

Supporting Information

B(C₆F₅)₃-Catalyzed β -C(sp³)-H alkylation of tertiary amines with 2-Aryl-3*H*-indol-3-ones

Chang-Peng Zou, Tao Ma, Xiu-Xiu Qiao, Xi-Xi Wu, Ganpeng Li,

Yonghui He*, and Xiao-Jing Zhao*^[a]

Key Laboratory of Chemistry in Ethnic Medicinal Resources, Key Laboratory of Natural Products Synthetic Biology of Ethnic Medicinal Endophytes, State Ethnic Affairs Commission & Ministry of Education, School of Ethnic Medicine, Yunnan Minzu University, Kunming, 650500, China

E-mail: heyonghui@ymu.edu.cn, zhaoxj@ymu.edu.cn

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1. General information

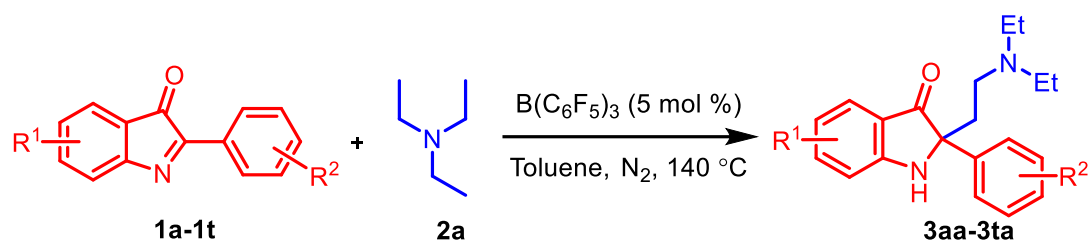
All reactions were performed using oven-dried or flame-dried glassware equipped with a magnetic stir bar before used.

All reagents were purchased from commercial suppliers and used without further purification. All solvents were purified by standard operating method.

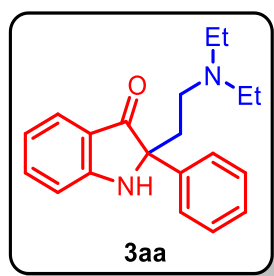
Thin-layer chromatography was performed with EMD silica gel 60 GF₂₅₄ plates eluting with solvents indicated, visualized by a 254 nm UV lamp and stained with phosphomolybdic acid (PMA). **¹H NMR**, **¹³C NMR** and **¹⁹F NMR** spectra were obtained on Bruker AM-400, Chemical shifts were denoted in ppm (δ), and calibrated by using residual undeuterated solvent CDCl₃ (7.26 ppm), tetramethylsilane (0.00 ppm) as internal reference for ¹H NMR and the deuterated solvent CDCl₃ (77.00 ppm) tetramethylsilane (0.00 ppm) as internal standard for ¹³C NMR; solvent *d*₆-DMSO (2.50 ppm), tetramethylsilane (0.00 ppm) as internal reference for ¹H NMR and the deuterated solvent DMSO-*d*₆ (39.50 ppm) tetramethylsilane (0.00 ppm) as internal standard for ¹³C NMR multiplicities are as indicated: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. **High-resolution mass spectral analysis (HRMS) data** was measured on a Bruker impact II (Q-TOF) mass spectrum by means of the ESI technique.

The 2-aryl indoles ^[1] and 2-aryl substituted 3*H*-indol-3-ones **1** were prepared according to the reported procedures. ^[2]

2. General procedure for the synthesis of products



General procedure for the preparation of **3aa-3ta**: Unless otherwise noted, reactions were performed: Cat. $B(C_6F_5)_3$ (5 mol%), **1** (0.10 mmol), **2** (0.50 mmol) were added to **toluene** (1.0 mL) under N_2 atmosphere at $140\text{ }^\circ\text{C}$. Then stirred for 8-72 h. The resulting mixture was concentrated under vacuum, the crude product was purified by flash column chromatography using ethyl acetate/ petroleum ether/ triethylamine to give the title compound **3aa-3ta**.



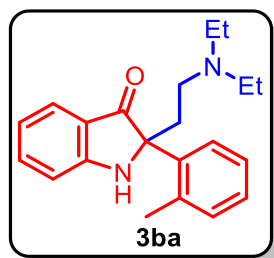
2-(2-(diethylamino)ethyl)-2-phenylindolin-3-one (3aa) According to the general procedure, **3aa** was obtained using 2-phenyl-3H-indol-3-one **1a** (20.7 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 72% yield (22.2 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $DMSO-d_6$) δ 7.96 (s, 1H), 7.52 (d, J = 7.9 Hz, 2H), 7.46 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 7.8 Hz, 3H), 7.25 (t, J = 7.2 Hz, 1H), 6.99 (d, J = 8.3 Hz, 1H), 6.68 (t, J = 7.4 Hz, 1H), 2.43 – 2.21 (m, 6H), 2.20 – 2.13 (m, 1H), 2.10 – 2.02 (m, 1H), 0.80 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, $DMSO-d_6$) δ 200.8, 160.8, 139.7, 137.1, 128.3, 127.2, 125.5, 124.2, 118.1, 117.2, 111.6, 70.4, 47.6, 46.0, 35.0, 11.4.

HRMS (ESI) m/z calcd. for $C_{20}H_{25}N_2O$ ($M+H$) $^+$: 309.1962, found 309.1965.



2-(2-(diethylamino)ethyl)-2-(o-tolyl)indolin-3-one (3ba) According to the general procedure, **3ba** was obtained using 2-(o-tolyl)-3H-indol-3-one **1b** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 43% yield (13.8 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

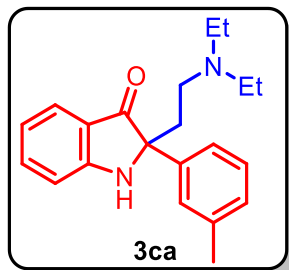
R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $DMSO-d_6$) δ 7.76 (s, 1H), 7.55 – 7.40 (m, 3H), 7.20 – 7.16 (m, 2H), 7.11 (d,

$J = 5.5$ Hz, 1H), 6.91 (d, $J = 8.2$ Hz, 1H), 6.72 (t, $J = 7.3$ Hz, 1H), 2.39 – 2.25 (m, 6H), 2.18 – 2.14 (m, 1H), 2.08 (s, 3H), 1.86 (d, $J = 21.2$ Hz, 1H), 0.83 (t, $J = 7.0$ Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 202.6, 160.4, 137.8, 137.3, 137.0, 132.1, 127.7, 127.5, 125.8, 123.7, 119.9, 117.5, 111.9, 70.9, 47.1, 46.2, 34.3, 20.9, 11.6.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2117.



