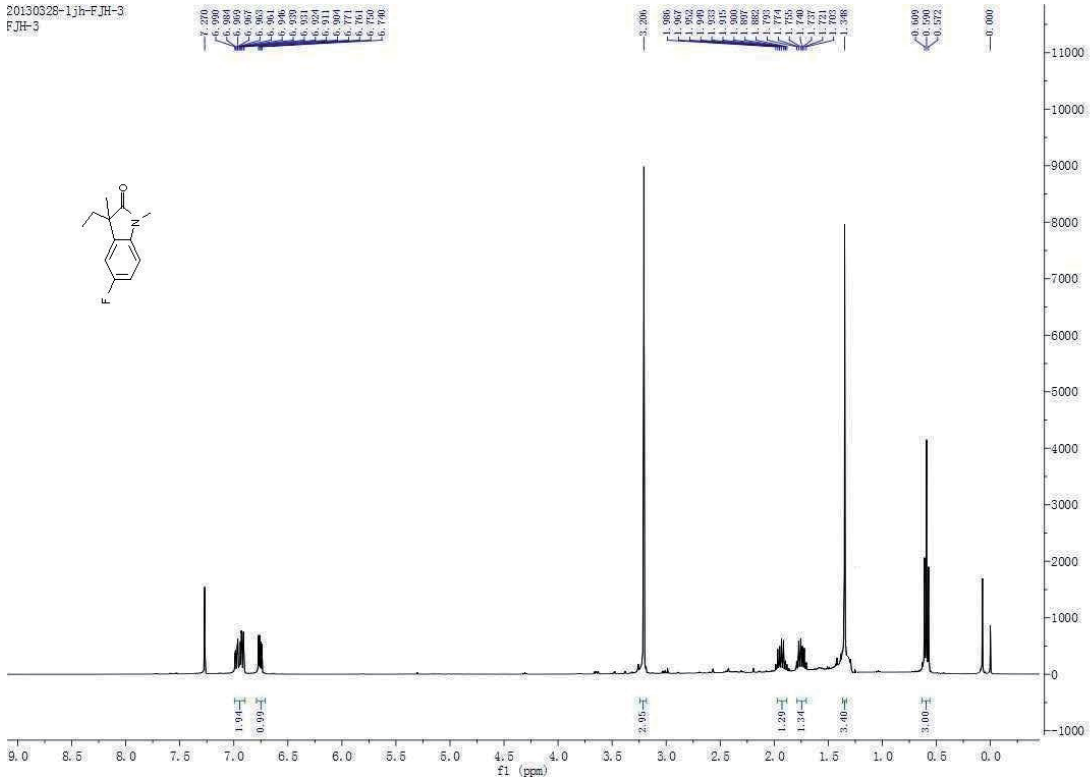


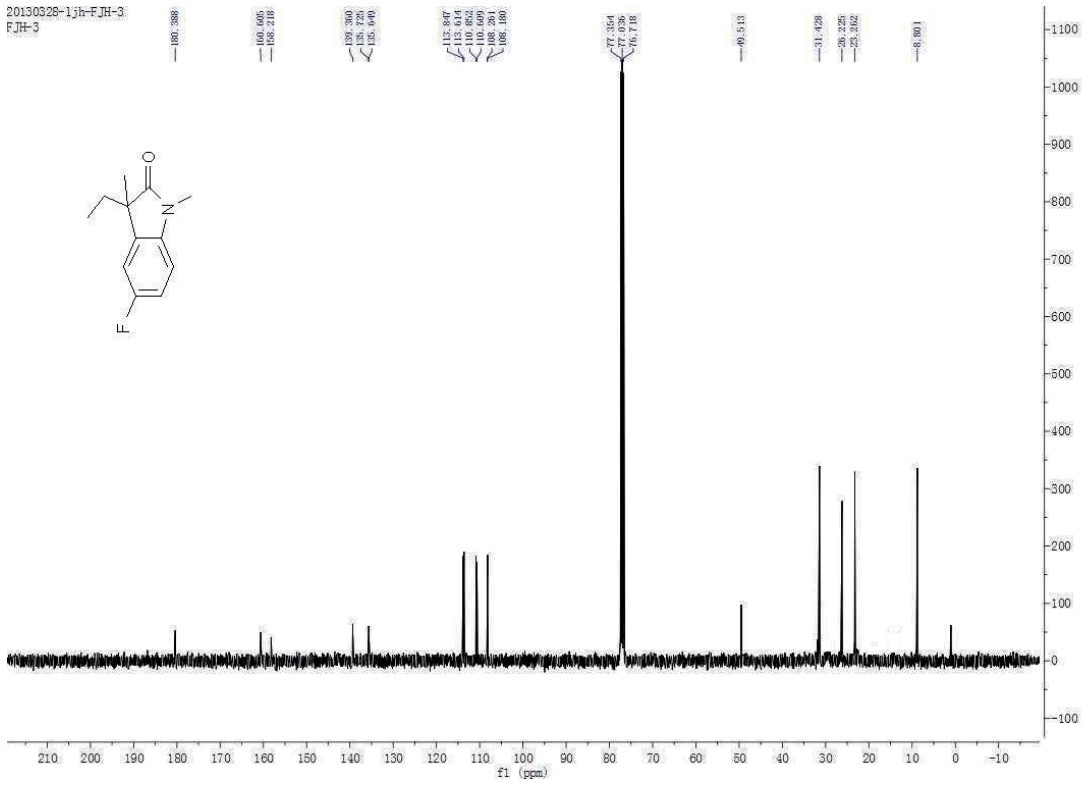
5-Chloro-3-ethyl-1,3-dimethylindolin-2-one (3ia)



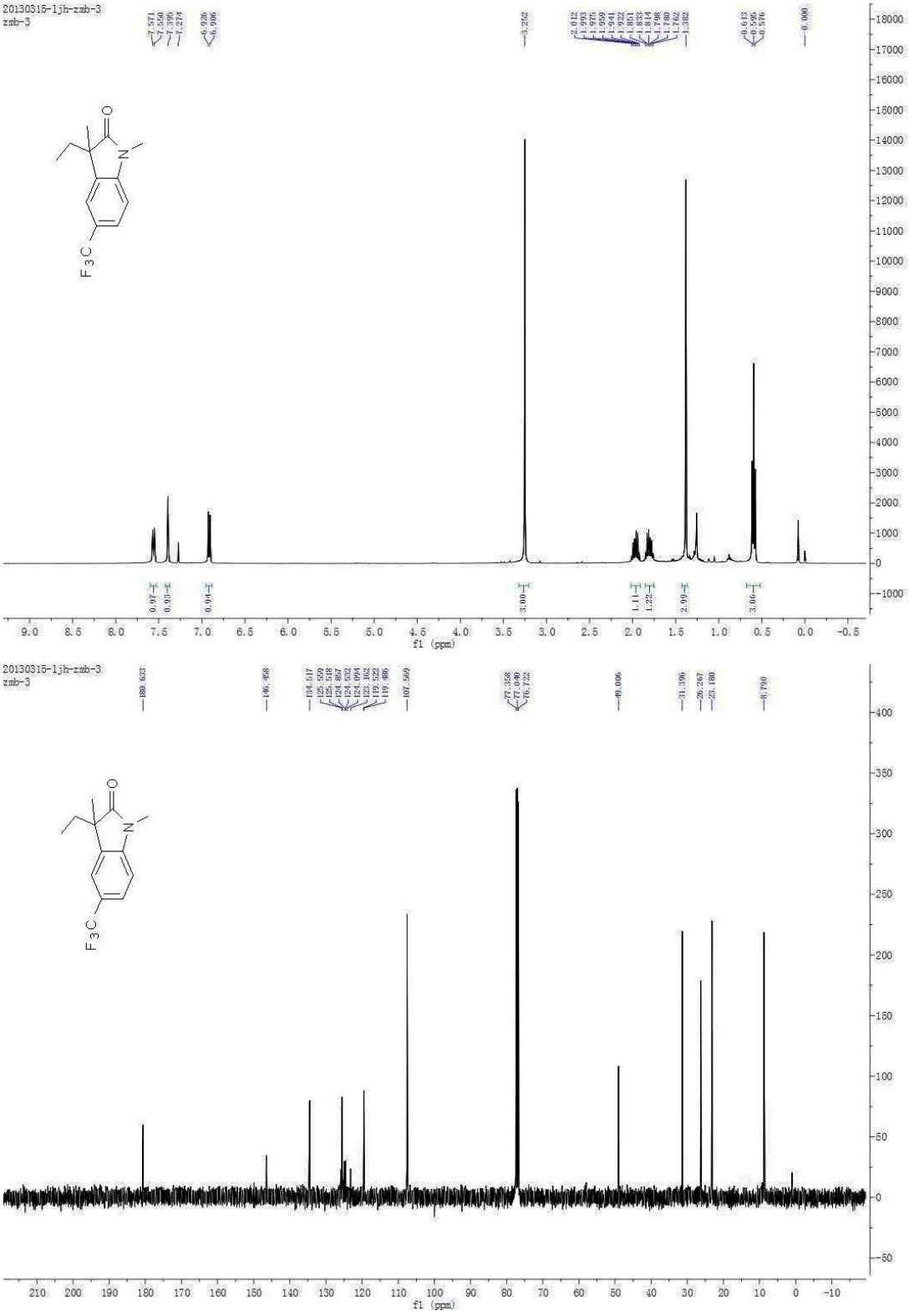


3-Ethyl-5-fluoro-1,3-dimethylindolin-2-one (3ja)

