Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Visible light-induced aerobic oxidative cross-coupling reaction: preparation of α-indolyl glycine derivatives

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Supporting Information

A. General Information

Unless otherwise noted, all reagents were purchased from commercial sources (Adamas, Energy, Aldrich) and used as received without further purification. The liquid ^{1}H and ^{13}C NMR spectra were recorded on a Bruker-Avance DPX 400 MHz spectrometer using the solvent $CDCl_3$. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform δ 7.26), carbon (chloroform δ 77.16). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), td (doublet of triplet). Mass spectral data (MS) was recorded using an Agilent-6110 mass spectrometer. For thin layer chromatography (TLC), pre-coated Qingdao Haiyang TLC plates (GF254) were used, and compounds were visualized with a UV light at 254 nm. Flash chromatographic separations were performed on 200-300 mesh silica gel (from Qingdao Haiyang Chem. Company, Ltd.).

All amino acid derivatives[1] are known compounds and are prepared according to the reported methods. The final cross-coupling reaction was carried out in an open vial under ambient condition. A 30 W blue LED light bath was used and then covered with aluminum foil. Place a fan directly in front of the lamp (Figure 1).

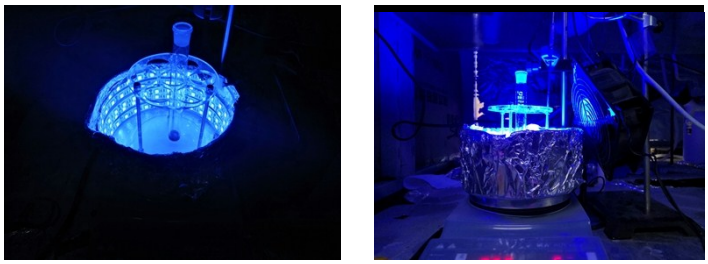
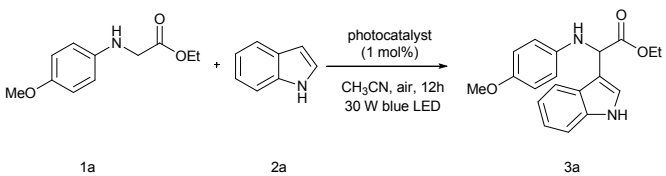


Figure 1

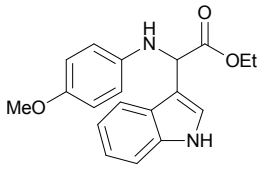
B. Visible light-induced oxidative cross-coupling reaction of glycine derivatives with indoles



1a 2a 3a

General procedure A: Glycine derivative **1a** (20.9 mg, 0.0999 mmol), Rose Bengal (1.0 mg, 1 mol%) and Indole **2a** (11.7 mg, 0.0999 mmol) were placed in a 10 mL reaction vessel. An acetonitrile solution (1.5 mL) was added to the vessel. The reaction mixture was irradiated with a 30 W blue LED bulb for 12 hours with stirring at room temperature. The solvent was removed under vacuum and the residue was subjected to flash chromatography on silica gel using petroleum ether/ethyl acetate (4:1) as an eluent to give **3a** as a brownish oil (26.31 mg, 81% yield.).

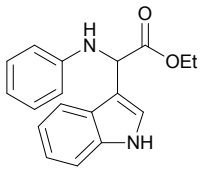
Ethyl 2-(1H-indol-3-yl)-2-[(4-methoxyphenyl)amino]acetate **3a**[2]



3a

A brownish oil. Following the general procedure A. 26.31 mg, 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$), δ 8.39 (s, 1H), 7.86 (d, *J* = 7.7 Hz, 1H), 7.31 (d, *J* = 7.7 Hz, 1H), 7.23 (td, *J* = 7.5, 1.2 Hz, 1H), 7.21–7.16 (m, 1H), 7.14 (d, *J* = 2.5 Hz, 1H), 6.81 (d, *J* = 8.9 Hz, 2H), 6.67 (d, *J* = 8.9 Hz, 2H), 5.36 (s, 1H), 4.52 (s, 1H), 4.27 (dq, J = 10.8, 7.1 Hz, 1H), 4.15 (dq, J = 10.8, 7.1 Hz, 1H), 3.75 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$), δ 173.03, 152.63, 140.90, 136.54, 125.87, 123.22, 122.50, 120.02, 119.52, 115.00, 114.96, 112.57, 111.55, 61.60, 55.80, 55.35, 14.21. MS (ESI) *m/z* calcd for $C_{19}H_{20}N_2O_3$ $(M\text{-}H)^-$ = 323.1, found = 323.1.

Ethyl 2-(1H-indol-3-yl)-2-(phenylamino)acetate **3b**[2]

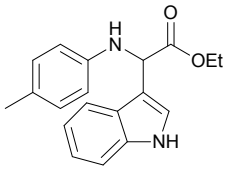


3b

A yellow oil. Following the general procedure A. 20.43 mg, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$), δ 8.20 (s, 1H), 7.86 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.36–7.32 (m, 1H), 7.29–7.20 (m, 1H), 7.22–7.14 (m, 4H), 6.79–6.73 (m, 1H), 6.67 (dt, *J* = 7.7, 1.1 Hz, 2H), 5.42 (s, 1H), 4.79 (s, 1H), 4.29 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.15 (dq, *J* = 10.8, 7.1 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$), δ 172.78,

146.66, 136.59, 129.36, 125.87, 123.26, 122.59, 120.10, 119.61, 118.22, 113.53, 112.51, 111.55, 61.72, 54.41, 14.23. MS (ESI) *m/z* calcd for $C_{18}H_{18}N_2O_2$ $(M\text{-}H)^-$ = 293.1, found = 293.1.

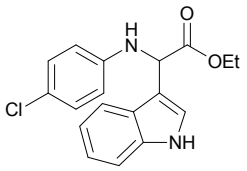
Ethyl 2-(1H-indol-3-yl)-2-(p-tolylamino)acetate **3c**[3]



3c

A dark yellow green oil. Following the general procedure A. 22.55 mg, 73% yield. ^{1}H NMR (400 MHz, $CDCl_3$) , δ 8.21 (s, 1H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.25–7.14 (m, 3H), 7.00 (d, *J* = 7.8 Hz, 2H), 6.60 (d, *J* = 8.4 Hz, 2H), 5.40 (s, 1H), 4.61 (s, 1H), 4.28 (dq, *J* = 10.7, 7.1 Hz, 1H), 4.15 (dq, *J* = 10.7, 7.1 Hz, 1H), 2.25 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.91, 144.41, 136.58, 129.86, 127.44, 125.92, 123.20, 122.56, 120.06, 119.63, 113.70, 112.71, 111.52, 61.64, 54.71, 20.52, 14.25. MS (ESI) *m/z* calcd for $C_{19}H_{20}N_2O_2$ $(M\text{-}H)^-$ = 307.1, found = 307.1.

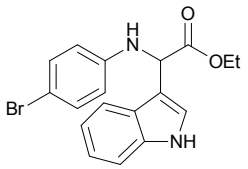
Ethyl 2-((4-chlorophenyl)amino)-2-(1H-indol-3-yl)acetate **3d**[3]



3d

A dark yellow green oil. Following the general procedure A. 21.05 mg, 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.85–7.78 (m, 1H), 7.37 (dt, *J* = 8.2, 1.0 Hz, 1H), 7.29–7.20 (m, 1H), 7.21–7.15 (m, 2H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 5.35 (d, *J* = 4.9 Hz, 1H), 4.82 (s, 1H), 4.27 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.14 (dq, *J* = 10.7, 7.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.39, 145.17, 136.63, 129.19, 125.84, 123.21, 122.80, 122.77, 120.26, 119.62, 114.62, 112.34, 111.57, 61.87, 54.44, 14.25. MS (ESI) *m/z* calcd for $C_{18}H_{17}{}^{35}ClN_2O_2$ $(M\text{-}H)^-$ = 327.1, found = 327.1, MS (ESI) m/z calcd for $C_{18}H_{17}{}^{37}ClN_2O_2(M\text{-}H)^-$ = 329.1, found 329.1.

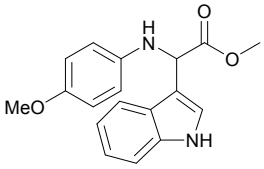
Ethyl 2-((4-bromophenyl)amino)-2-(1H-indol-3-yl)acetate **3e**[4]



3e

A yellow oil. Following the general procedure A. 22.12 mg, 59% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27–8.18 (m, 1H), 7.85–7.78 (m, 1H), 7.37 (dt, *J* = 8.1, 1.0 Hz, 1H), 7.29–7.14 (m, 5H), 6.51 (d, *J* = 8.8 Hz, 2H), 5.35 (d, *J* = 4.4 Hz, 1H), 4.84 (s, 1H), 4.27 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.14 (dq, *J* = 10.7, 7.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.36, 145.56, 136.61, 132.04, 125.80, 123.22, 122.75, 120.24, 119.58, 115.09, 112.19, 111.58, 109.85, 61.88, 54.30, 29.82, 14.24. MS (ESI) *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2O_2$ $(M-H)^-$ = 371.0, found = 371.0. MS (ESI) *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2O_2$ $(M-H)^-$ = 373.0, found = 373.0

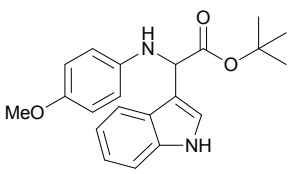
Methyl 2-(1H-indol-3-yl)-2-((4-methoxyphenyl)amino)acetate **3f**



3f

A yellow oil. Following the general procedure A. 26.48 mg, 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (s, 1H), 7.83 (d, *J* = 7.9 Hz, 1H), 7.33 (s, 1H), 7.25–7.16 (m, 2H), 6.77 (d, *J* = 8.9 Hz, 2H), 6.63 (d, *J* = 8.9 Hz, 2H), 5.36 (s, 1H), 4.48 (s, 1H), 3.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.49, 152.71, 140.85, 136.55, 125.88, 123.21, 122.65, 120.21, 119.47, 115.01, 114.98, 112.68, 111.57, 55.83, 55.20, 52.60. IR (KBr) ν_{max} 3396, 3360, 2918, 1730, 1621, 1514, 1457, 1362, 1340. HRMS (ESI) *m/z* calcd for $C_{18}H_{18}N_2O_3$ $(M-H)^-$ = 309.1239, found = 309.1237.

Tert-butyl 2-(1H-indol-3-yl)-2-((4-methoxyphenyl)amino)acetate **3g**

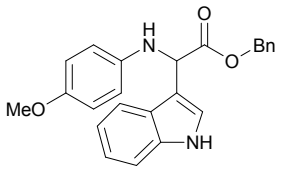


3g

A brownish oil. Following the general procedure A. 27.95 mg, 79% yield. ^{1}H NMR (400 MHz,

$CDCl_3$) δ 8.25 (s, 1H), 7.86–7.82 (m, 1H), 7.34 (dt, *J* = 8.2, 1.0 Hz, 1H), 7.25–7.14 (m, 3H), 6.76 (d, *J* = 9.0 Hz, 2H), 6.62 (d, *J* = 8.9 Hz, 2H), 5.25 (d, *J* = 0.8 Hz, 1H), 4.50 (s, 1H), 3.73 (s, 3H), 1.41 (s, 9H). IR (KBr) ν_{max} 3648, 3613, 2980, 1732, 1614, 1510, 1456, 1395, 1367, 1339, 1319. ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.13, 152.51, 141.12, 136.64, 126.10, 122.98, 122.43, 119.84, 119.82, 114.96, 114.94, 113.25, 111.44, 82.14, 55.91, 55.86, 28.07. HRMS (ESI) *m/z* calcd for $C_{21}H_{24}N_2O_3Na$ $(M+Na)^+$ = 375.1685, found = 365.1676.

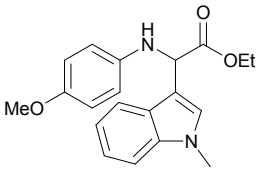
Benzyl 2-(1H-indol-3-yl)-2-(4-methoxyphenylamino)acetate **3h**[5]



3h

A dark red oil. Following the general procedure A. 29.15 mg, 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.84–7.78 (m, 1H), 7.36–7.33 (m, 1H), 7.29 (dd, *J* = 5.0, 2.0 Hz, 3H), 7.22 (td, *J* = 9.5, 2.4 Hz, 3H), 7.18–7.12 (m, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.63 (d, *J* = 8.8 Hz, 2H), 5.42 (s, 1H), 5.25 (d, *J* = 12.4 Hz, 1H), 5.10 (d, *J* = 12.3 Hz, 1H), 4.50 (s, 1H), 3.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.86, 152.74, 140.84, 136.54, 128.56, 128.34, 128.27, 123.19, 122.66, 120.17, 119.64, 115.06, 115.00, 112.57, 111.51, 67.21, 55.85, 55.44. MS (ESI) *m/z* calcd for $C_{24}H_{22}N_2O_3$ $(M-H)^-$ = 385.1, found = 385.1.

Ethyl 2-[(4-methoxyphenyl)amino]-2-(1-methyl-1H-indol-3-yl)acetate **3j**[2]

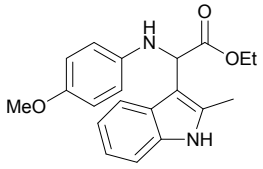


3j

A yellow oil. Following the general procedure A. 26.43 mg, 78% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 1H), 7.33 (s, 2H), 7.20 (s, 1H), 7.14 (s, 1H), 6.78 (d, *J* = 8.9 Hz, 2H), 6.65 (d, *J* = 8.9 Hz, 2H), 5.35 (s, 1H), 4.53 (s, 1H), 4.29 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.15 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.75 (s, 6H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.97, 152.59, 140.97, 137.37, 127.64, 126.43, 122.13, 119.75, 119.63, 114.93, 114.88, 111.24, 109.57, 61.54, 55.80, 55.24, 32.95,

14.26. MS (ESI) *m/z* calcd for $C_{20}H_{22}N_2O_3$ $(M-H)^-$ = 337.1, found = 337.3.

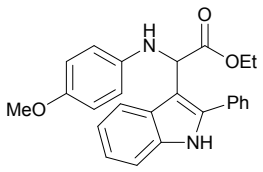
(S)-Ethyl 2-(4-Methoxyphenylamino)-2-(2-methyl-1H-indol-3-yl)acetate **3k**[6]



3k

A yellow oil. Following the general procedure A. 23.84 mg, 70% yield. ^{1}H NMR (400 MHz, CDCl3) δ 7.97 (s, 1H), 7.82 (dd, J = 6.3, 2.6 Hz, 1H), 7.27 (dd, J = 6.0, 2.2 Hz, 1H), 7.18–7.11 (m, 2H), 6.63 (d, *J* = 8.9 Hz, 2H), 6.39 (d, *J* = 8.9 Hz, 2H), 5.26 (s, 1H), 4.56 (s, 1H), 4.26 (dq, J = 10.8, 7.1 Hz, 1H), 4.10 (dq, J = 10.8, 7.1 Hz, 1H), 3.74 (s, 3H), 2.49 (s, 3H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.71, 152.54, 141.12, 135.26, 133.35, 127.02, 121.53, 120.01, 118.99, 114.99, 114.77, 110.54, 107.98, 61.47, 55.87, 55.10, 14.28, 12.27. MS (ESI) *m/z* calcd for $C_{20}H_{22}N_2O_3$ $(M-H)^-$ = 337.1, found = 337.1.