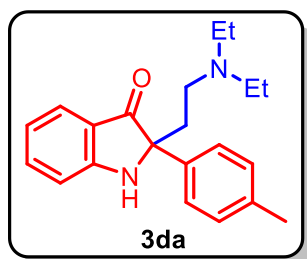
2-(2-(diethylamino)ethyl)-2-(m-tolyl)indolin-3-one (3ca) According to the general procedure, **3ca** was obtained using 2-(m-tolyl)-3H-indol-3-one **1c** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 83% yield (26.7 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

$R_f = 0.30$ (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.93 (s, 1H), 7.47 – 7.43 (m, 1H), 7.32 (t, $J = 9.8$ Hz, 3H), 7.21 (t, $J = 7.6$ Hz, 1H), 7.06 (d, $J = 7.4$ Hz, 1H), 6.98 (d, $J = 8.3$ Hz, 1H), 6.70 – 6.64 (m, 1H), 2.41 – 2.32 (m, 3H), 2.28 (s, 3H), 2.27 – 2.19 (m, 3H), 2.18 – 2.10 (m, 1H), 2.07 – 1.99 (m, 1H), 0.79 (t, $J = 7.1$ Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 200.9, 160.8, 139.6, 137.3, 137.1, 128.2, 127.8, 126.0, 124.2, 122.7, 118.2, 117.2, 111.6, 70.4, 47.6, 46.0, 35.1, 21.2, 11.5.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2117.



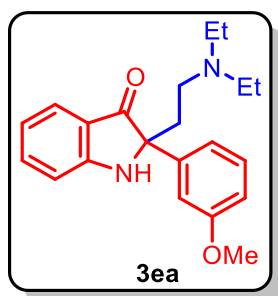
2-(2-(diethylamino)ethyl)-2-(p-tolyl)indolin-3-one (3da) According to the general procedure, **3da** was obtained using 2-(p-tolyl)-3H-indol-3-one **1d** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 71% yield (22.8 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

$R_f = 0.30$ (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.92 (s, 1H), 7.44 (t, $J = 7.7$ Hz, 1H), 7.39 (d, $J = 8.1$ Hz, 2H), 7.33 (d, $J = 7.7$ Hz, 1H), 7.13 (d, $J = 8.1$ Hz, 2H), 6.97 (d, $J = 8.3$ Hz, 1H), 6.67 (t, $J = 7.4$ Hz, 1H), 2.42 – 2.33 (m, 3H), 2.31 – 2.19 (m, 6H), 2.16 – 2.09 (m, 1H), 2.08 – 1.99 (m, 1H), 0.80 (t, $J = 7.1$ Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 201.0, 160.8, 137.1, 136.6, 136.3, 128.9, 125.5, 124.2, 118.2, 117.2, 111.6, 70.3, 47.6, 46.0, 35.0, 20.5, 11.5.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2121.



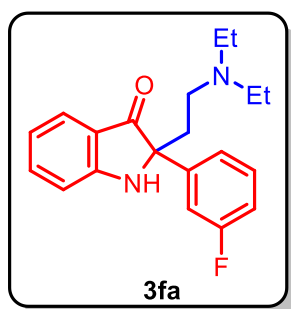
2-(2-(diethylamino)ethyl)-2-(3-methoxyphenyl)indolin-3-one (3ea) According to the general procedure, **3ea** was obtained using 2-(3-methoxyphenyl)-3H-indol-3-one **1e** (23.7 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 89% yield (30.1 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.95 (s, 1H), 7.49 – 7.42 (m, 1H), 7.34 (d, J = 7.6 Hz, 1H), 7.25 (t, J = 8.0 Hz, 1H), 7.10 (d, J = 8.4 Hz, 1H), 7.07 – 7.04 (m, 1H), 6.98 (d, J = 8.3 Hz, 1H), 6.84 (dd, J = 7.9, 2.3 Hz, 1H), 6.68 (t, J = 7.3 Hz, 1H), 3.72 (s, 3H), 2.43 – 2.32 (m, 3H), 2.30 – 2.19 (m, 3H), 2.16 – 2.00 (m, 2H), 0.80 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 200.6, 160.7, 159.2, 141.3, 137.1, 129.4, 124.2, 118.2, 117.8, 117.3, 112.1, 111.8, 111.6, 70.4, 55.0, 47.6, 46.0, 35.2, 11.4.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 339.2067, found 339.2067.



2-(2-(diethylamino)ethyl)-2-(3-fluorophenyl)indolin-3-one (3fa) According to the general procedure, **3fa** was obtained using 2-(3-fluorophenyl)-3H-indol-3-one **1f** (22.5 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 60% yield (19.5 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

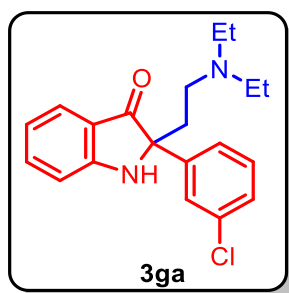
R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.99 (s, 1H), 7.49 – 7.45 (m, 1H), 7.41 – 7.34 (m, 3H), 7.33 – 7.28 (m, 1H), 7.15 – 7.06 (m, 1H), 7.00 (d, J = 8.3 Hz, 1H), 6.70 (t, J = 7.4 Hz, 1H), 2.44 – 2.31 (m, 3H), 2.28 – 2.20 (m, 3H), 2.19 – 2.13 (m, 1H), 2.00 – 2.07 (m, 1H), 0.78 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 200.2, 162.2 (d, J = 242.0 Hz), 160.7, 142.9 (d, J = 7.0 Hz), 137.3, 130.3 (d, J = 8.0 Hz), 124.3, 121.7 (d, J = 2.0 Hz), 118.1, 117.5, 114.0 (d, J = 21.0 Hz), 112.5 (d, J = 23.0 Hz), 111.8, 70.2 (d, J = 2.0 Hz), 47.6, 45.9, 35.4, 11.3.

$^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -113.0.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{FN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 327.1867, found 327.1868.



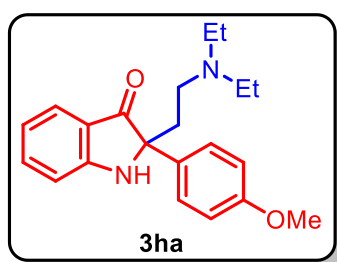
2-(3-chlorophenyl)-2-(2-(diethylamino)ethyl)indolin-3-one (3ga) According to the general procedure, **3ga** was obtained using 2-(3-chlorophenyl)-3H-indol-3-one **1g** (24.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 50% yield (17.1 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 8.01 (s, 1H), 7.54 – 7.50 (m, 2H), 7.49 – 7.44 (m, 1H), 7.40 – 7.31 (m, 3H), 7.00 (d, J = 8.3 Hz, 1H), 6.70 (t, J = 7.3 Hz, 1H), 2.42 – 2.31 (m, 3H), 2.26 – 2.19 (m, 3H), 2.18 – 2.14 (m, 1H), 2.07 – 1.97 (m, 1H), 0.77 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 200.3, 160.8, 142.5, 137.4, 133.2, 130.4, 127.3, 125.4, 124.5, 124.4, 118.1, 117.7, 111.9, 70.1, 47.6, 45.9, 35.3, 11.4.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{ClN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 343.1572, found 343.1570.