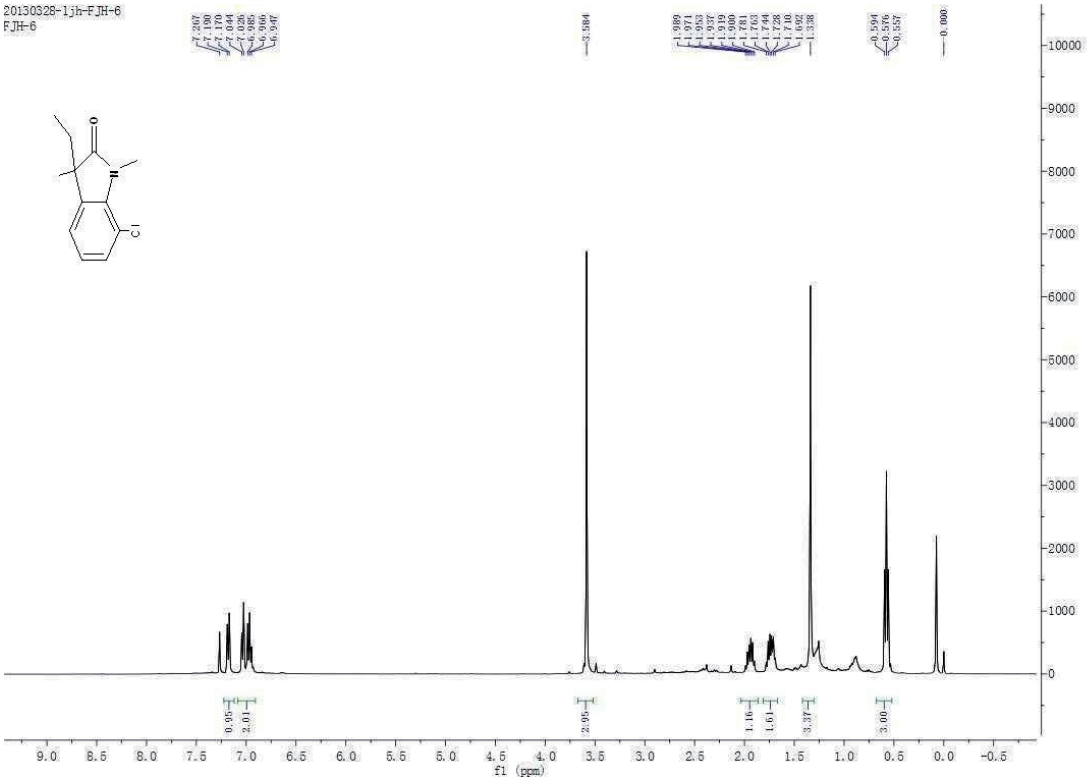


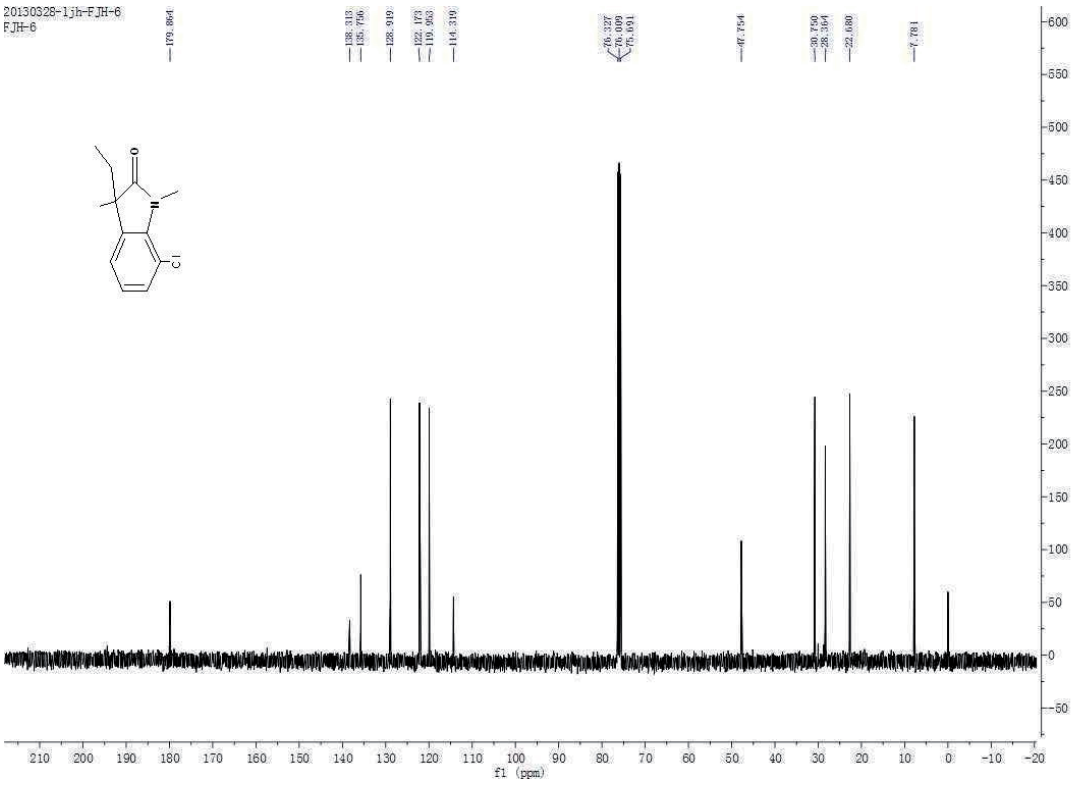


3-Ethyl-1, 