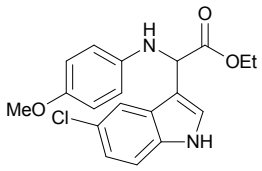
Ethyl 2-((4-methoxyphenyl)amino)-2-(2-phenyl-1H-indol-3-yl)acetate **3l**



3l

A yellow solid. Following the general procedure A. 30.21 mg, 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.73–7.69 (m, 2H), 7.52 (t, *J* = 7.2 Hz, 2H), 7.45 (s, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.22 (s, 1H), 7.16 (s, 1H), 6.63 (d, *J* = 8.9 Hz, 2H), 6.39 (d, *J* = 8.9 Hz, 2H), 5.39 (s, 1H), 4.62 (s, 1H), 4.28 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.15 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.67 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). IR (KBr) ν_{max} 3735, 3725, 3400, 2979, 1732, 1682, 1514, 1489, 1456, 1362, 1339, 817, 746, 698. ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.84, 152.39, 140.74, 137.51, 136.03, 132.27, 129.07, 128.98, 128.69, 126.76, 122.67, 120.45, 120.31, 114.82, 114.79, 111.20, 108.79, 61.65, 55.82, 54.91, 14.26. HRMS (ESI) *m/z* calcd for $C_{25}H_{24}N_2O_3Na$ $(M+Na)^+$= 423.1685, found = 423.1678.

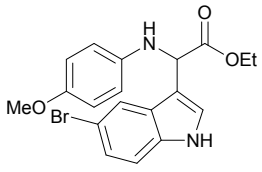
(S)-Ethyl 2-(5-Chloro-1H-indol-3-yl)-2-(4-methoxyphenylamino)acetate **3m**[6]



3m

A dark red oil. Following the general procedure A. 24.84 mg, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.82 (d, *J* = 1.8 Hz, 1H), 7.22 (d, *J* = 8.6 Hz, 1H), 7.19–7.14 (m, 2H), 6.76 (d, *J* = 8.8 Hz, 2H), 6.61 (d, *J* = 8.8 Hz, 2H), 5.27 (s, 1H), 4.65–4.35 (m, 1H), 4.26 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.15 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.73 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.61, 152.65, 140.57, 134.88, 126.86, 125.75, 124.49, 122.83, 119.11, 114.98, 114.90, 112.49, 61.75, 55.75, 55.13, 14.14. MS (ESI) *m/z* calcd for $C_{19}H_{19}{}^{35}ClN_2O_3$ $(M-H)^-$ = 357.1, found = 357.0. MS (ESI) *m/z* calcd for $C_{19}H_{19}{}^{37}ClN_2O_3$ $(M-H)^-$ = 359.1, found = 359.0

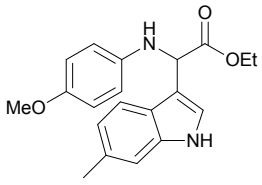
(S)-Ethyl 2-(5-Bromo-1H-indol-3-yl)-2-(4-methoxyphenylamino)acetate **3n**[6]



3n

A dark red oil. Following the general procedure A. 26.25 mg, 65% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.98 (d, *J* = 1.8 Hz, 1H), 7.29 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.19–7.15 (m, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.64 (d, *J* = 8.8 Hz, 2H), 5.27 (s, 1H), 4.49 (s, 1H), 4.24 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.16 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.73 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.64, 152.76, 140.65, 135.25, 127.61, 125.47, 124.39, 122.32, 115.08, 115.00, 113.40, 112.98, 112.57, 61.83, 55.84, 55.22, 14.22. MS (ESI) *m/z* calcd for $C_{19}H_{19}{}^{79}BrN_2O_3$ $(M-H)^-$ = 401.0, found = 401.0. MS (ESI) *m/z* calcd for $C_{19}H_{19}{}^{81}BrN_2O_3$ $(M-H)^-$ = 403.0, found = 403.0.

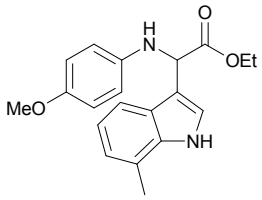
(S)-Ethyl 2-(4-Methoxyphenylamino)-2-(6-methyl-1H-indol-3-yl)acetate **3o**[6]



3o

A yellow oil. Following the general procedure A. 24.47 mg, 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.12–7.07 (m, 2H), 7.02 (d, *J* = 8.3 Hz, 1H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 5.32 (s, 1H), 4.50 (s, 1H), 4.27 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.14 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.74 (s, 3H), 2.47 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.02, 152.61, 140.95, 137.03, 132.38, 123.73, 122.56, 121.82, 119.21, 114.97, 114.95, 112.49, 111.46, 61.56, 55.81, 55.43, 21.75, 14.24. MS (ESI) *m/z* calcd for $C_{20}H_{22}N_2O_3$ $(M-H)^-$ = 337.1, found = 337.2.

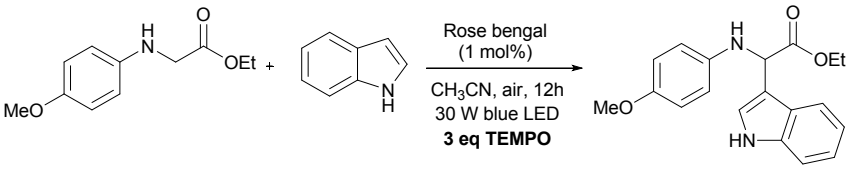
(S)-Ethyl 2-(4-Methoxyphenylamino)-2-(7-methyl-1H-indol-3-yl)acetate **3p**[6]



3p

A dark red oil. Following the general procedure A. 25.82 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.23 (d, *J* = 2.6 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 1H), 7.04 (d, *J* = 7.1 Hz, 1H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 5.33 (s, 1H), 4.26 (dq, *J* = 10.8, 7.1Hz, 1H), 4.13 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.72 (s, 3H), 2.48 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.93, 152.64, 140.96, 136.20, 125.56, 123.15, 122.83, 120.65, 120.37, 117.41, 114.98, 114.96, 113.49, 61.60, 55.86, 55.44, 16.67, 14.29. MS (ESI) *m/z* calcd for $C_{20}H_{22}N_2O_3$ $(M-H)^-$ = 337.1 , found = 337.2.

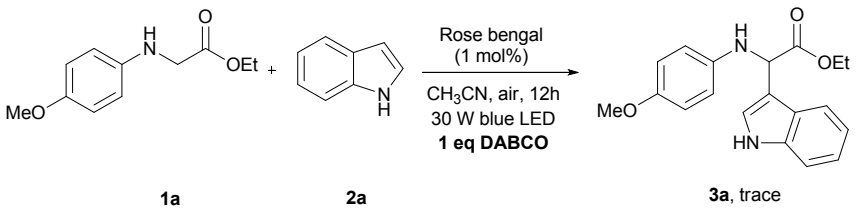
C. Control experiments



1a **2a** **3a,** trace

General procedure B: Glycine derivative 1a (0.1 mmol), Rose Bengal (1 mol%), Indole 2a (0.1 mmol) and a radical scavenger 3 eq 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) were placed in a 10 mL reaction vessel. An acetonitrile solution (1.5 mL) was added to the vessel. The reaction mixture was irradiated with a 30 W blue LED bulb for 12 hours with stirring at room temperature, and only

traces of product were observed.

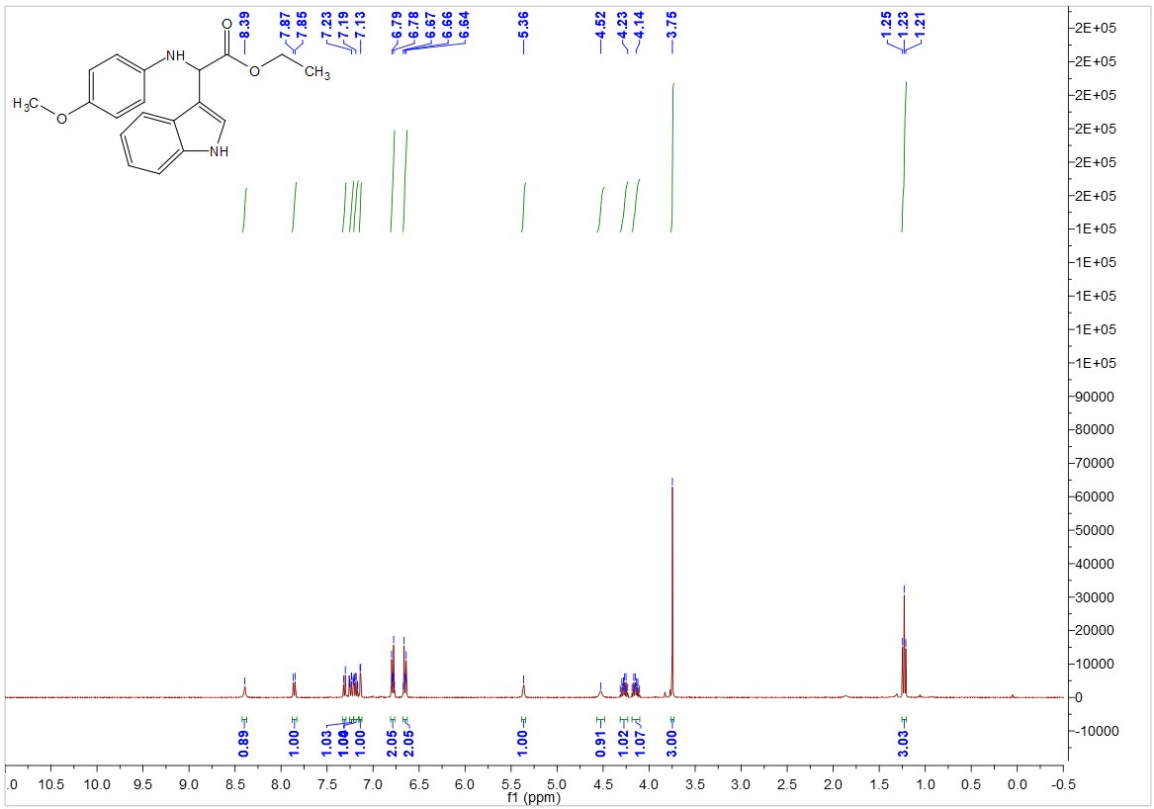


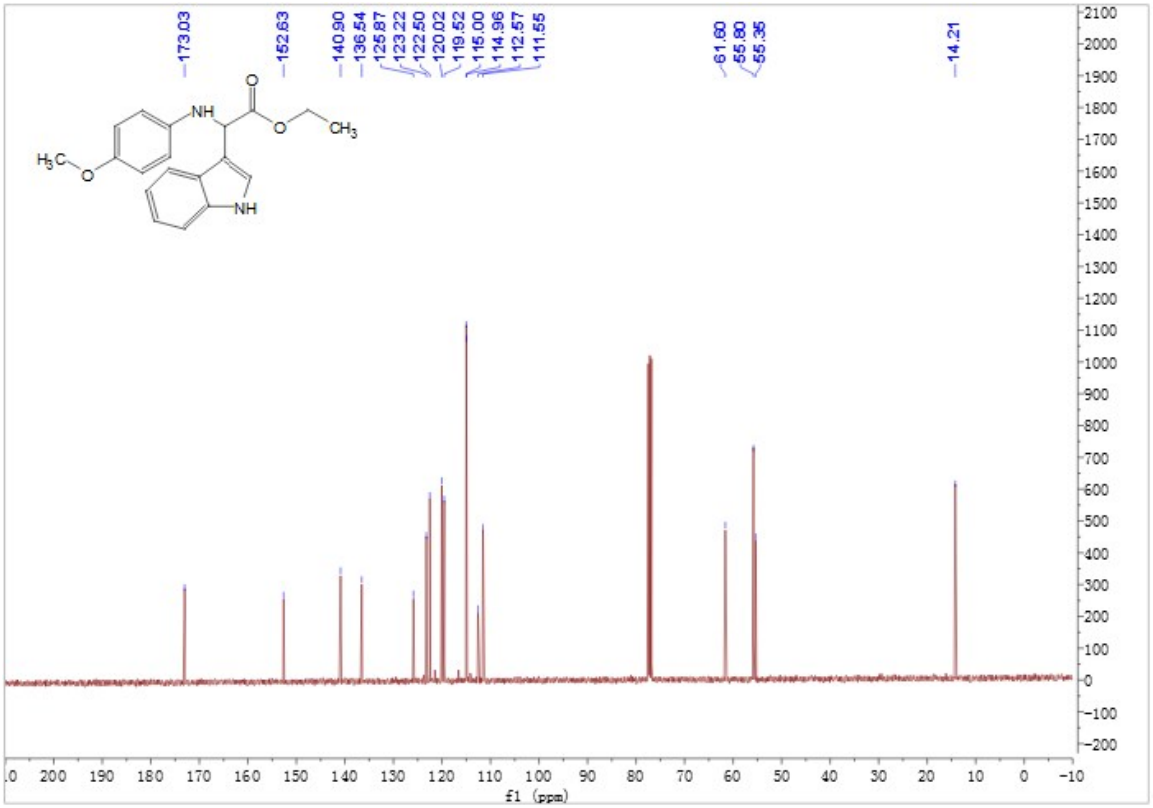
General procedure C: Glycine derivative 1a (0.1 mmol), Rose Bengal (1 mol%), Indole 2a (0.1 mmol) and a single oxygen quencher 1 eq 1,4-Diazabicyclo[2.2.2]octane (DABCO) were placed in a 10 mL reaction vessel. An acetonitrile solution (1.5 mL) was added to the vessel. The reaction mixture was irradiated with a 30 W blue LED bulb for 12 hours with stirring at room temperature, and only traces of product were observed.