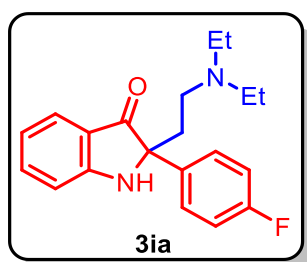
2-(2-(diethylamino)ethyl)-2-(4-methoxyphenyl)indolin-3-one (3ha) According to the general procedure, **3ha** was obtained using 2-(4-methoxyphenyl)-3H-indol-3-one **1h** (23.7 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 66% yield (22.3 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.90 (s, 1H), 7.47 – 7.39 (m, 3H), 7.34 (d, J = 7.6 Hz, 1H), 6.97 (d, J = 8.3 Hz, 1H), 6.91 – 6.84 (m, 2H), 6.70 – 6.64 (m, 1H), 3.71 (s, 3H), 2.42 – 2.33 (m, 3H), 2.30 – 2.20 (m, 3H), 2.13 – 2.07 (m, 1H), 2.05 – 1.98 (m, 1H), 0.80 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 201.2, 160.8, 158.5, 137.1, 131.4, 126.7, 124.3, 118.2, 117.2, 113.7, 111.6, 70.0, 55.0, 47.6, 46.0, 35.0, 11.5.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_2$ ($\text{M}+\text{H}$) $^+$: 339.2067, found 339.2067.



2-(2-(diethylamino)ethyl)-2-(4-fluorophenyl)indolin-3-one (3ia) According to the general procedure, **3ia** was obtained using 2-(4-fluorophenyl)-3H-indol-3-one **1i** (22.5 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 42% yield (13.7 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

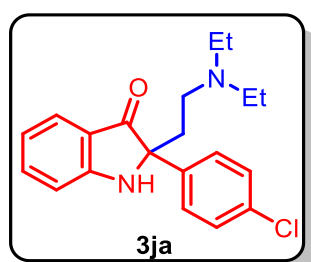
R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.98 (s, 1H), 7.57 – 7.53 (m, 2H), 7.48 – 7.42 (m, 1H), 7.35 (d, J = 7.7 Hz, 1H), 7.20 – 7.13 (m, 2H), 6.98 (d, J = 8.3 Hz, 1H), 6.69 (t, J = 7.4 Hz, 1H), 2.40 – 2.32 (m, 3H), 2.28 – 2.19 (m, 3H), 2.19 – 2.11 (m, 1H), 2.06 – 1.98 (m, 1H), 0.78 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 200.8, 161.5 (d, J = 242.0 Hz), 160.7, 137.2, 135.9 (d, J = 3.0 Hz), 127.6 (d, J = 8.0 Hz), 124.3, 118.1, 117.4, 115.1 (d, J = 21.0 Hz), 111.7, 70.0, 47.6, 46.0, 35.3, 11.4.

$^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -115.9.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{FN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 327.1867, found 327.1868.



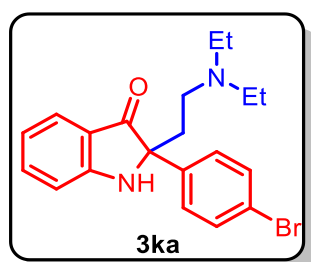
2-(4-chlorophenyl)-2-(2-(diethylamino)ethyl)indolin-3-one (3ja) According to the general procedure, **3ja** was obtained using 2-(4-chlorophenyl)-3H-indol-3-one **1j** (24.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 30% yield (10.3 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 8.00 (s, 1H), 7.56 – 7.50 (m, 2H), 7.50 – 7.43 (m, 1H), 7.43 – 7.37 (m, 2H), 7.35 (d, J = 7.6 Hz, 1H), 6.99 (d, J = 8.3 Hz, 1H), 6.69 (t, J = 7.4 Hz, 1H), 2.41 – 2.31 (m, 3H), 2.27 – 2.19 (m, 3H), 2.19 – 2.10 (m, 1H), 2.05 – 1.98 (m, 1H), 0.78 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 200.6, 160.9, 138.9, 137.4, 132.1, 128.4, 127.6, 124.4, 118.1, 117.6, 111.9, 70.1, 47.7, 46.0, 35.1, 11.4.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{ClN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 343.1572, found 343.1576.



2-(4-bromophenyl)-2-(2-(diethylamino)ethyl)indolin-3-one (3ka) According to the general procedure, **3ka** was obtained using 2-(4-bromophenyl)-3H-indol-3-one **1k** (27.2 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 53% yield (20.4 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

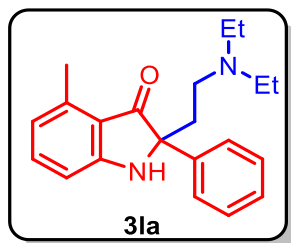
R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.99 (s, 1H), 7.54 (d, J = 8.7 Hz, 2H), 7.49 – 7.44 (m, 3H), 7.35

(d, $J = 7.6$ Hz, 1H), 6.99 (d, $J = 8.3$ Hz, 1H), 6.69 (t, $J = 7.4$ Hz, 1H), 2.41 – 2.32 (m, 3H), 2.28 – 2.20 (m, 3H), 2.19 – 2.11 (m, 1H), 2.06 – 1.99 (m, 1H), 0.78 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 200.4, 160.8, 139.3, 137.3, 131.2, 127.9, 124.3, 120.6, 118.1, 117.5, 111.8, 70.1, 47.6, 45.9, 35.1, 11.3.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{BrN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 387.1067, found 387.1065.



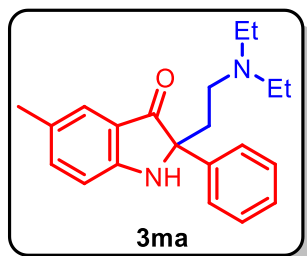
2-(2-(diethylamino)ethyl)-4-methyl-2-phenylindolin-3-one (3la) According to the general procedure, **3la** was obtained using 4-methyl-2-phenyl-3H-indol-3-one **II** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 80% yield (25.7 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

$R_f = 0.30$ (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

^1H NMR (400 MHz, DMSO- d_6) δ 7.87 (s, 1H), 7.53 – 7.48 (m, 2H), 7.35 – 7.29 (m, 3H), 7.27 – 7.23 (m, 1H), 6.78 (d, $J = 8.2$ Hz, 1H), 6.42 (d, $J = 7.2$ Hz, 1H), 2.43 – 2.33 (m, 6H), 2.28 – 2.19 (m, 3H), 2.18 – 2.10 (m, 1H), 2.07 – 2.00 (m, 1H), 0.80 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 201.4, 161.3, 140.0, 138.7, 136.5, 128.3, 127.1, 125.6, 118.5, 116.0, 108.9, 70.2, 47.7, 46.0, 35.1, 17.6, 11.5.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2115.



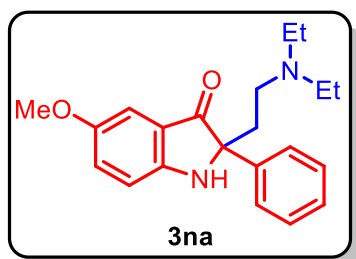
2-(2-(diethylamino)ethyl)-5-methyl-2-phenylindolin-3-one (3ma) According to the general procedure, **3ma** was obtained using 5-methyl-2-phenyl-3H-indol-3-one **1m** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 70% yield (22.5 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

$R_f = 0.30$ (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

^1H NMR (400 MHz, DMSO- d_6) δ 7.77 (s, 1H), 7.50 (d, $J = 7.3$ Hz, 2H), 7.34 – 7.29 (m, 3H), 7.24 (t, $J = 7.2$ Hz, 1H), 7.14 (s, 1H), 6.92 (d, $J = 8.3$ Hz, 1H), 2.40 – 2.32 (m, 3H), 2.30 – 2.21 (m, 3H), 2.19 (s, 3H), 2.15 – 2.02 (m, 2H), 0.81 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 200.9, 159.5, 139.8, 138.6, 128.3, 127.1, 126.2, 125.6, 123.5, 118.2, 111.7, 70.8, 47.6, 46.1, 35.0, 20.0, 11.6.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2114.