3-dimethyl-5-(trifluoromethyl)indolin-2-one (3ka)

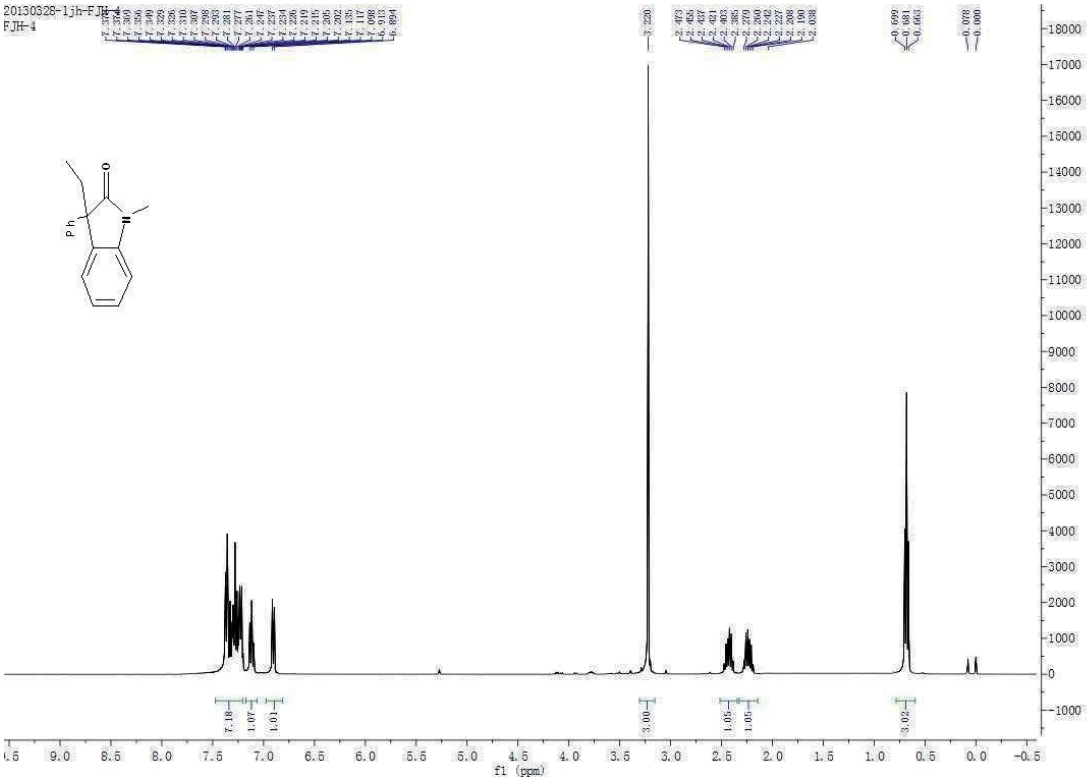


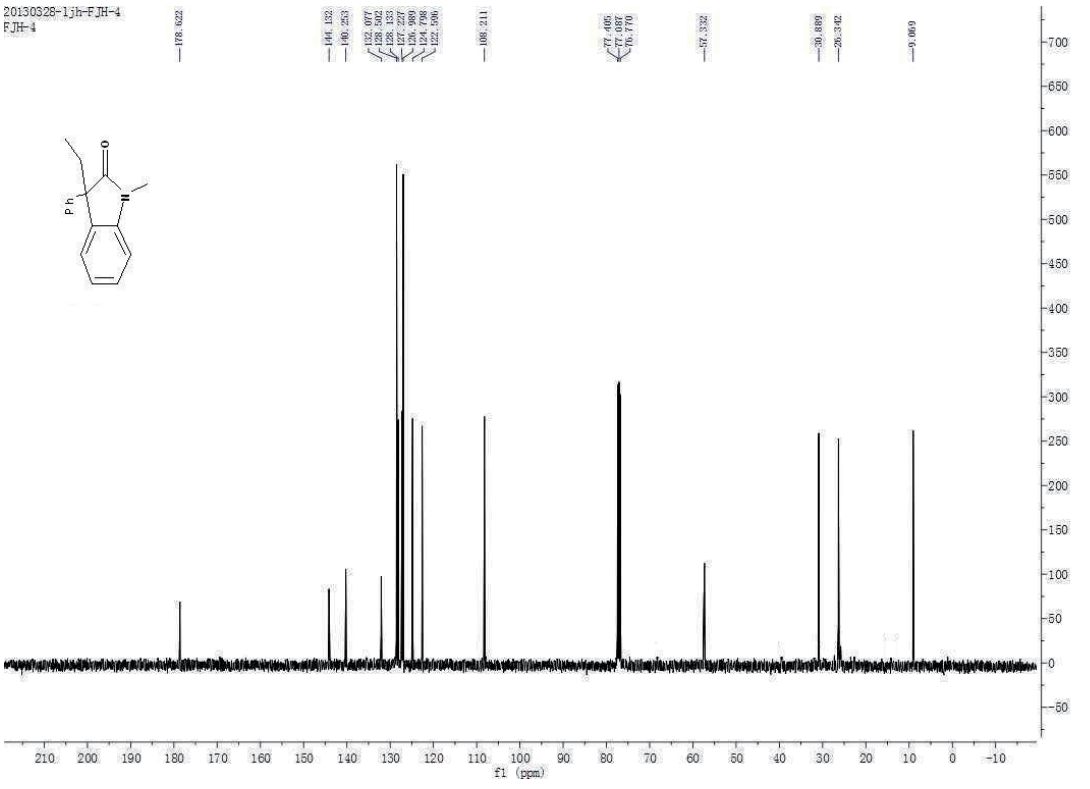