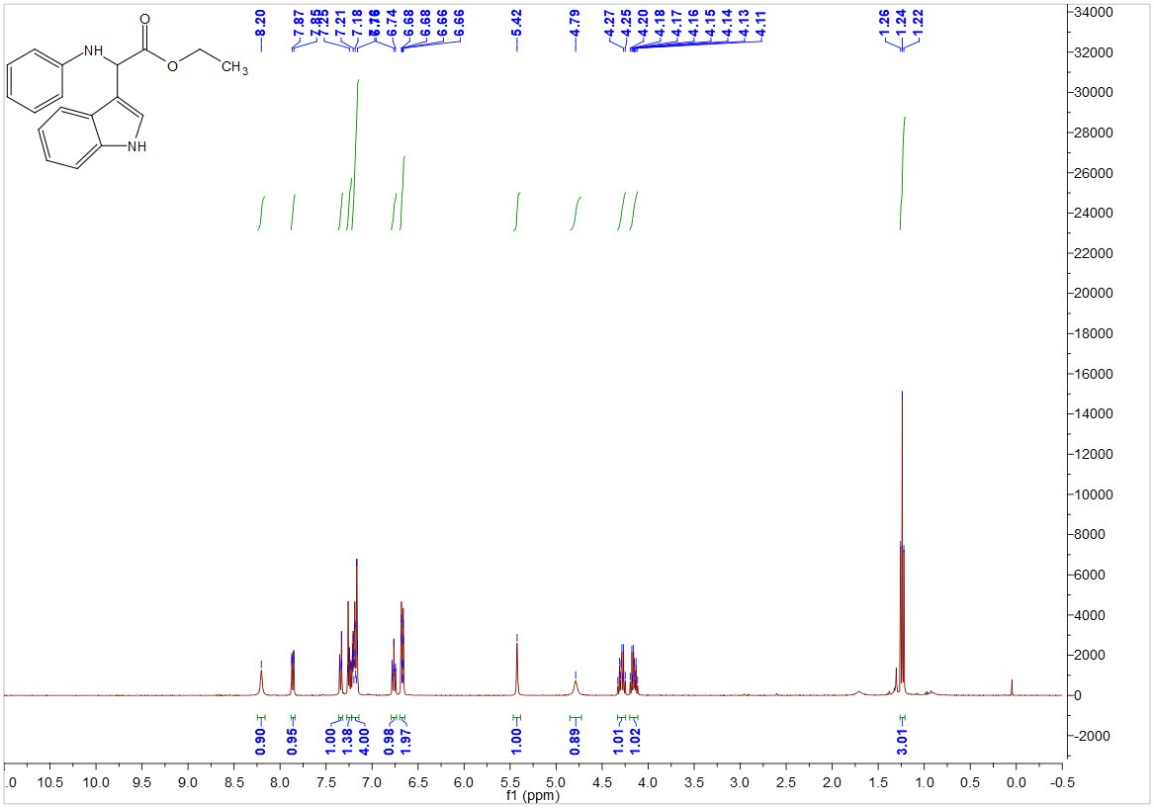
D. References

1. M. Laurent, M. Cérésiat and J. Marchand-Brynaert. *Eur. J. Org. Chem*., **2006**, *16*, 3755–3766.
2. M. S. Segundo, I. Guerrero and A. Correa. *Org. Lett*., **2017**, *19*, 5288–5291.
3. C. Huo, C. Wang, M. X. Wu, X. D. Jia, H. X. Xie and Y. Yuan. *Adv. Synth. Catal*., **2014**, *356*, 411–415.
4. C. Huo, Y. Yuan, M. Wu, X. Jia, X. Wang, F. Chen and J. Tang. *Angew. Chem. Int. Ed*., **2014**, 53, 1–5.
5. Q. Kang, Z.-An. Zhao and S.-Li. You. *Tetrahedron*, **2009**, 65, 1603–1607.
6. X. W. Wang, Y. Z. Hua and M. C. Wang. *J. Org. Chem*., **2016**, *81*, 9227−9234.