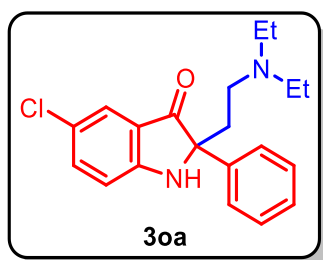
2-(2-(diethylamino)ethyl)-5-methoxy-2-phenylindolin-3-one (3na) According to the general procedure, **3na** was obtained using 5-methoxy-2-phenyl-3H-indol-3-one **1n** (23.7 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 67% yield (22.6 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.63 (s, 1H), 7.51 (d, *J* = 7.3 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.25 (t, *J* = 7.2 Hz, 1H), 7.17 (dd, *J* = 8.8, 2.7 Hz, 1H), 6.98 (d, *J* = 8.8 Hz, 1H), 6.82 (d, *J* = 2.6 Hz, 1H), 3.67 (s, 3H), 2.43 – 2.32 (m, 3H), 2.31 – 2.21 (m, 3H), 2.16 – 2.04 (m, 2H), 0.81 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 201.1, 156.9, 152.0, 139.8, 128.3, 127.5, 127.2, 125.6, 118.0, 113.4, 104.6, 71.4, 55.5, 47.7, 46.1, 35.0, 11.6.

HRMS (ESI) *m/z* calcd. for C₂₁H₂₇N₂O₂ (M+H)⁺: 339.2067, found 339.2062.



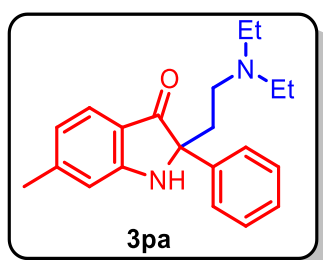
5-chloro-2-(2-(diethylamino)ethyl)-2-phenylindolin-3-one (3oa) According to the general procedure, **3oa** was obtained using 5-chloro-2-phenyl-3H-indol-3-one **1o** (24.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 59% yield (20.2 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.12 (s, 1H), 7.51 – 7.48 (m, 2H), 7.45 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.35 – 7.31 (m, 3H), 7.29 – 7.23 (m, 1H), 7.01 (d, *J* = 8.7 Hz, 1H), 2.46 – 2.40 (m, 1H), 2.38 – 2.29 (m, 3H), 2.26 – 2.17 (m, 3H), 2.05 – 1.98 (m, 1H), 0.76 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 199.5, 159.1, 139.5, 136.5, 128.5, 127.4, 125.5, 123.1, 120.9, 119.5, 113.2, 71.2, 47.7, 45.7, 35.4, 11.1.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₄ClN₂O (M+H)⁺: 343.1572, found 343.1577.



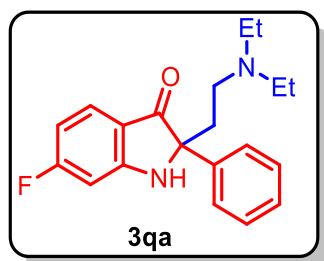
2-(2-(diethylamino)ethyl)-6-methyl-2-phenylindolin-3-one (3pa) According to the general procedure, **3pa** was obtained using 6-methyl-2-phenyl-3H-indol-3-one **1p** (22.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 84% yield (27.0 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.90 (s, 1H), 7.53 – 7.47 (m, 2H), 7.34 – 7.30 (m, 2H), 7.24 (t, J = 7.5 Hz, 2H), 6.79 (s, 1H), 6.51 (dd, J = 7.9, 0.7 Hz, 1H), 2.43 – 2.33 (m, 3H), 2.32 – 2.22 (m, 6H), 2.15 – 2.02 (m, 2H), 0.82 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 200.1, 161.3, 148.2, 139.8, 128.3, 127.1, 125.5, 124.1, 119.1, 115.8, 111.5, 70.7, 47.5, 46.2, 34.9, 22.0, 11.6.

HRMS (ESI) m/z calcd. for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 323.2118, found 323.2128.



2-(2-(diethylamino)ethyl)-6-fluoro-2-phenylindolin-3-one (3qa) According to the general procedure, **3qa** was obtained using 6-fluoro-2-phenyl-3H-indol-3-one **1q** (22.5 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 38% yield (12.4 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

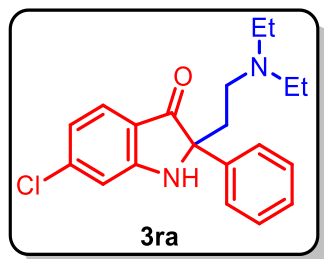
R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

$^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 8.26 (s, 1H), 7.49 (d, J = 7.4 Hz, 2H), 7.42 – 7.38 (m, 1H), 7.33 (t, J = 7.5 Hz, 2H), 7.26 (t, J = 7.2 Hz, 1H), 6.73 (dd, J = 10.5, 2.1 Hz, 1H), 6.48 (td, J = 9.3, 2.1 Hz, 1H), 2.43 – 2.32 (m, 3H), 2.28 – 2.21 (m, 3H), 2.21 – 2.16 (m, 1H), 2.09 – 2.00 (m, 1H), 0.78 (t, J = 7.1 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 199.0, 168.7 (d, J = 250.0 Hz), 162.2 (d, J = 15.0 Hz), 139.5, 128.5, 127.4, 127.0 (d, J = 12.0 Hz), 125.5, 115.2, 105.7 (d, J = 24.0 Hz), 97.4 (d, J = 25.0 Hz), 71.3, 47.6, 46.0, 35.0, 11.3.

$^{19}\text{F NMR}$ (376 MHz, $\text{DMSO-}d_6$) δ -101.4.

HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{24}\text{FN}_2\text{O}$ ($\text{M}+\text{H}$) $^+$: 327.1867, found 327.1865.



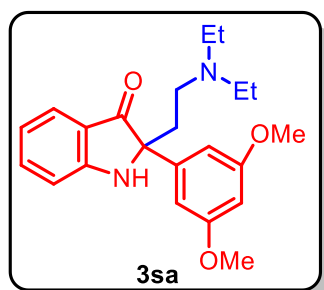
6-chloro-2-(2-(diethylamino)ethyl)-2-phenylindolin-3-one (3ra) According to the general procedure, **3ra** was obtained using 6-chloro-2-phenyl-3H-indol-3-one **1r** (24.1 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 30% yield (10.3 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.19 (s, 1H), 7.49 (d, *J* = 7.5 Hz, 2H), 7.38 – 7.31 (m, 3H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.01 (d, *J* = 1.7 Hz, 1H), 6.68 (dd, *J* = 8.2, 1.7 Hz, 1H), 2.44 – 2.34 (m, 3H), 2.32 – 2.26 (m, 1H), 2.25 – 2.18 (m, 3H), 2.05 – 1.99 (m, 1H), 0.77 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 199.4, 161.0, 141.9, 139.5, 128.5, 127.5, 125.9, 125.5, 117.5, 117.30, 110.9, 71.0, 47.7, 45.8, 35.2, 11.2.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₄ClN₂O (M+H)⁺: 343.1572, found 343.1568.