7-Chloro-3-ethyl-1,3-dimethylindolin-2-one (3la)



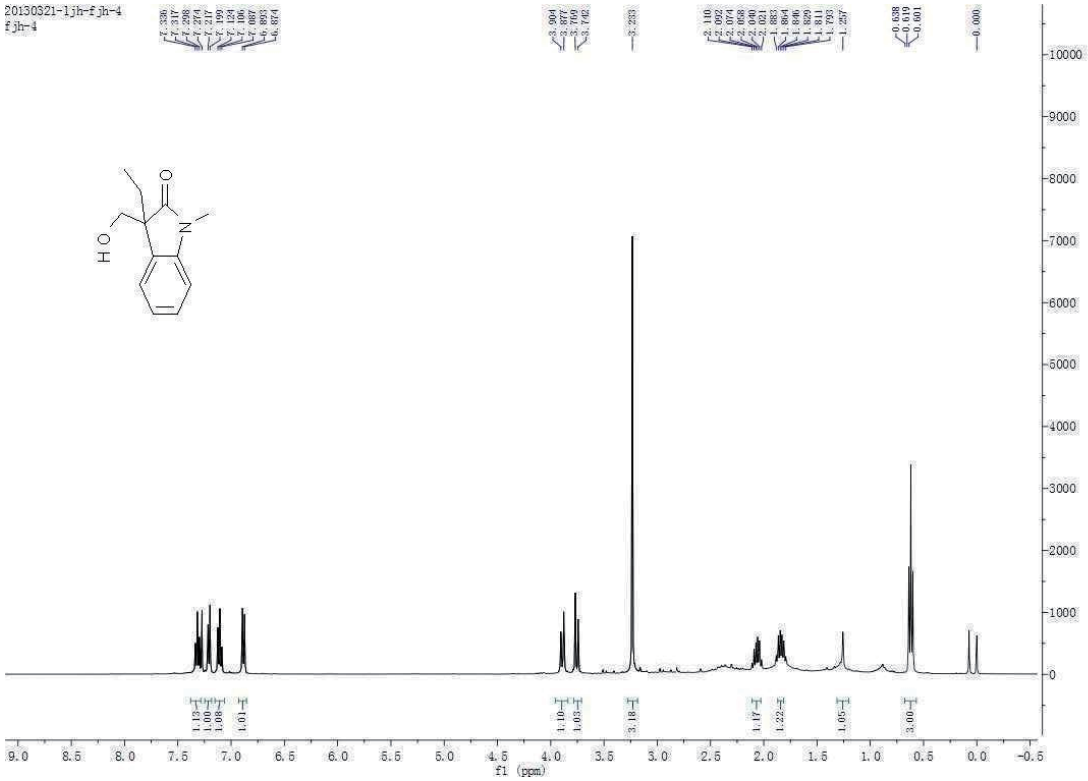