E. ^{1}H and ^{13}C NMR Spectra



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4.20
4.18
4.17
4.16
4.15
4.14
4.13
4.11
1.26
1.24
1.22
0.90
0.95
1.00
1.38
4.00
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1.97
1.00
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1.01
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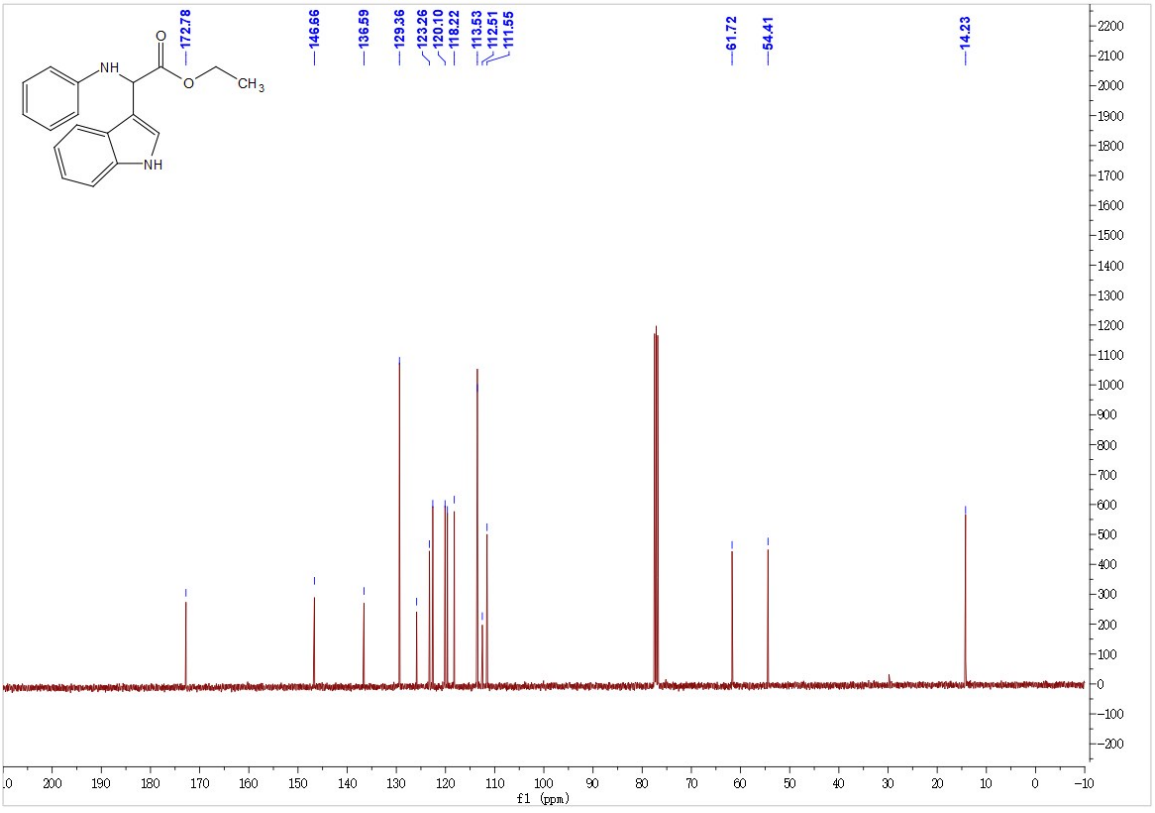
3.01
f1 (ppm)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61.72
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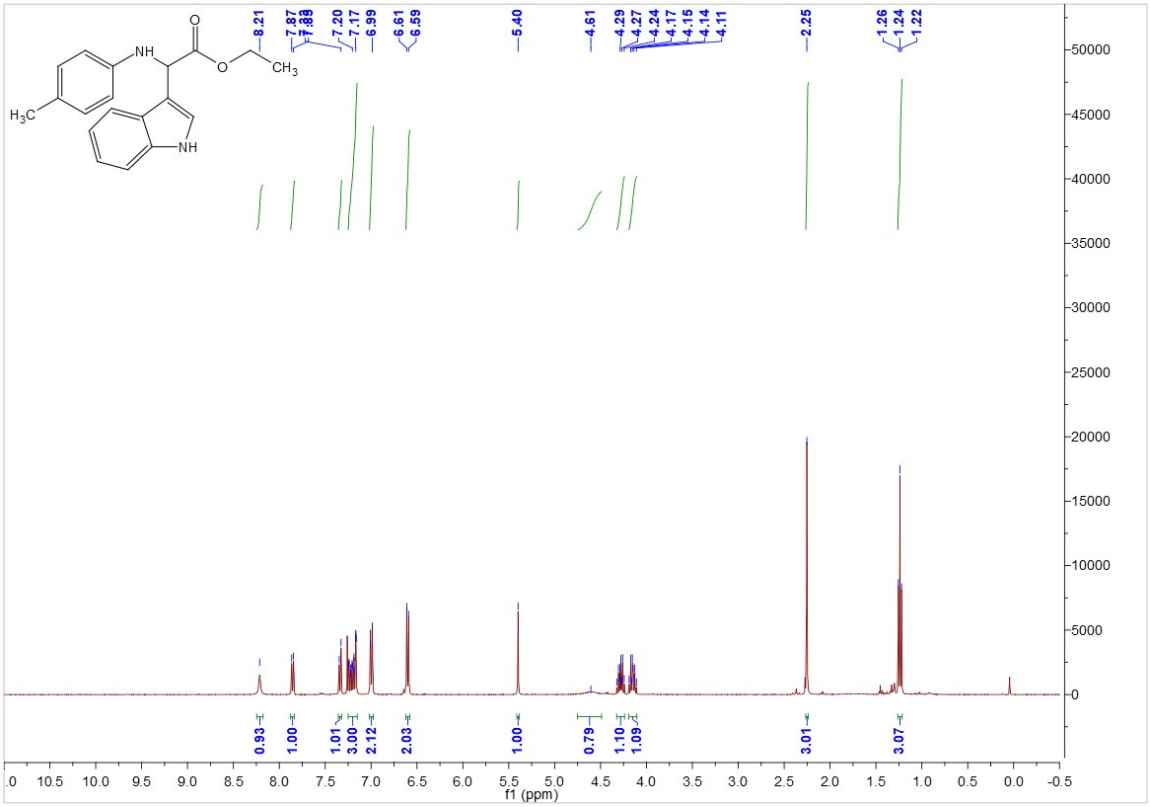
14.23
f1 (ppm)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4.14
4.11
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1.26
1.24
1.22
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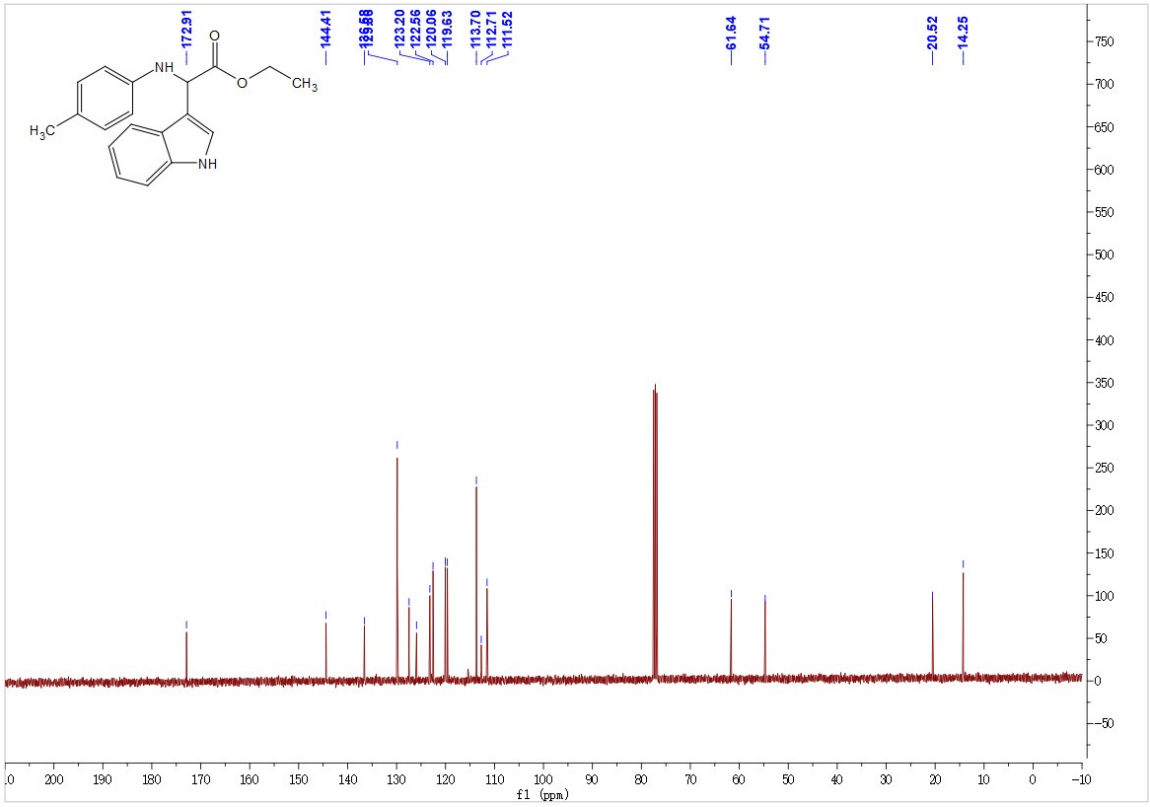
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112.71
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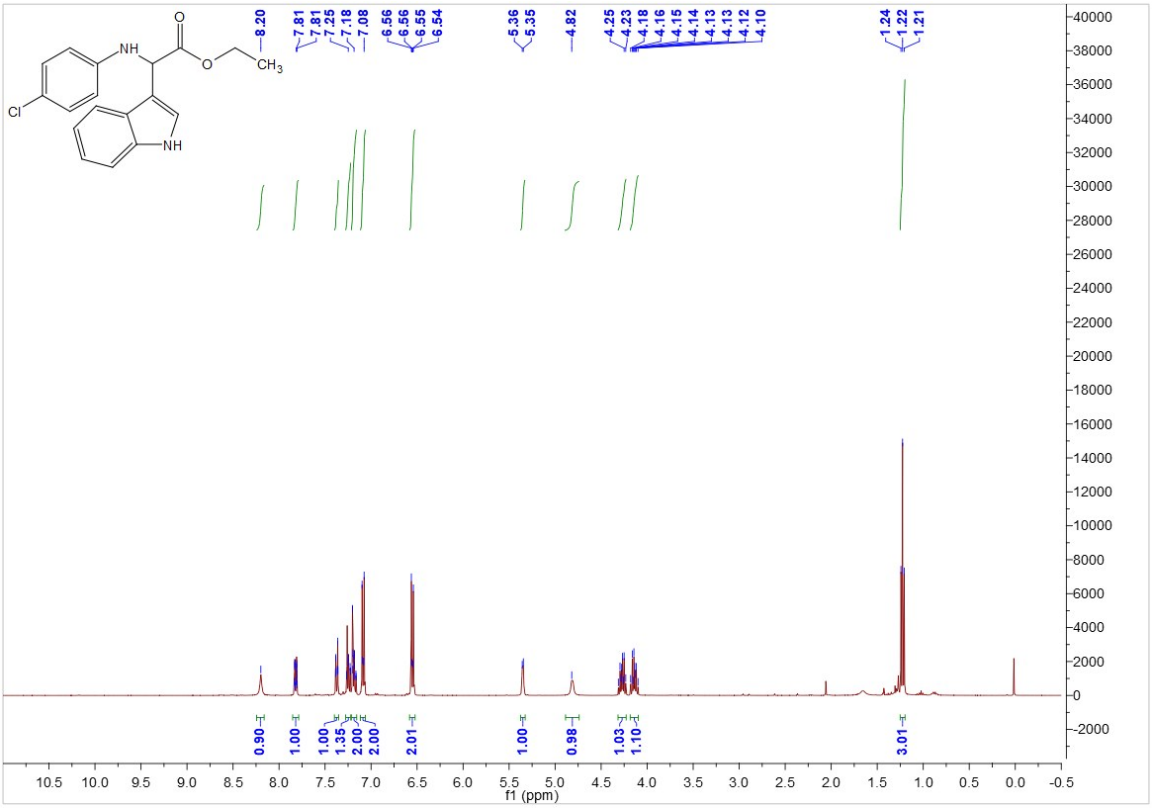
54.71
20.52
14.25
f1 (ppm)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4.23