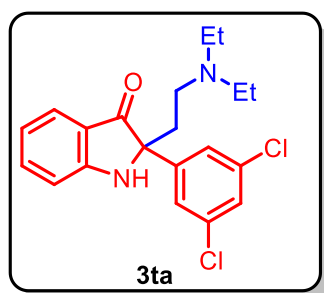
2-(2-(diethylamino)ethyl)-2-(3,5-dimethoxyphenyl)indolin-3-one (3sa) According to the general procedure, **3sa** was obtained using 2-(3,5-dimethoxyphenyl)-3H-indol-3-one **1s** (26.7 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 62% yield (22.8 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.94 (s, 1H), 7.47 – 7.42 (m, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 6.97 (d, *J* = 8.3 Hz, 1H), 6.69 – 6.65 (m, 3H), 6.41 (t, *J* = 2.2 Hz, 1H), 3.71 (s, 6H), 2.40 – 2.33 (m, 3H), 2.29 – 2.20 (m, 3H), 2.12 – 1.99 (m, 2H), 0.80 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 200.4, 160.7, 160.4, 142.1, 137.1, 124.2, 118.2, 117.3, 111.6, 104.0, 98.4, 70.4, 55.2, 47.7, 46.0, 35.3, 11.5.

HRMS (ESI) *m/z* calcd. for C₂₂H₂₉N₂O₃ (M+H)⁺: 369.2173, found 369.2173.



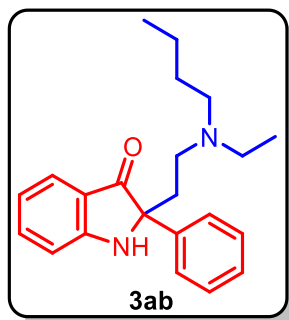
2-(3,5-dichlorophenyl)-2-(2-(diethylamino)ethyl)indolin-3-one (3ta) According to the general procedure, **3ta** was obtained using 2-(3,5-dichlorophenyl)-3H-indol-3-one **1t** (27.5 mg, 0.1 mmol) and triethylamine **2a** (50.6 mg, 0.5 mmol) in 48% yield (18.1 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.02 (s, 1H), 7.57 – 7.52 (m, 3H), 7.51 – 7.45 (m, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 6.72 (t, *J* = 7.3 Hz, 1H), 2.41 – 2.29 (m, 3H), 2.26 – 2.18 (m, 3H), 2.18 – 2.13 (m, 1H), 2.02 – 1.97 (m, 1H), 0.75 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, DMSO-*d*₆) δ 199.5, 160.5, 144.6, 137.4, 134.1, 127.0, 124.5, 124.3, 118.1, 117.8, 111.9, 69.8, 47.7, 45.7, 35.7, 11.2.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₃Cl₂N₂O (M+H)⁺: 377.1182, found 377.1187.



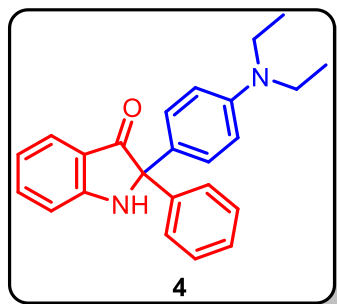
2-(2-(butyl(ethyl)amino)ethyl)-2-phenylindolin-3-one (3ab) According to the general procedure, **3ab** was obtained using 2-phenyl-3*H*-indol-3-one **1a** (20.7 mg, 0.1 mmol) and *N,N*-diethylbutan-1-amine **2b** (64.6 mg, 0.5 mmol) in 30% yield (10.1 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, CDCl₃) δ 7.74 – 7.61 (m, 3H), 7.54 (d, J = 7.7 Hz, 1H), 7.47 – 7.43 (m, 1H), 7.32 (t, J = 7.5 Hz, 2H), 7.25 – 7.22 (m, 1H), 6.92 (d, J = 8.3 Hz, 1H), 6.76 (t, J = 7.2 Hz, 1H), 2.62 – 2.51 (m, 2H), 2.50 – 2.43 (m, 1H), 2.33 – 2.17 (m, 3H), 2.15 – 2.07 (m, 1H), 1.99 – 1.90 (m, 1H), 1.30 – 1.22 (m, 4H), 0.92 – 0.84 (m, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 203.0, 160.9, 138.7, 137.2, 128.3, 127.3, 126.5, 125.4, 119.4, 118.5, 112.8, 72.4, 53.2, 50.4, 47.1, 34.5, 29.2, 20.8, 14.1, 11.7.

HRMS (ESI) m/z calcd. for C₂₂H₂₉N₂O (M+H)⁺: 337.2275, found 337.2278.



2-(4-(diethylamino)phenyl)-2-phenylindolin-3-one (4) According to the general procedure, **4** was obtained using 2-phenyl-3*H*-indol-3-one **1a** (20.7 mg, 0.1 mmol) and *N,N*-diethylaniline **2** (74.6 mg, 0.5 mmol) in 92% yield (32.7 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate :petroleum ether :triethylamine = 2:100:1).

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

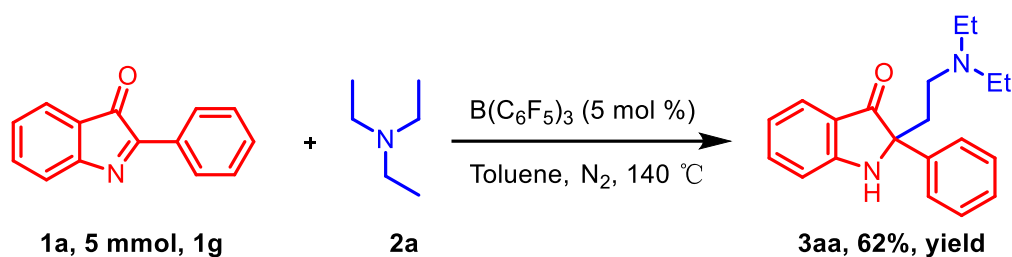
¹H NMR (400 MHz, DMSO-*d*₆) δ 8.38 (s, 1H), 7.50 – 7.45 (m, 1H), 7.43 (d, J = 7.7 Hz, 1H), 7.37 – 7.25 (m, 5H), 7.07 (d, J = 8.9 Hz, 2H), 6.94 (d, J = 8.3 Hz, 1H), 6.70 (t, J = 7.4 Hz, 1H), 6.58 (d, J = 9.0 Hz, 2H), 3.27 (q, J = 7.0 Hz, 4H), 1.03 (t, J = 7.0 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 200.8, 160.7, 146.7, 141.4, 137.8, 128.2, 128.1, 127.4, 127.1, 126.6, 124.6, 117.6, 117.5, 111.6, 111.1, 73.7, 43.7, 12.5.

HRMS (ESI) m/z calcd. for C₂₄H₂₅N₂O (M+H)⁺: 357.1962, found 357.1967.