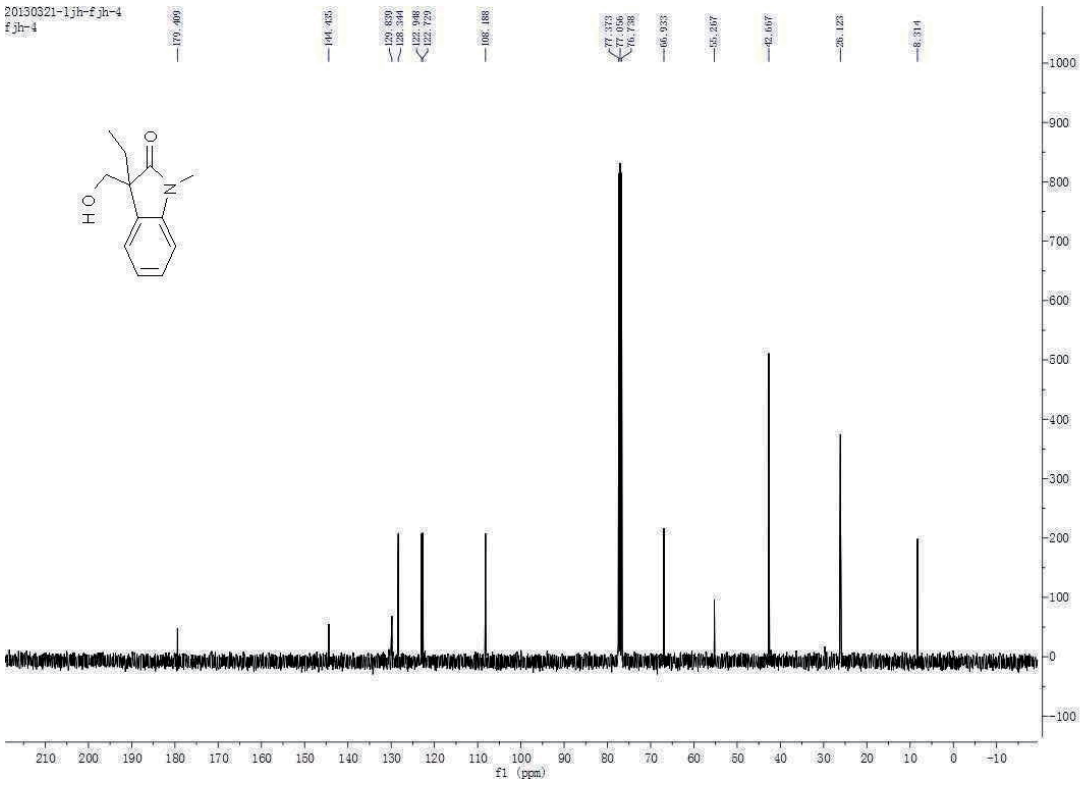
3-Ethyl-1-methyl-3-phenylindolin-2-one (3ma)



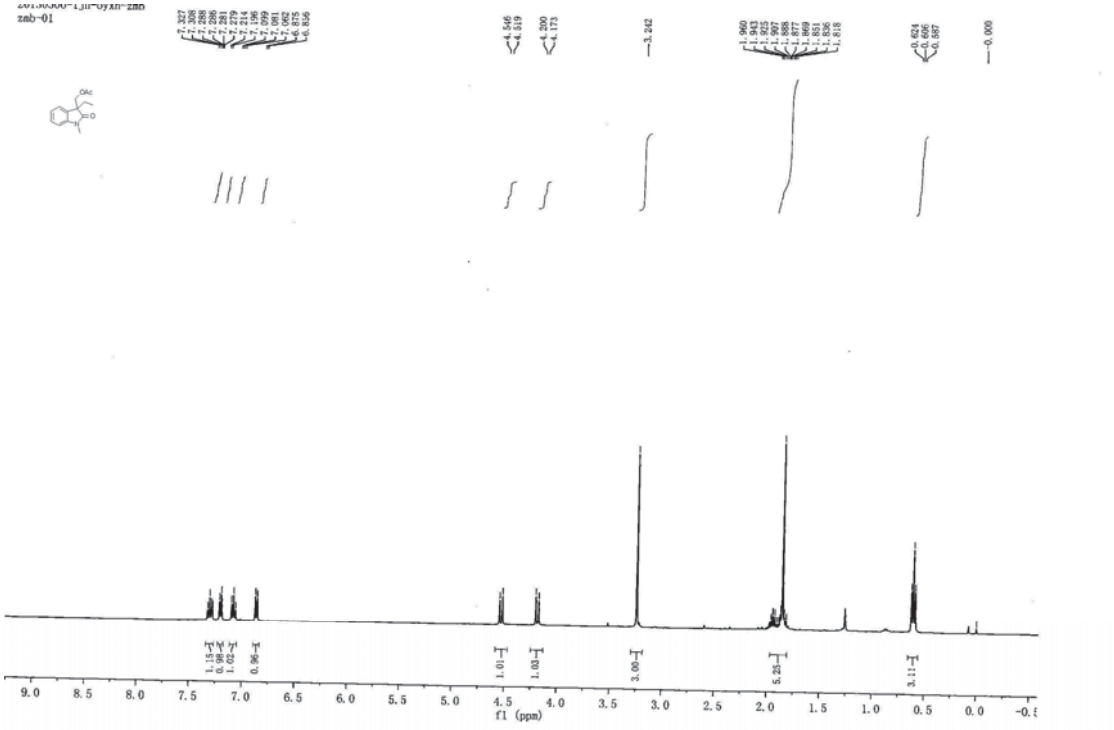