4.18
4.16
4.15
4.14
4.13
4.13
4.12
4.10
1.24
1.22
1.21
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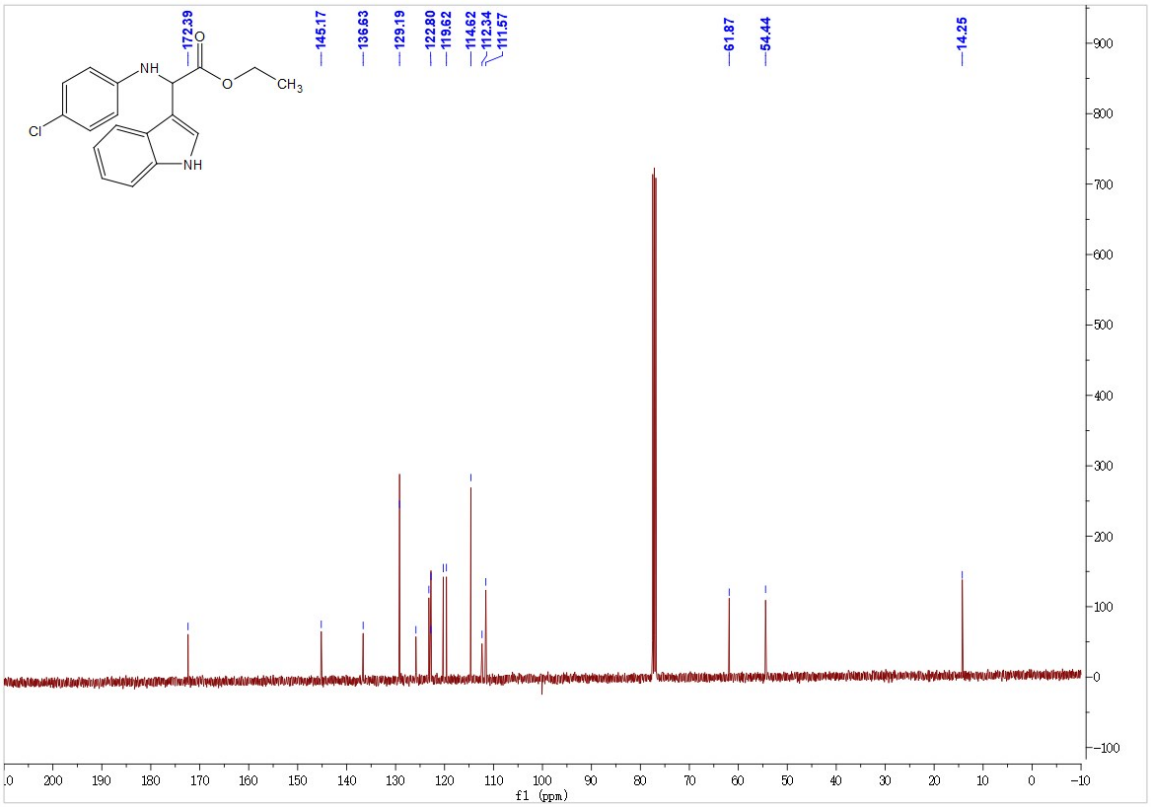
3.01
f1 (ppm)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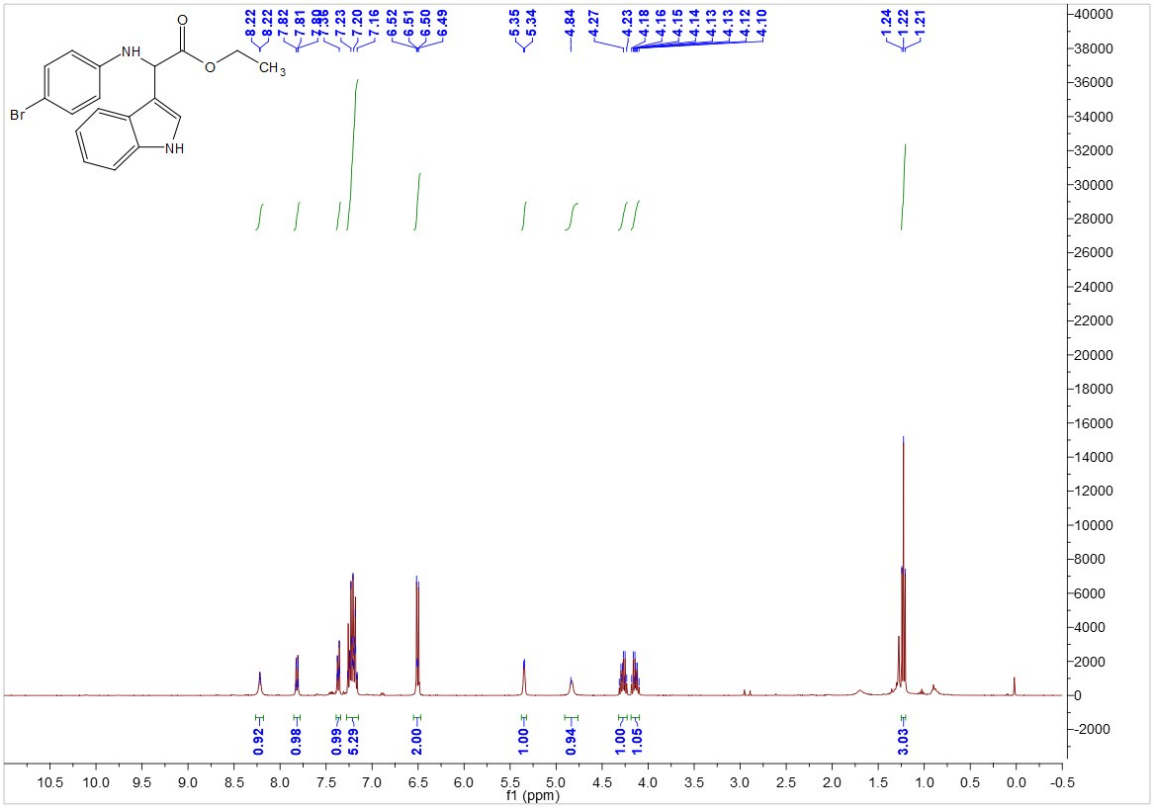
14.25
f1 (ppm)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4.18
4.16
4.15
4.14
4.13
4.13
4.12
4.10
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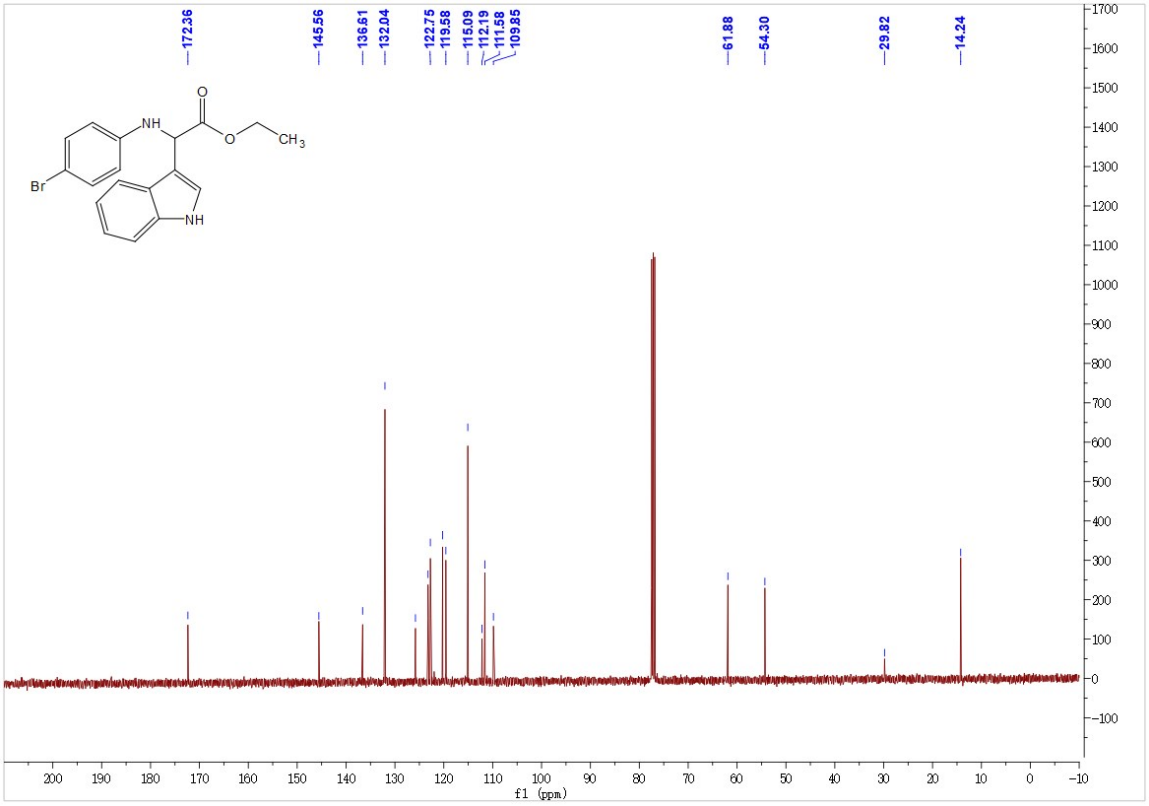
f1 (ppm)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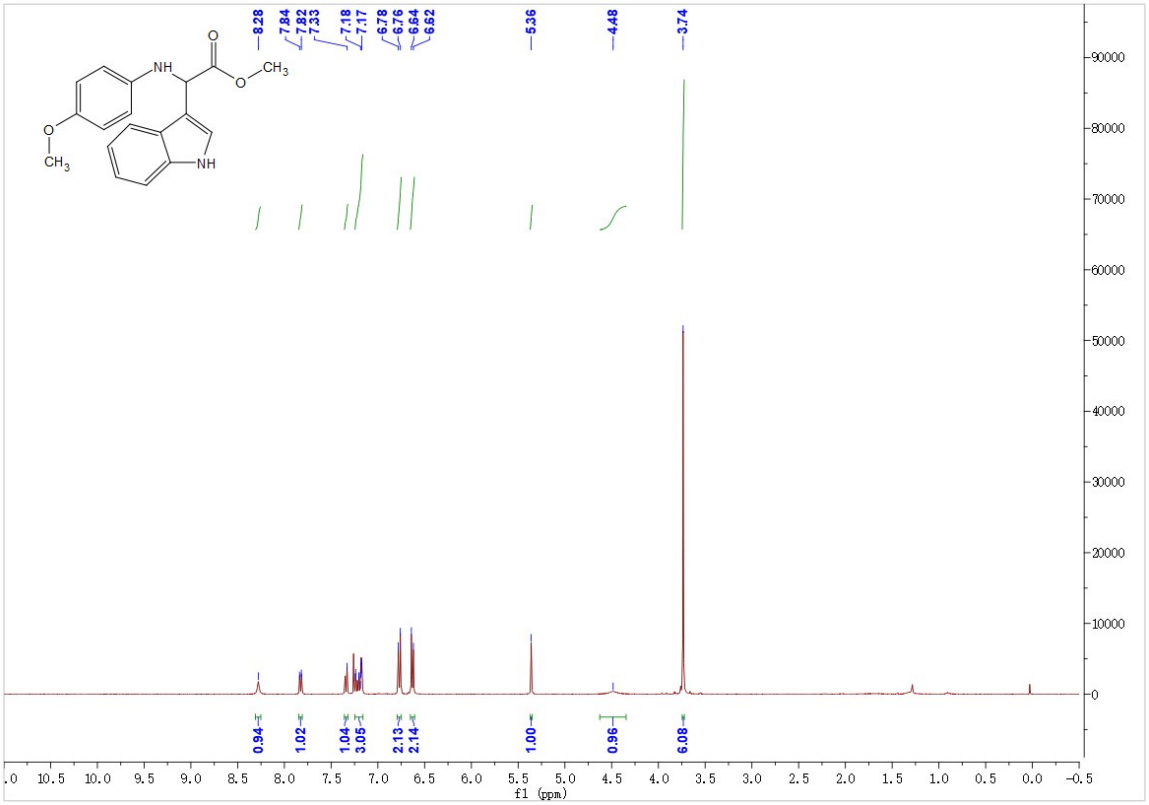
14.24
f1 (ppm)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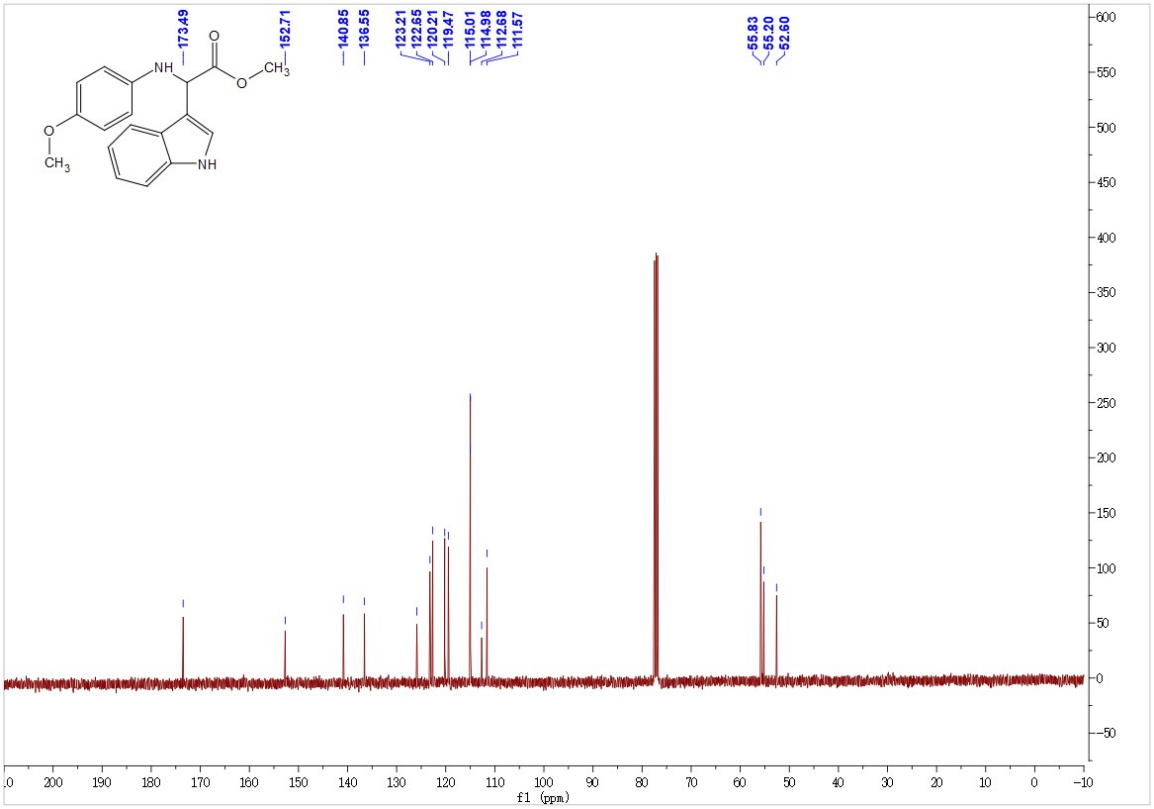
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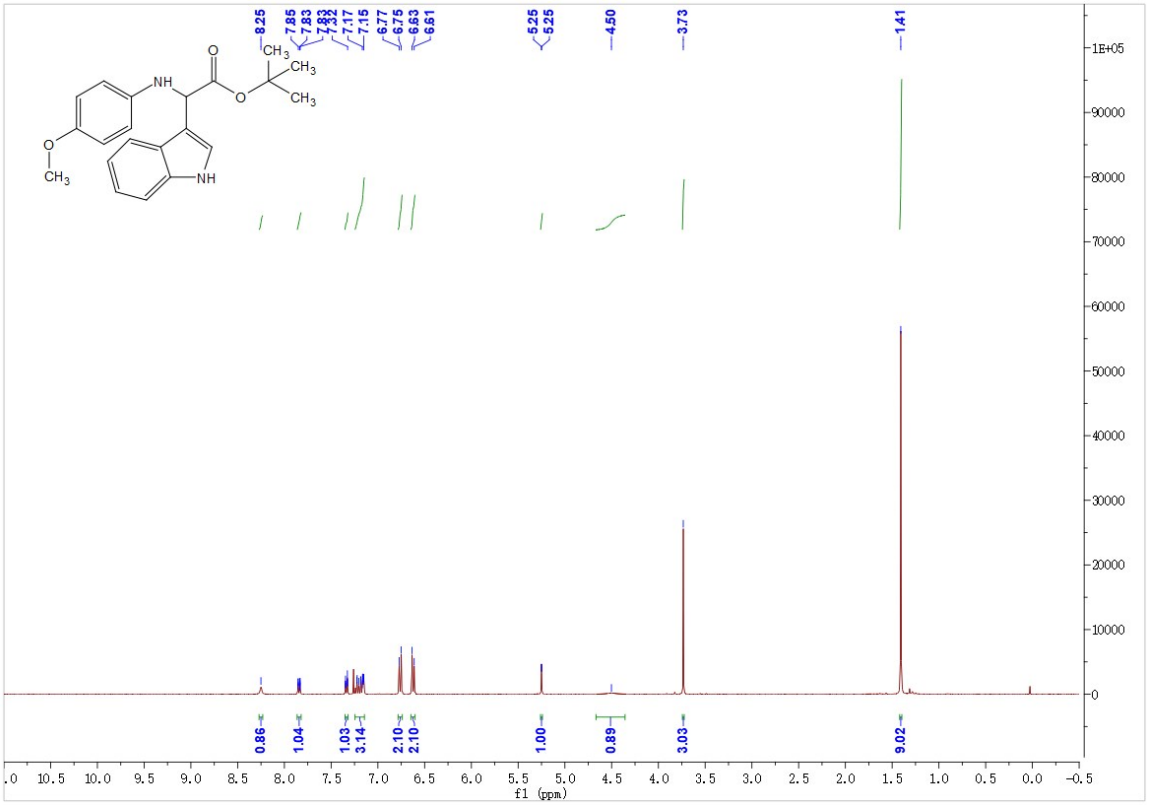
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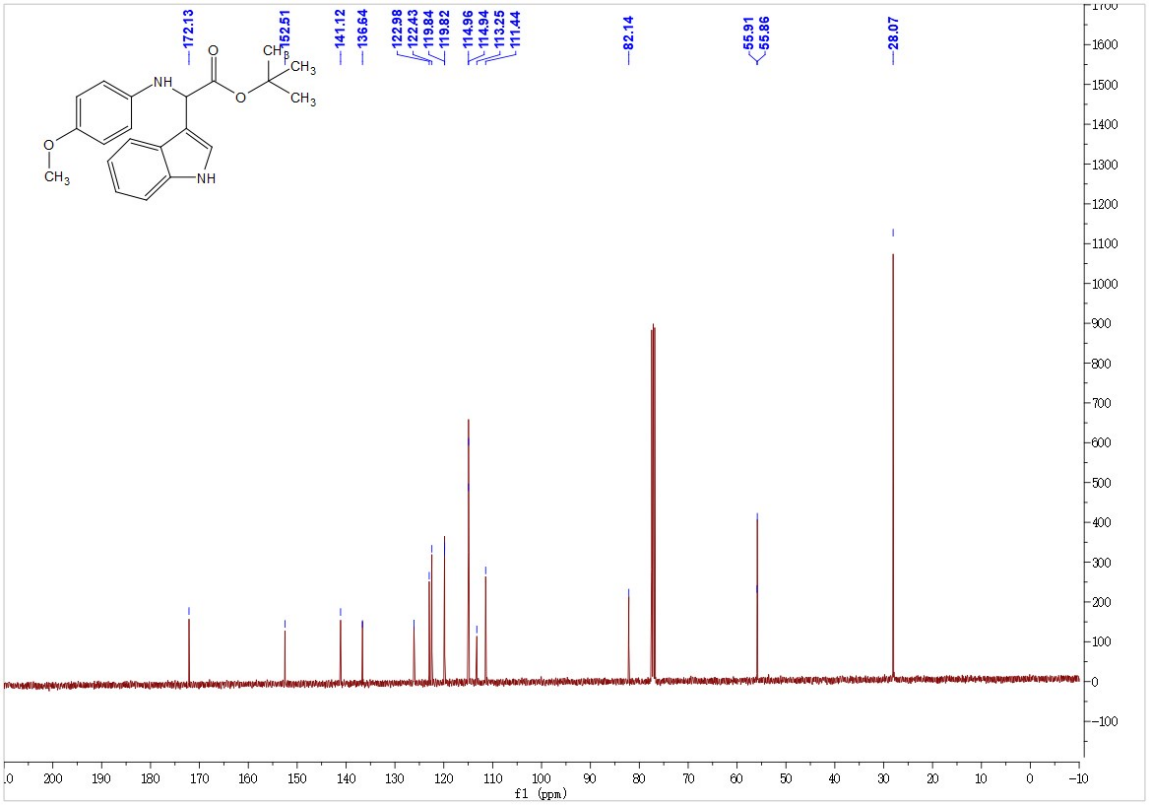
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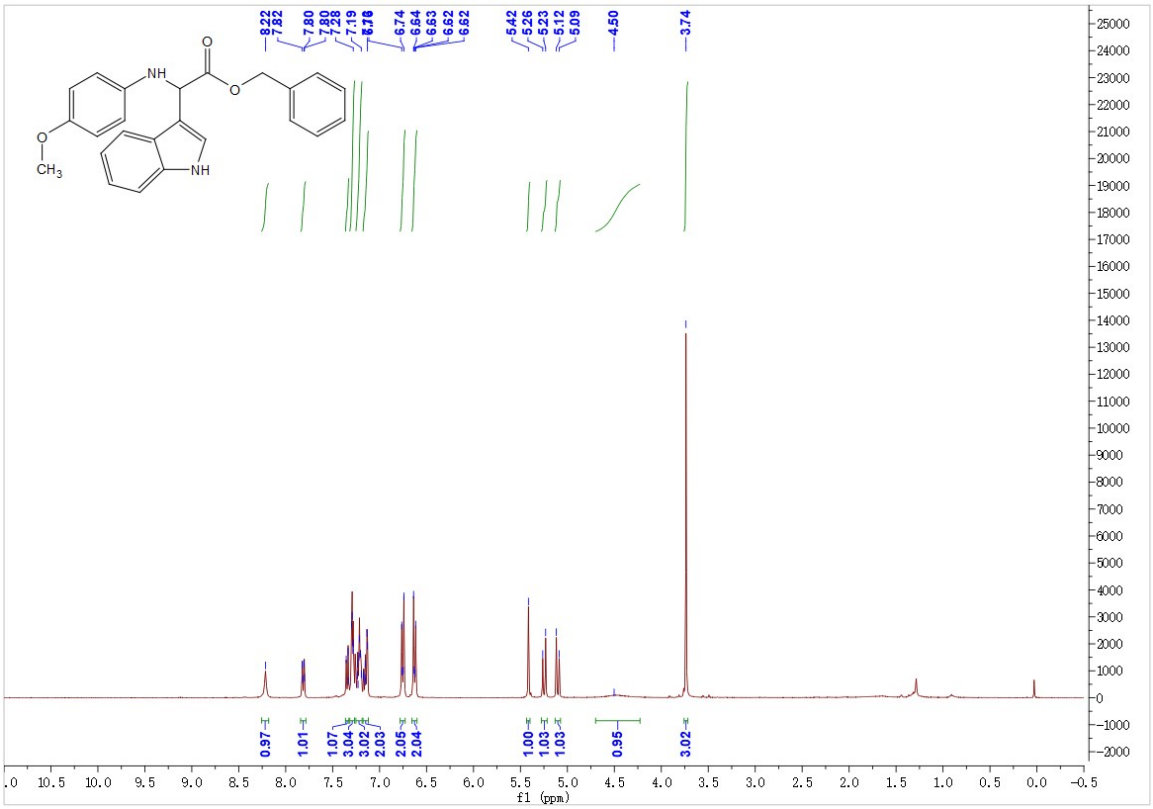
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O