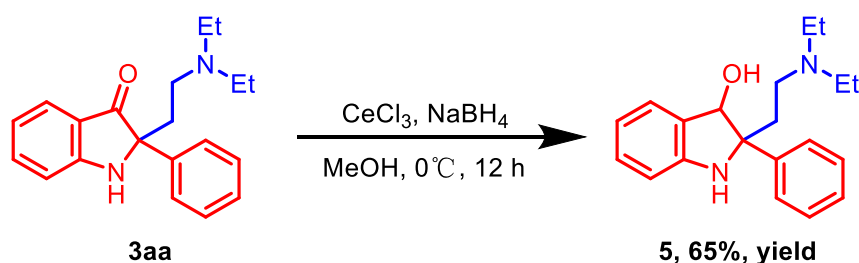
3. Gram-scale experiments and transformations of the products

a) Gram-scale synthesis of **3aa**



According to the general procedure, **3aa** was obtained using **1a** (1000 mg, 5 mmol), **2a** (2529.8 mg, 25 mmol) and **B(C₆F₅)₃** (128 mg, 5 mol%) in 62% yield (954 mg) as a yellow oily (silica gel flash chromatography: ethyl acetate: petroleum ether: triethylamine = 2:100:1).

b) Derivatizations of **3aa**



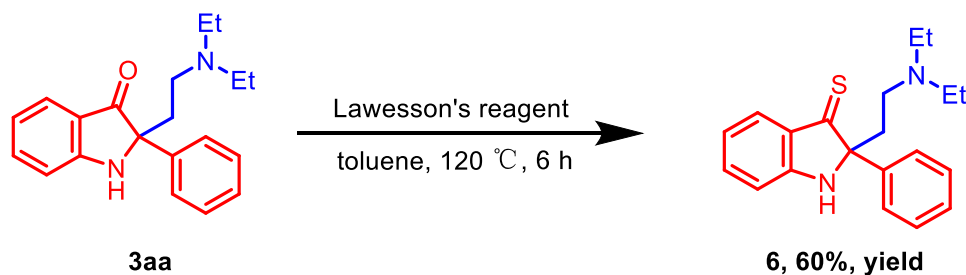
To a solution of the compound **3aa** (30.8 mg, 0.1 mmol) in anhydrous MeOH (1.0 mL) at 0 °C, CeCl₃ (29.6 mg, 0.12 mmol) was added and followed by the addition of NaBH₄ (5.7 mg, 0.15 mmol). The reaction mixture was allowed to stir for 1 h at the same temperature and then the bath temperature was slowly warmed to room temperature. When **3aa** was fully converted and excessive NaBH₄ was consumed completely, the mixture was concentrated in vacuo and the residue was purified via column chromatography on silica gel (ethyl acetate: petroleum ether: triethylamine = 2:100:1) to afford the product **5** (65%, 20 mg) as a white solid.

R_f = 0.30 (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.52 (d, *J* = 7.5 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.06 (d, *J* = 7.2 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.60 (d, *J* = 7.8 Hz, 1H), 6.55 (t, *J* = 7.3 Hz, 1H), 6.40 (s, 1H), 4.85 (s, 1H), 2.41 – 2.32 (m, 3H), 2.23 – 2.14 (m, 2H), 2.13 – 2.08 (m, 2H), 1.89 – 1.79 (m, 1H), 0.81 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 149.9, 148.0, 130.0, 128.6, 128.0, 126.1, 125.5, 124.5, 116.8, 108.2, 81.0, 73.0, 48.3, 45.9, 32.5, 11.1.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₇N₂O (M+H)⁺: 311.2118, found 311.2114.



A solution of the compound **3aa** (30.8 mg, 0.1 mmol) and Lawesson's reagent (48.6 mg, 0.12 mmol)

in toluene (1.5 mL) was heated to 120 °C at reflux under argon for 6 h. Then the mixture was concentrated in vacuo, and the residue was purified via column chromatography on silica gel (ethyl acetate: petroleum ether: triethylamine = 2:80:1) to afford the product **6** (60%, 19.5 mg) as a red oily. $R_f = 0.20$ (ethyl acetate: petroleum ether: triethylamine = 2:100:1).

^1H NMR (400 MHz, Acetone- d_6) δ 8.56 (s, 1H), 7.64 – 7.52 (m, 4H), 7.33 – 7.27 (m, 2H), 7.26 – 7.22 (m, 1H), 7.18 (d, $J = 8.4$ Hz, 1H), 6.79 – 6.75 (m, 1H), 2.63 – 2.50 (m, 4H), 2.44 – 2.38 (m, 1H), 2.36 – 2.30 (m, 2H), 2.21 – 2.13 (m, 1H), 0.91 (t, $J = 7.1$ Hz, 6H).

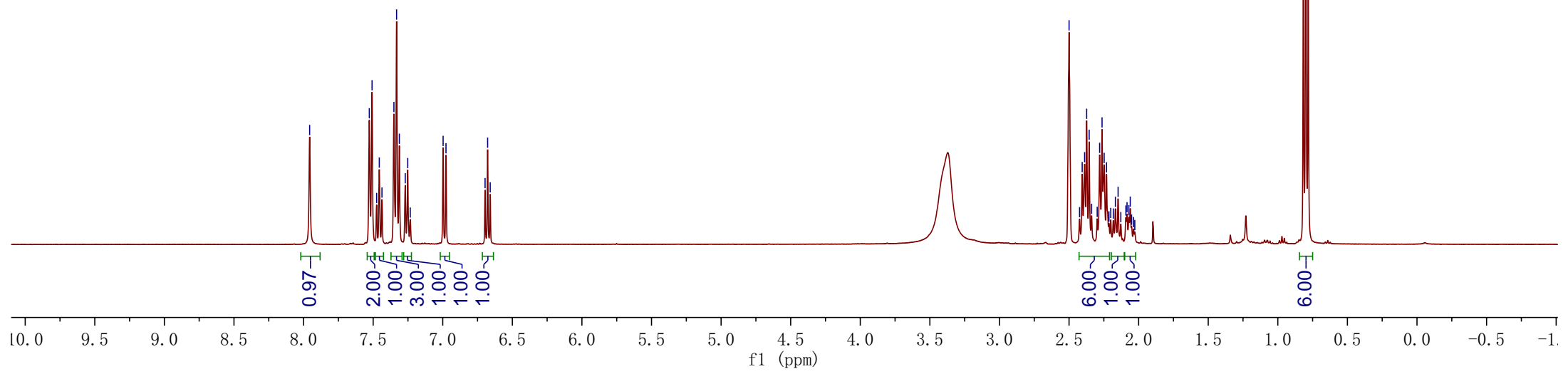
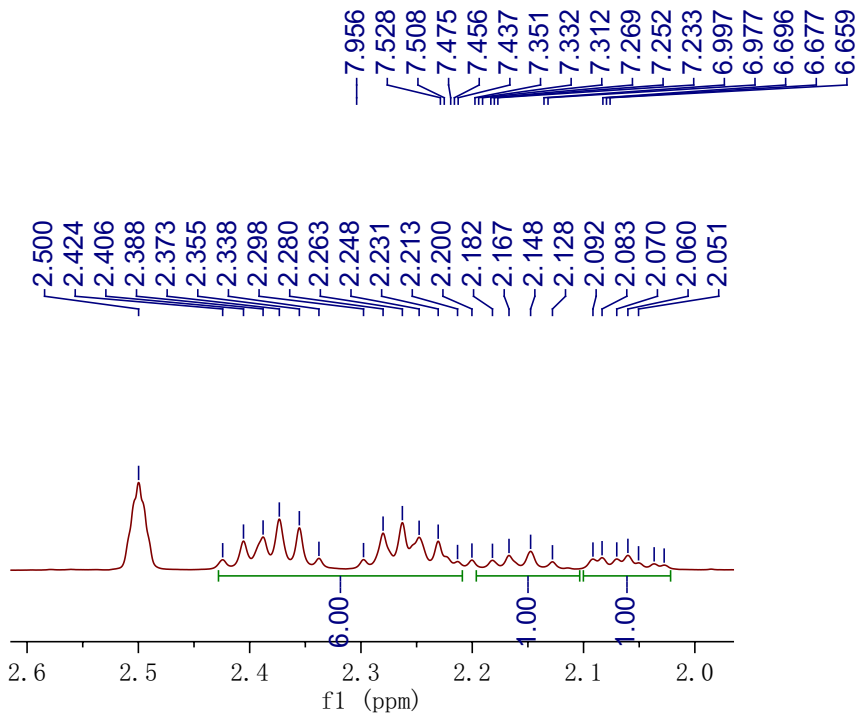
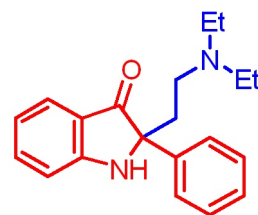
^{13}C NMR (100 MHz, Acetone- d_6) δ 235.1, 161.5, 140.6, 139.3, 133.2, 129.0, 128.1, 127.2, 125.9, 119.8, 113.5, 83.6, 49.5, 47.6, 39.5, 12.2.