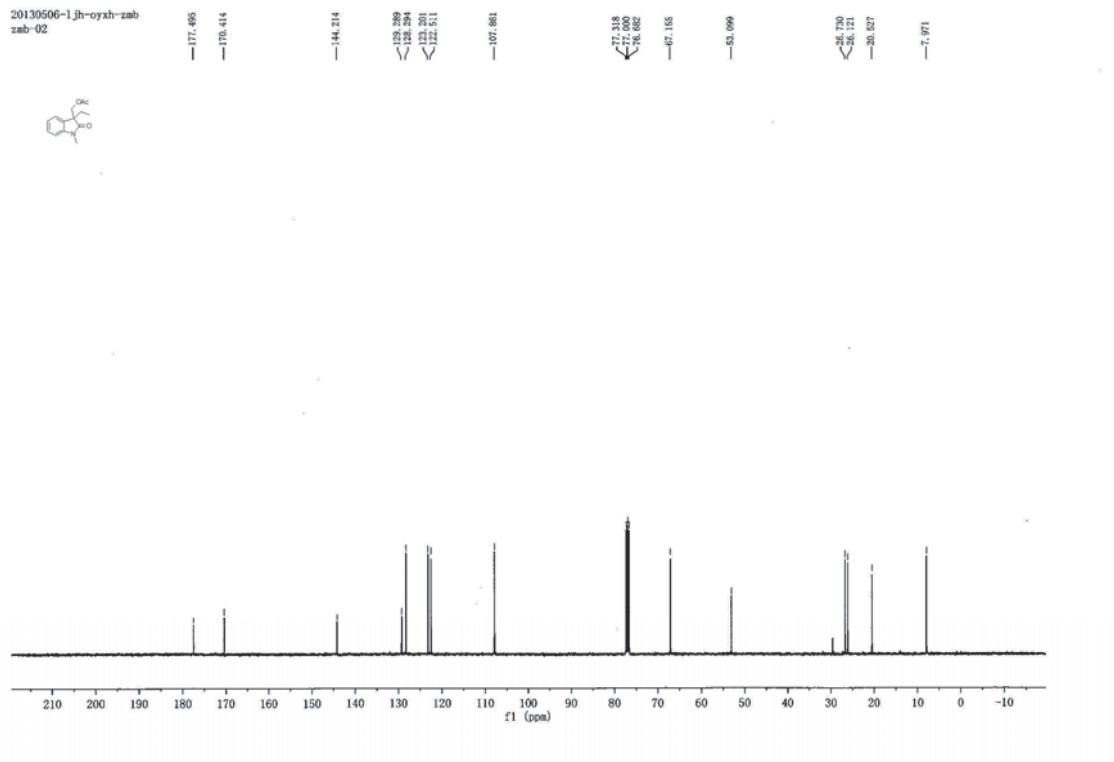
3-Ethyl-3-(hydroxymethyl)-1-methylindolin-2-one (3na)





(3-Ethyl-1-methyl-2-oxoindolin-3-yl)methyl acetate (3oa)





1-Methyl-3-(1-phenylethyl)indolin-2-one (3pa)