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NH
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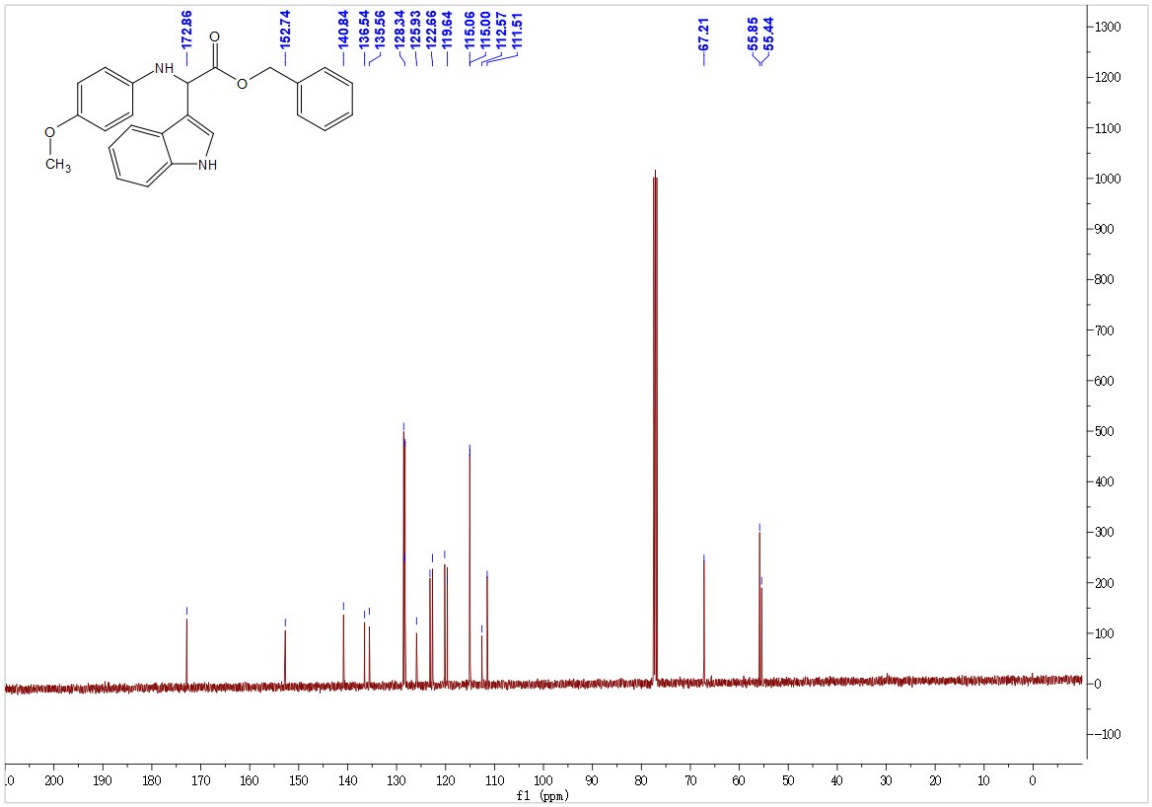
f1 (ppm)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NH
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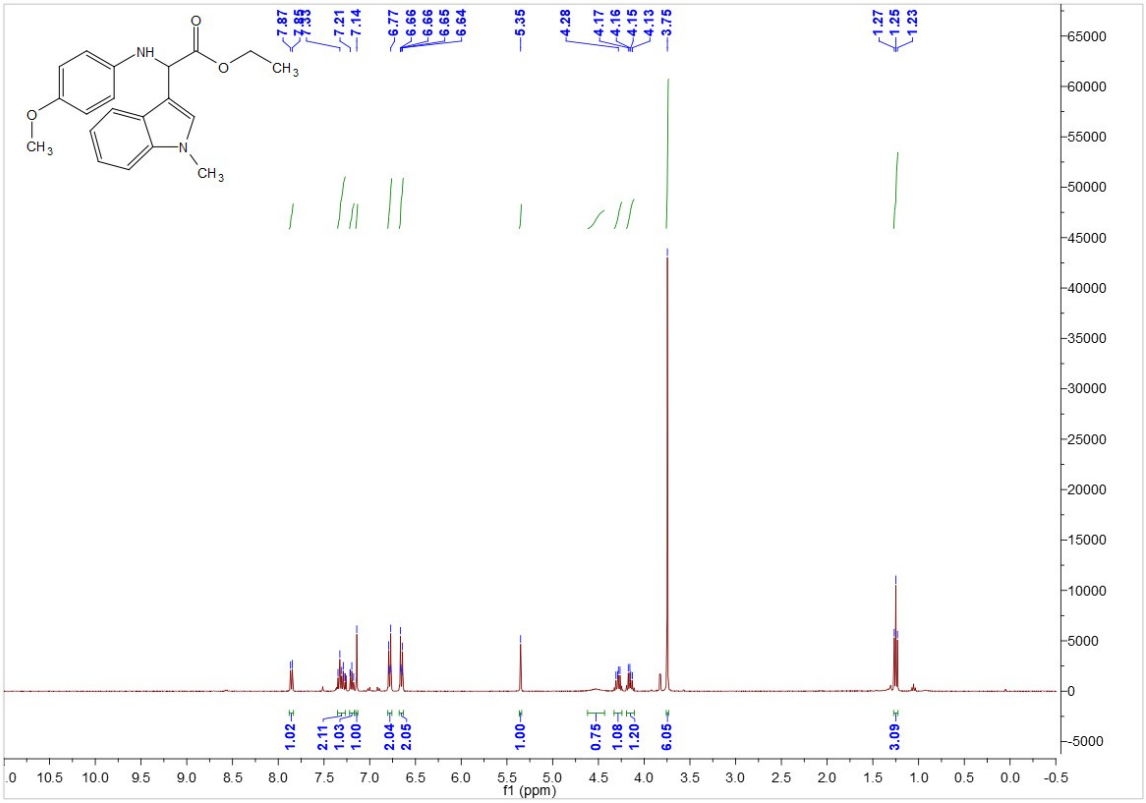
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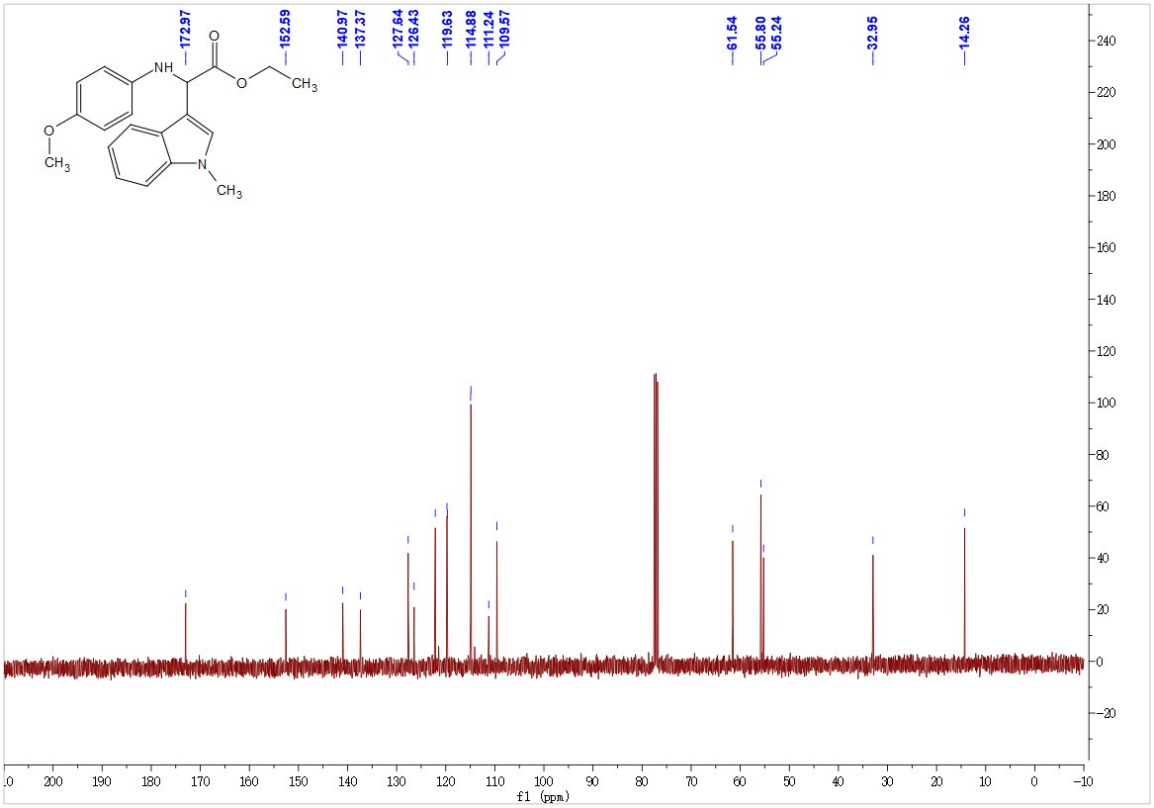
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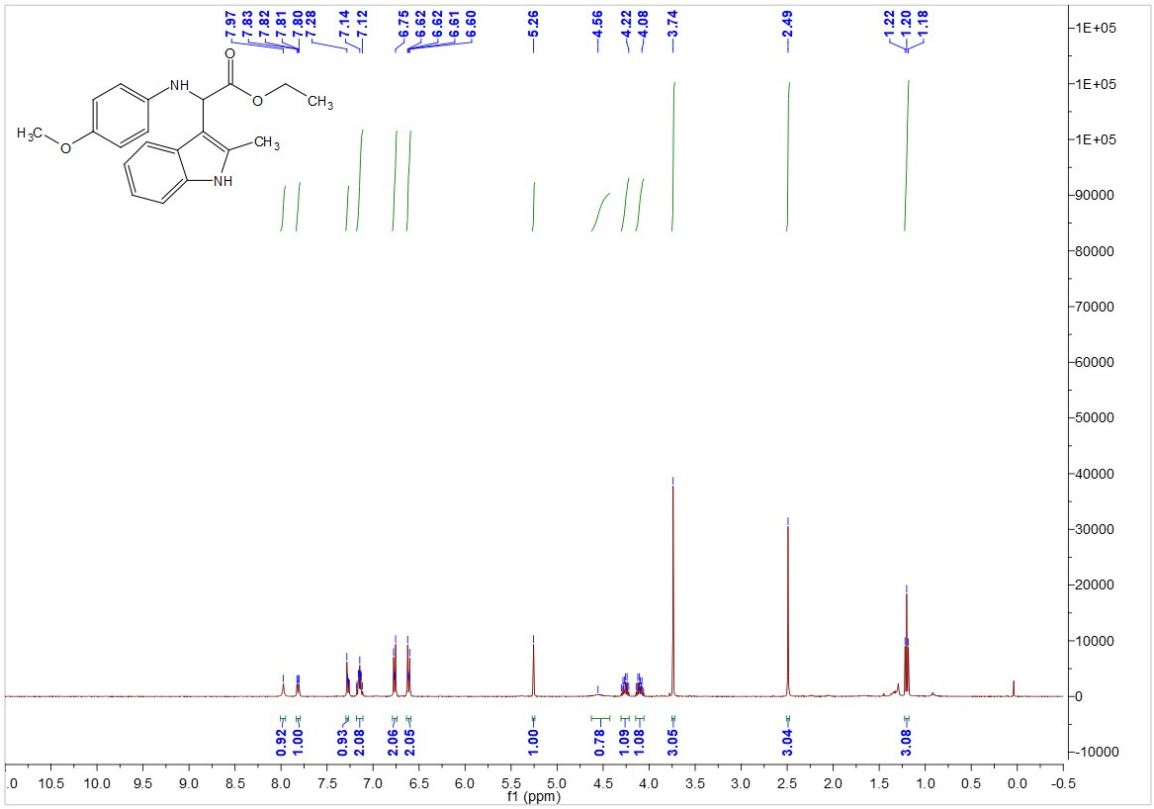
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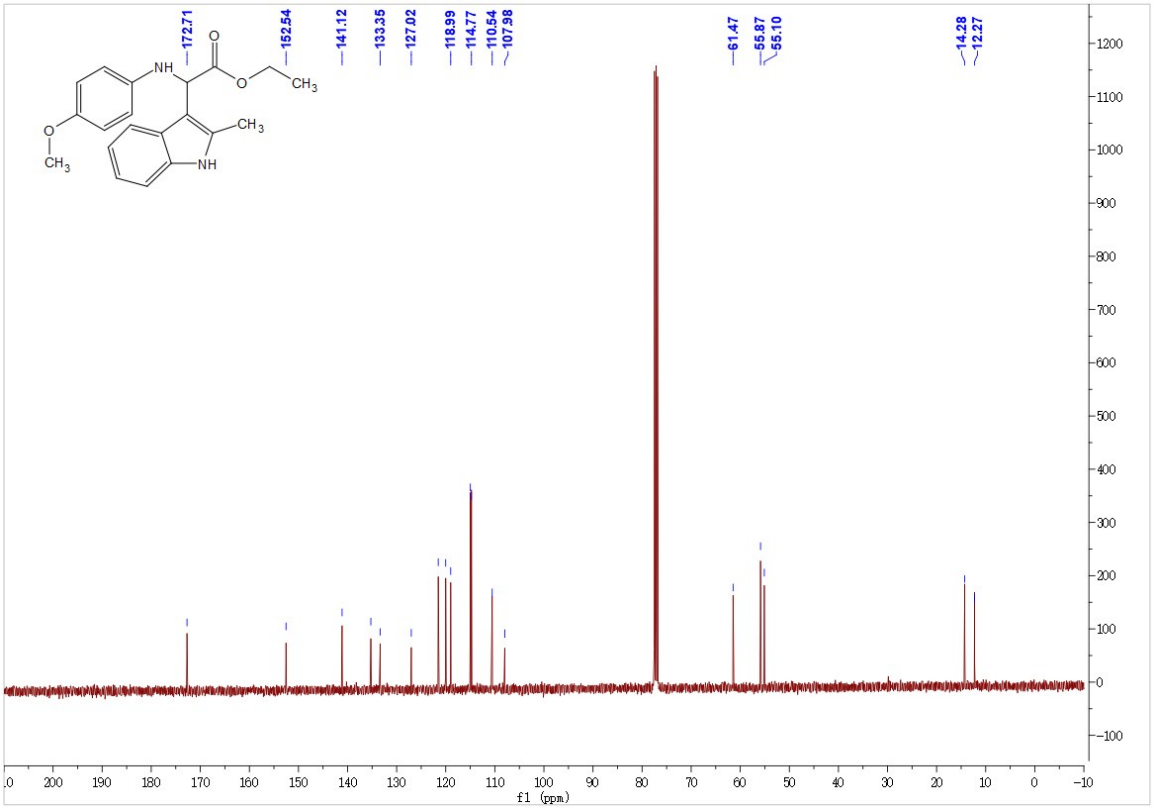
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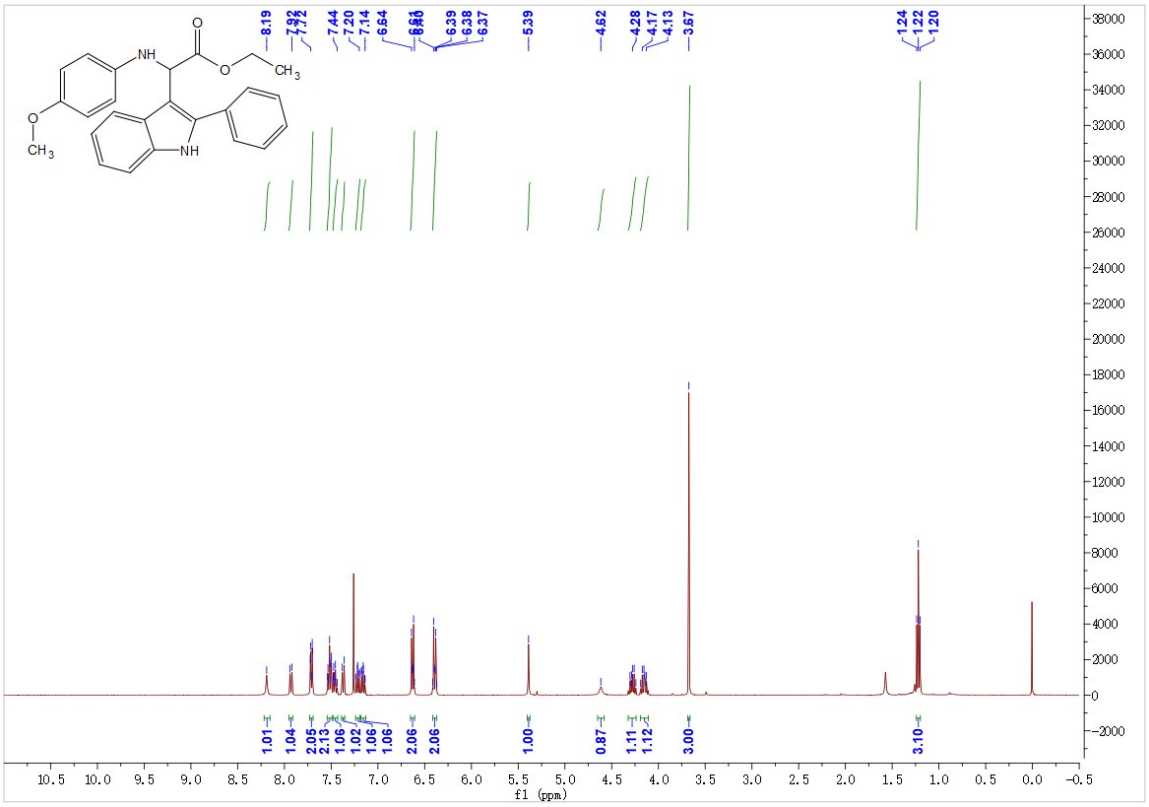
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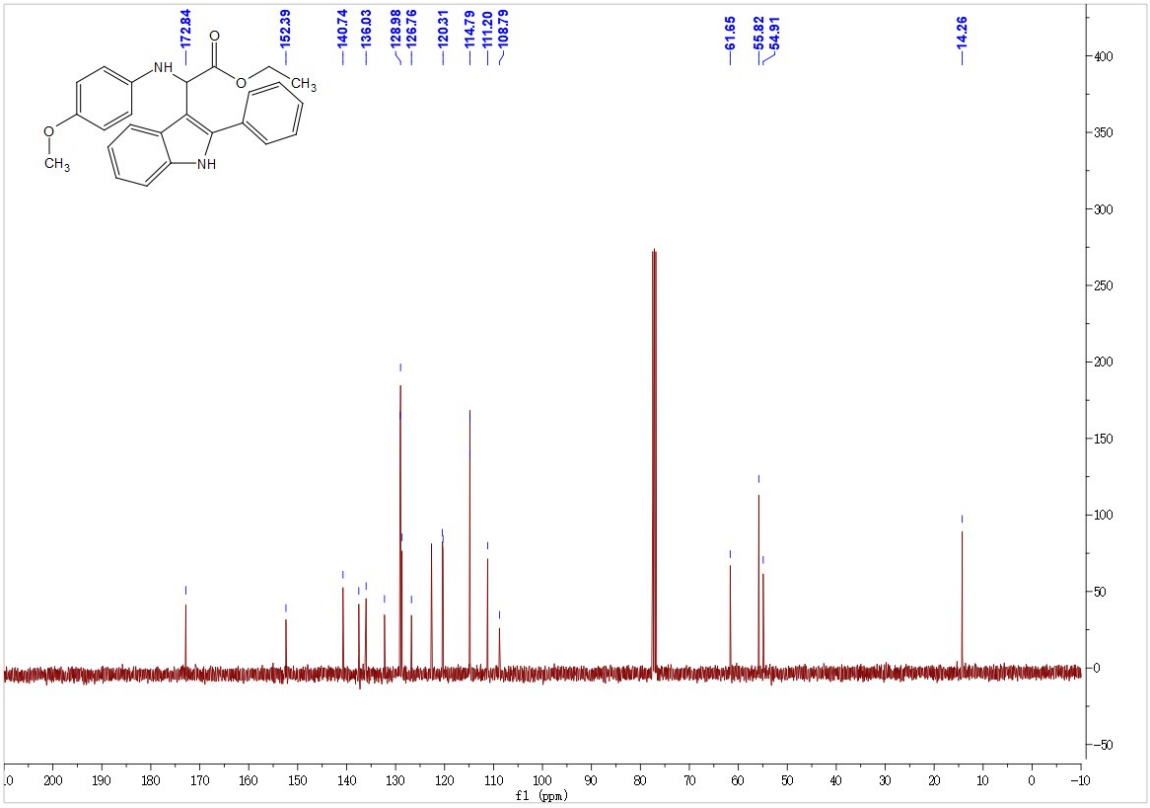
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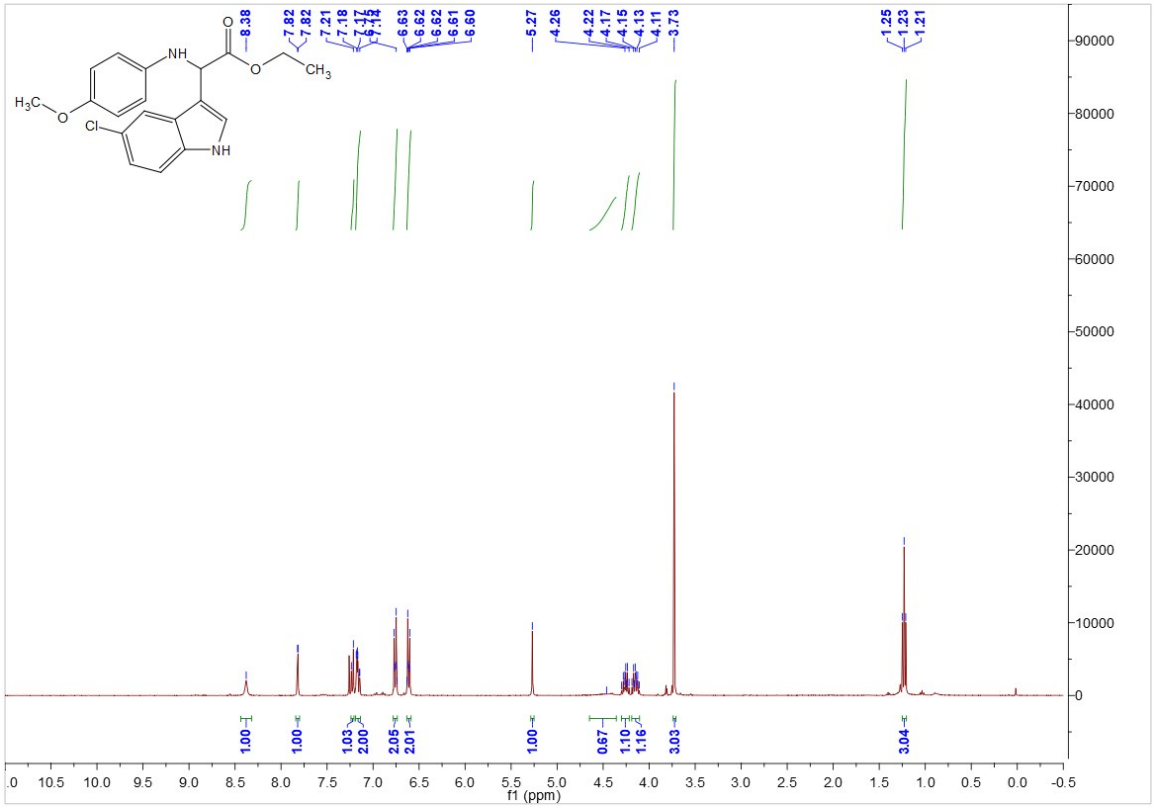
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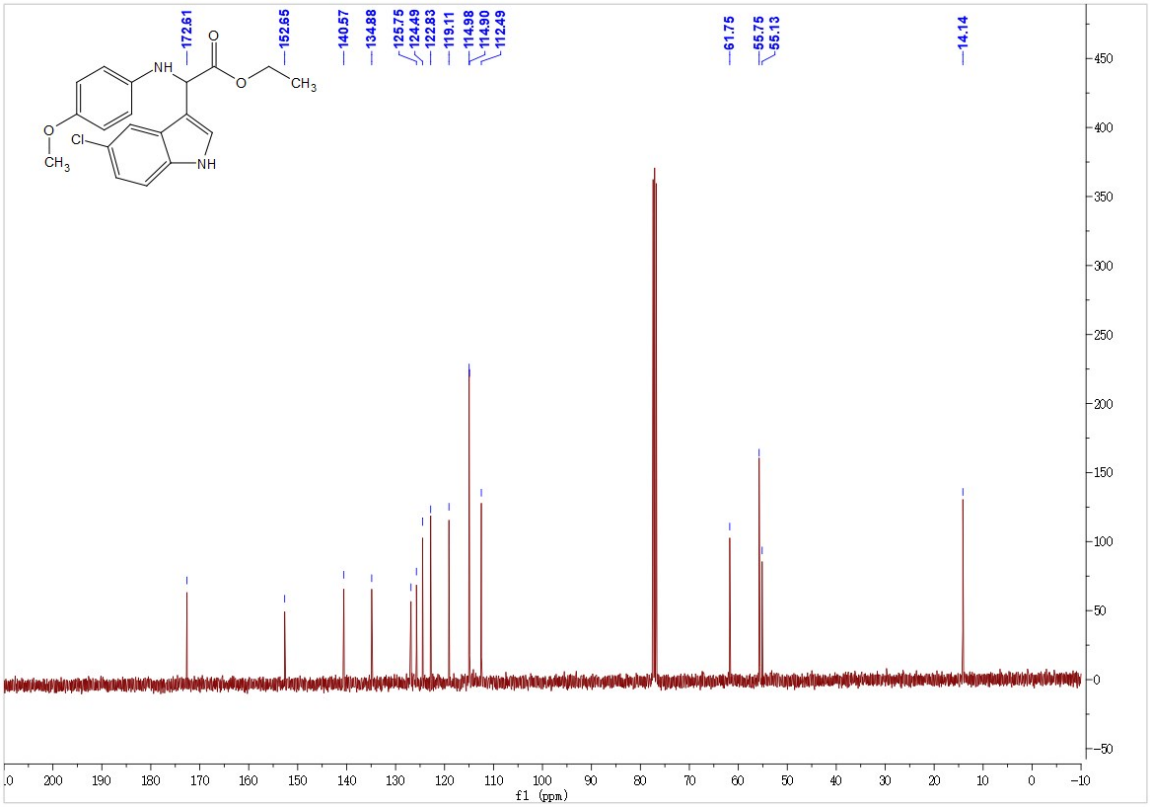
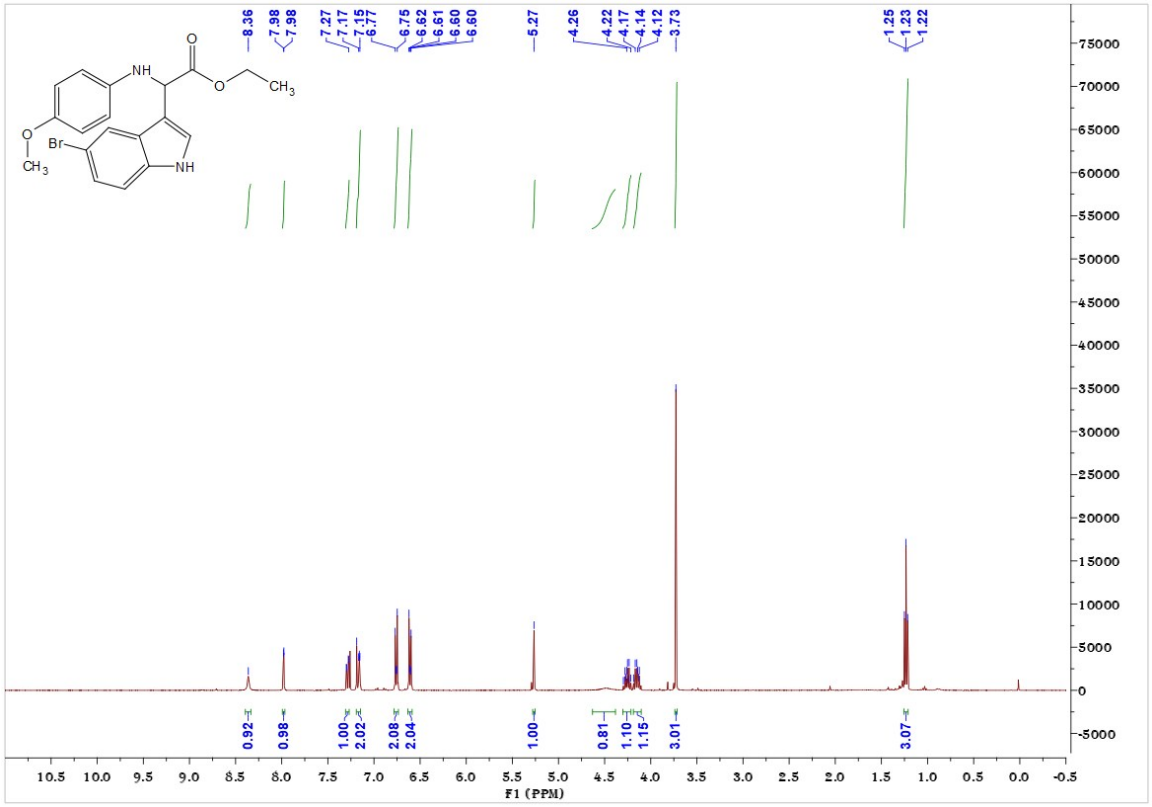