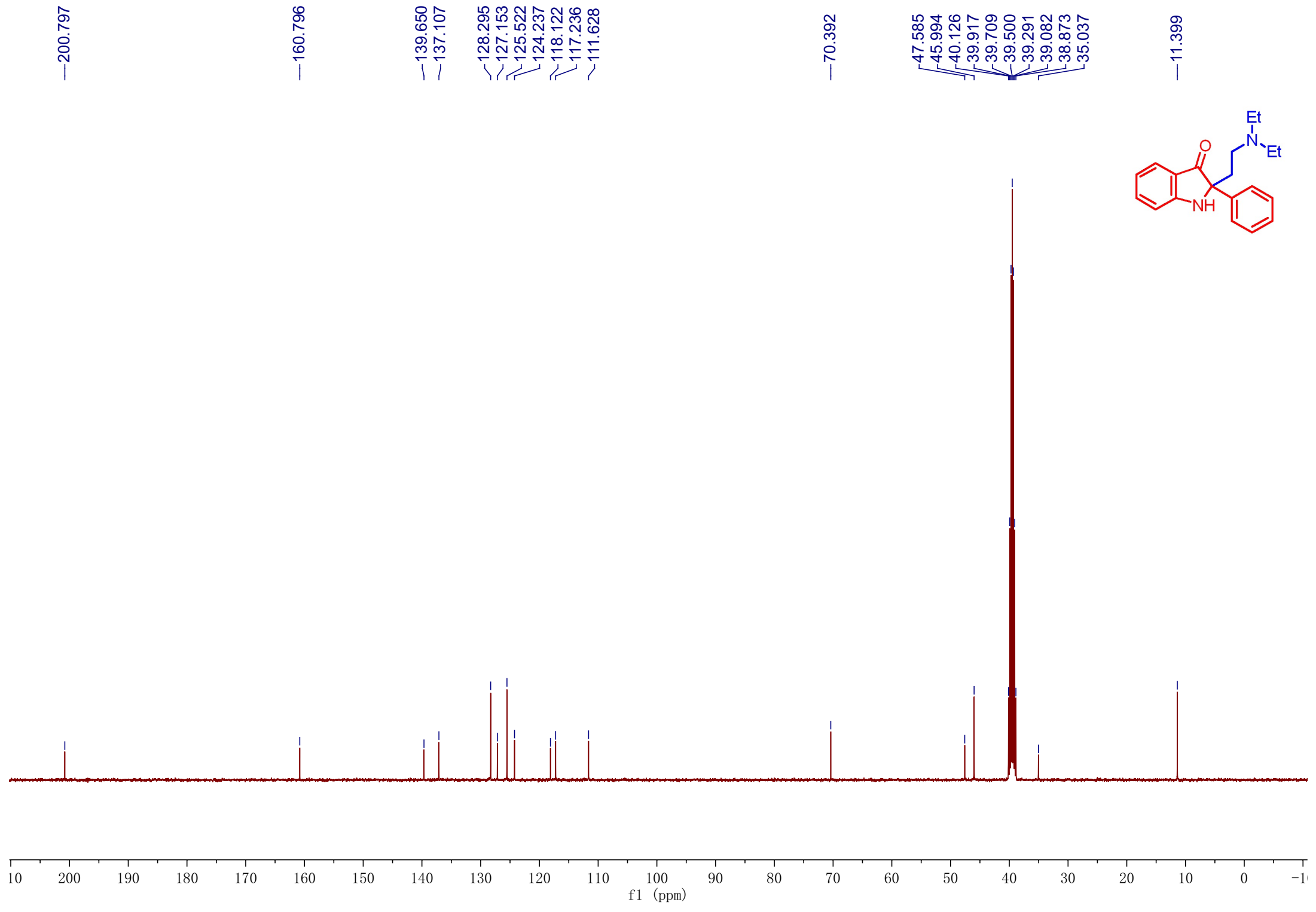
HRMS (ESI) m/z calcd. for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{S}$ ($\text{M}+\text{H}$) $^+$: 325.1733, found 325.1731.