172.61
152.65
140.57
134.88
125.75
124.49
122.83
119.11
114.98
114.90
112.49
61.75
55.75
55.13
14.14
f1 (ppm)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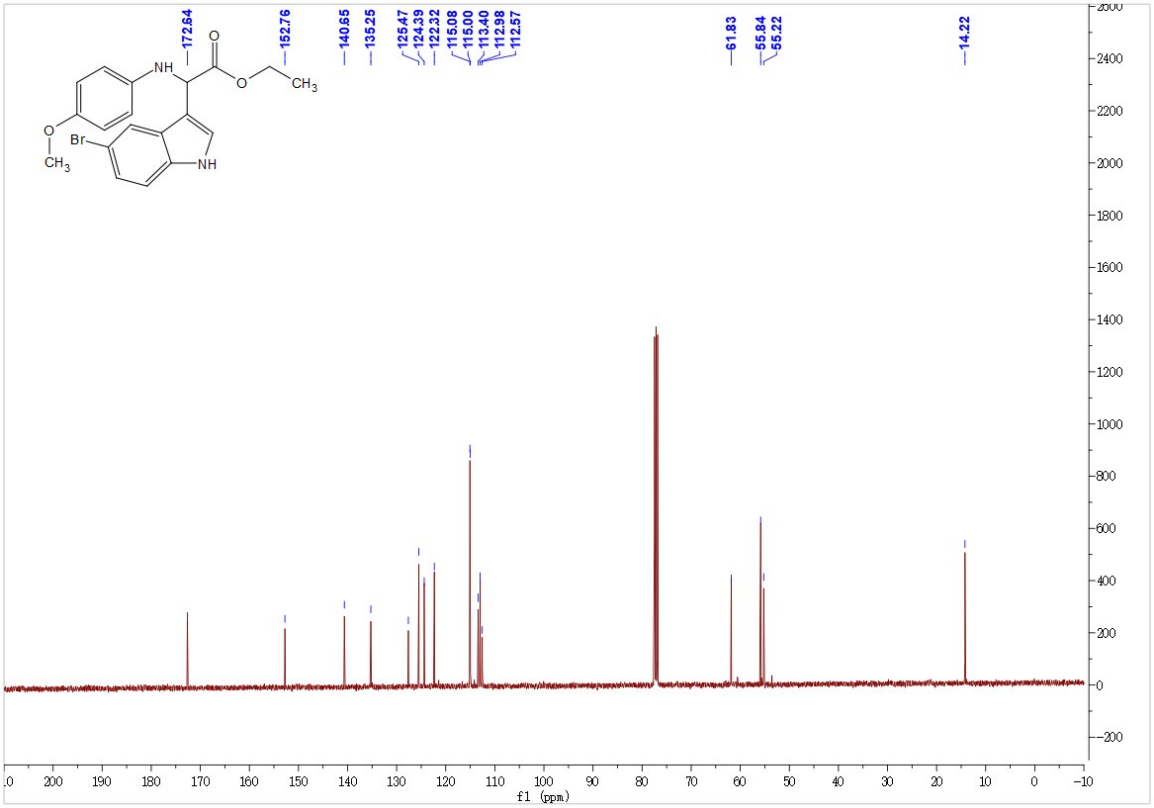
7.98
7.98
7.27
7.17
7.15
6.77
6.75
6.62
6.61
6.60
6.60
5.27
4.26
4.22
4.17
4.14
4.12
3.73
1.25
1.23
1.22
0.92
0.98
1.00
2.02
2.08
2.04
1.00
0.81
1.10
1.15
3.01
3.07
F1 (PPM)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152.76
140.65
135.25
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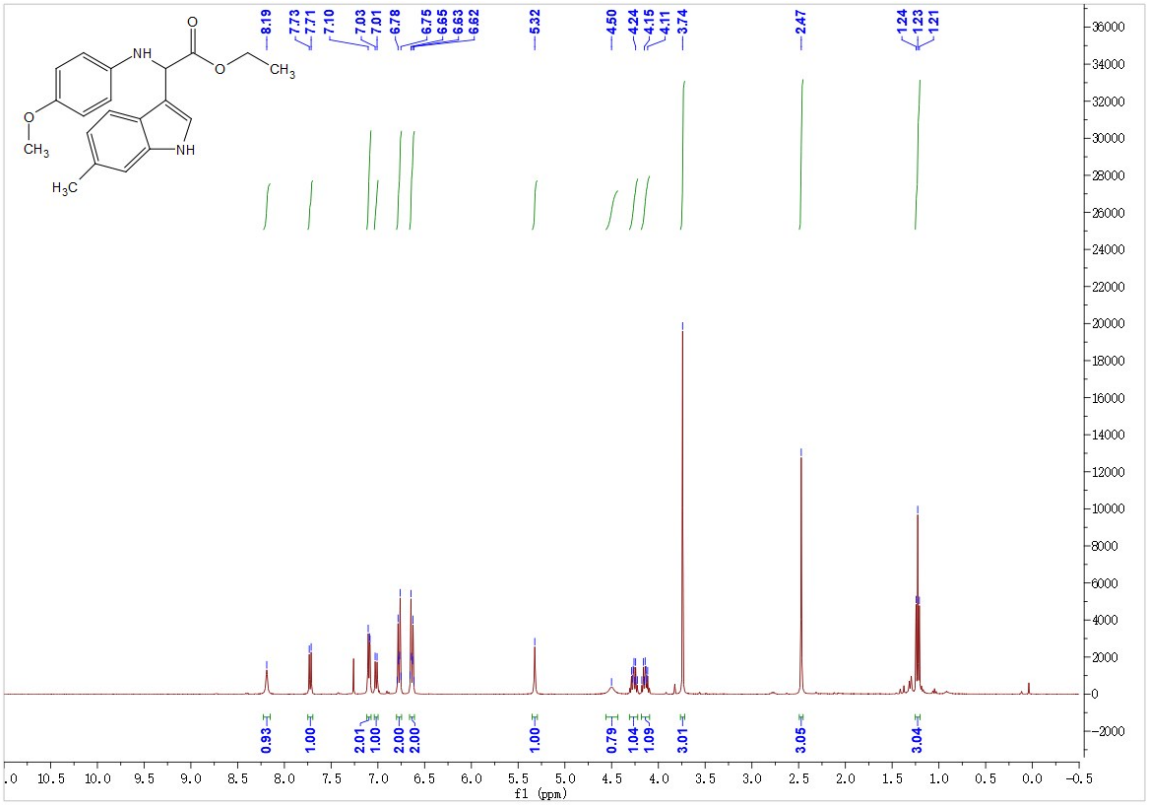
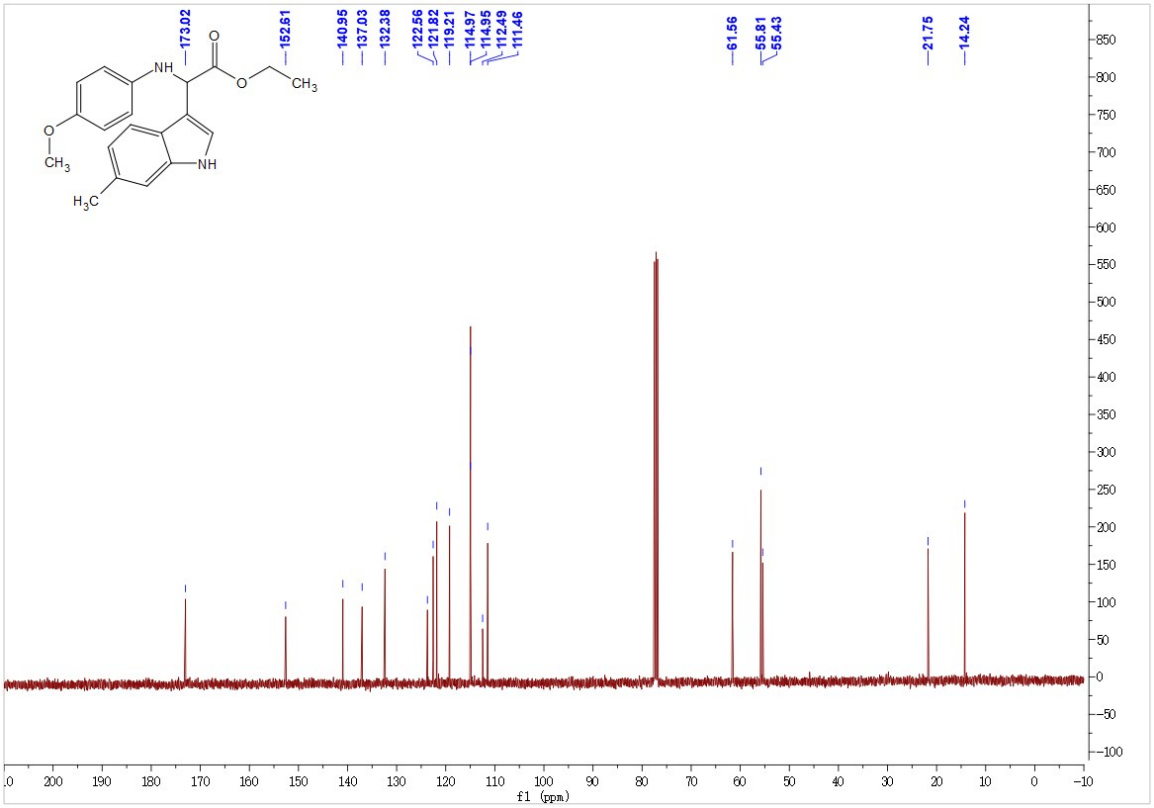
124.39
122.32
115.08
115.00
113.40
112.98
112.57
61.83
55.84
55.22
14.22
f1 (ppm)


8.19
7.73
7.71
7.10
7.03
7.01
6.78
6.75
6.65
6.63
6.62
5.32
4.50
4.24
4.15
4.11
3.74
2.47
1.24
1.23
1.21
0.93
1.00
2.01
1.00
2.00
2.00
1.00
0.79
1.04
1.09
3.01
3.05
3.04
f1 (ppm)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152.61
140.95
137.03
132.38
122.56
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119.21
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114.95
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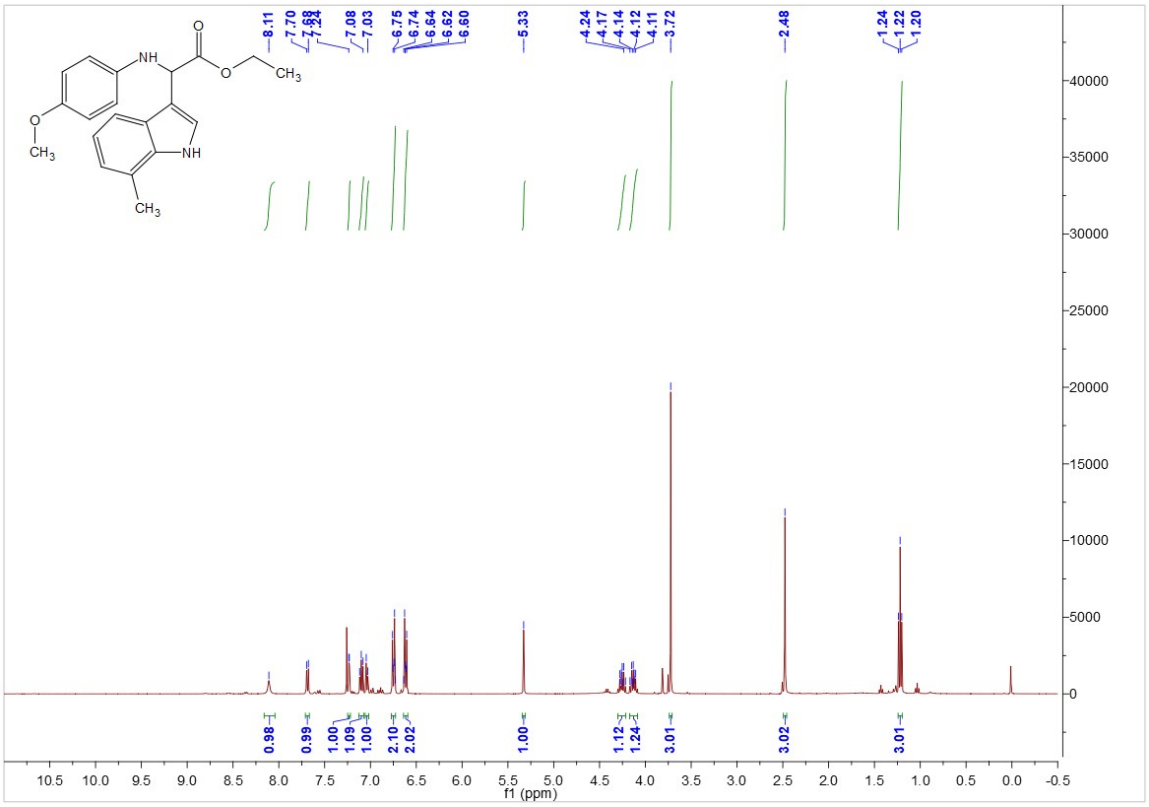
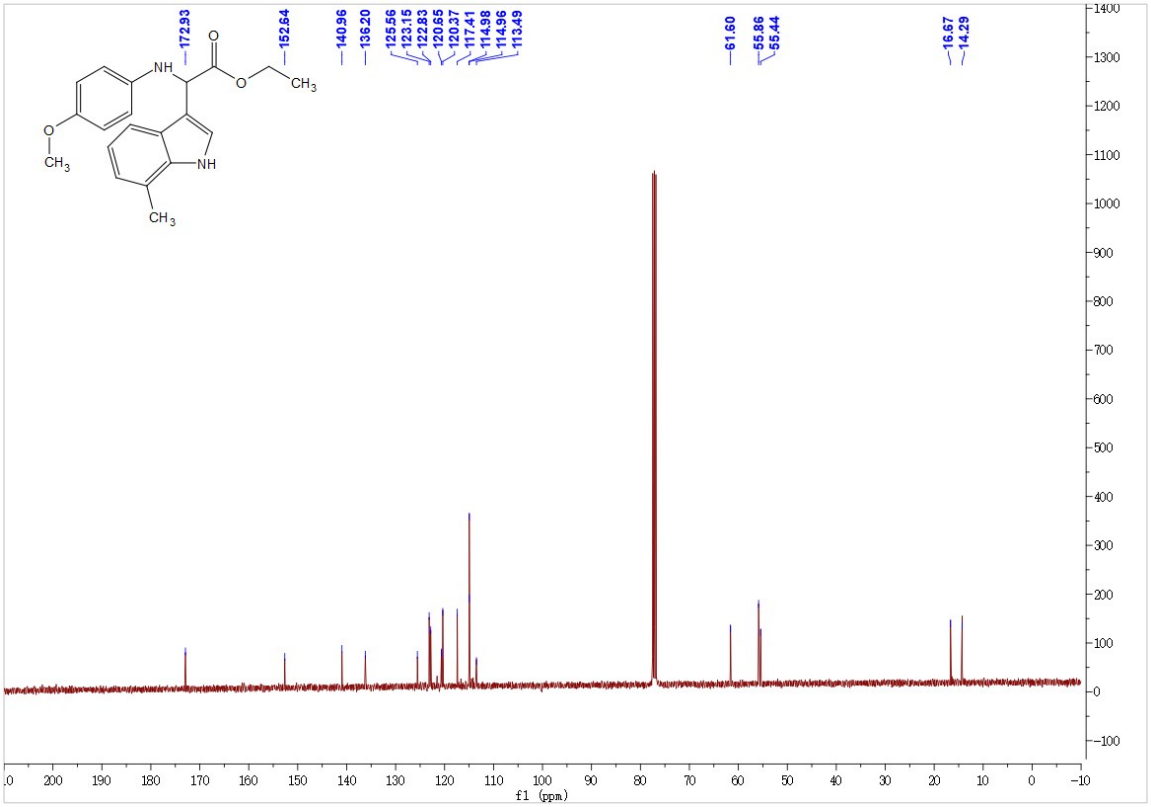
111.46
61.56
55.81
55.43
21.75
14.24
f1 (ppm)


8.11
7.70
7.08
7.03
6.75
6.74
6.64
6.62
6.60
5.33
4.24
4.17
4.14
4.12
4.11
3.72
2.48
1.24
1.22
1.20
0.98
0.99
1.00
1.09
1.00
2.10
2.02
1.00
1.12
1.24
3.01
3.02
3.01
f1 (ppm)


172.93
152.84
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136.20
125.56
123.15
122.83
120.85
120.37
117.41
114.98
114.96
113.49
61.60
55.86
55.44
16.67
14.29
f1 (ppm)