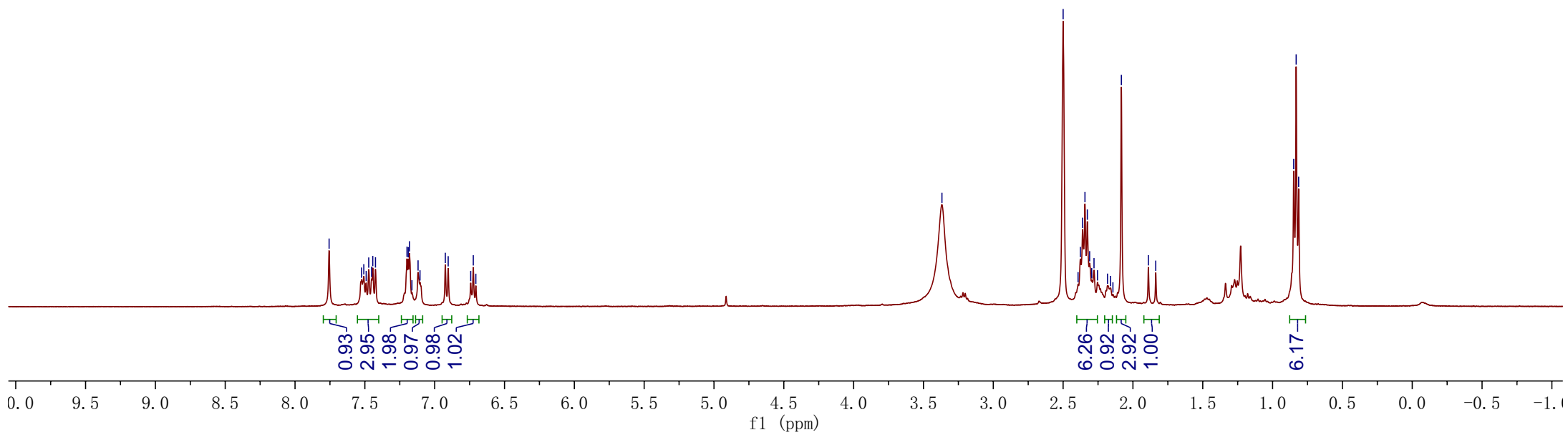
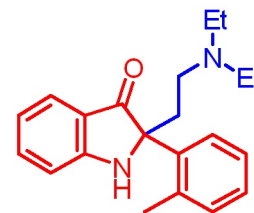
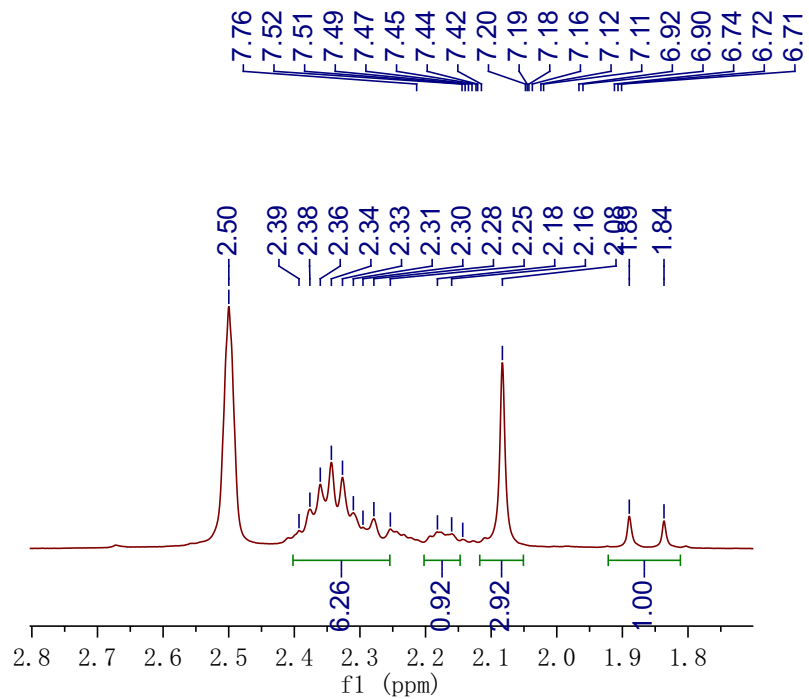
4. Reference

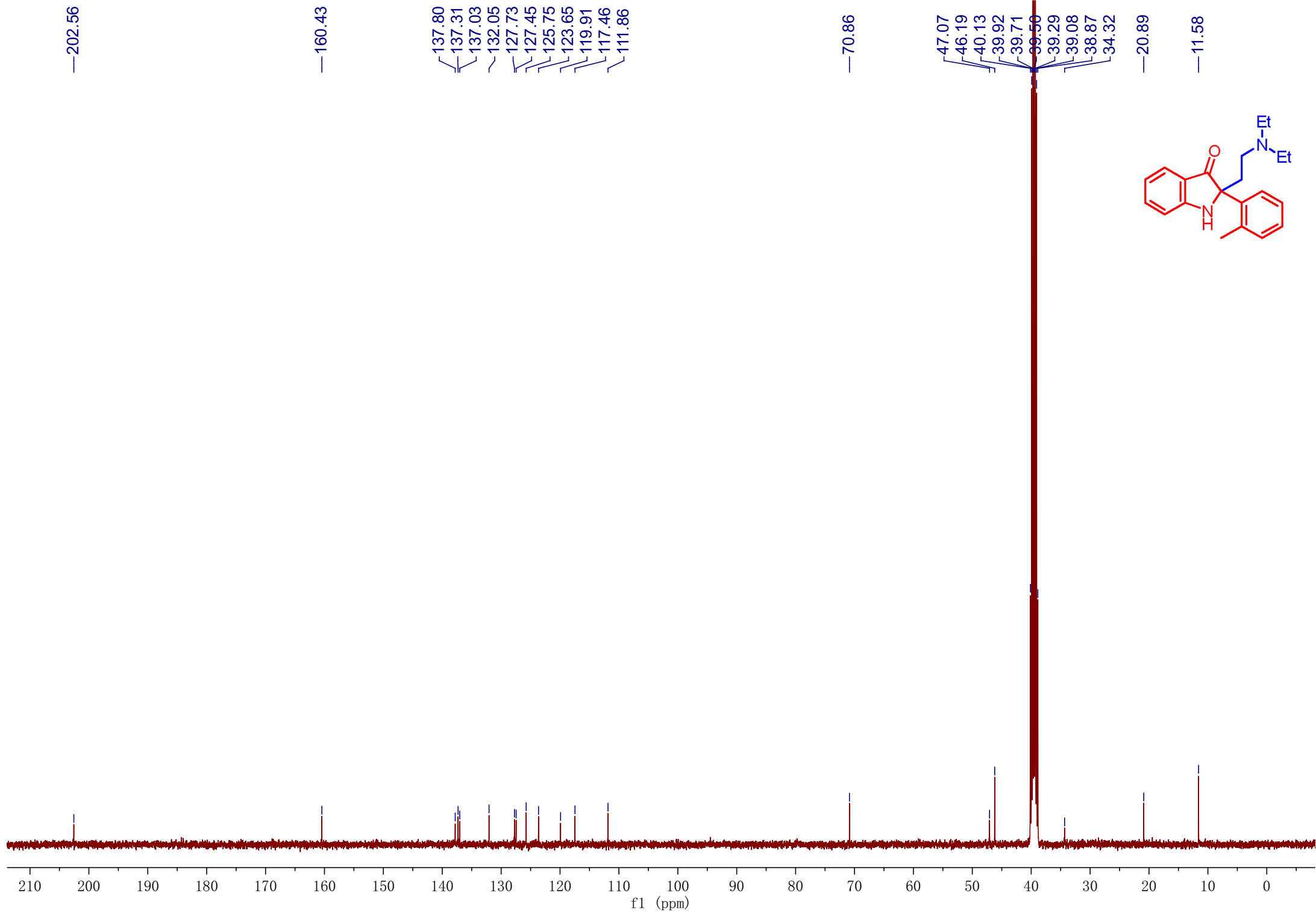
- [1]. Lian, X.-L.; Lei, H.; Quan, X.-J.; Ren, Z.-H.; Wang, Y.-Y.; Guan, Z.-H. *Chem. Commun.* **2013**, 49, 8196.
- [2]. (a) Lindsay, A. C.; Leung, I. K. H.; Sperry, J. *Org. Lett.* **2016**, 18, 5404. (b) Ling, K.-Q. *Synth. Commun.* **1995**, 25, 3831. (c) Liu, J.-X.; Zhou, Q.-Q.; Deng, J.-G.; Chen, Y.-C. *Org. Biomol. Chem.* **2013**, 11, 8175. (d) Najahi, E.; Valentin, A.; Fabre, P.-L.; Reybier, K.; Nepveu, F. *Eur. J. Med. Chem.* **2014**, 78, 269. (e) Huang, J.-R.; Qin, L.; Zhu, Y.-Q.; Song, Q.; Dong, L. *Chem. Commun.* **2015**, 51, 2844.

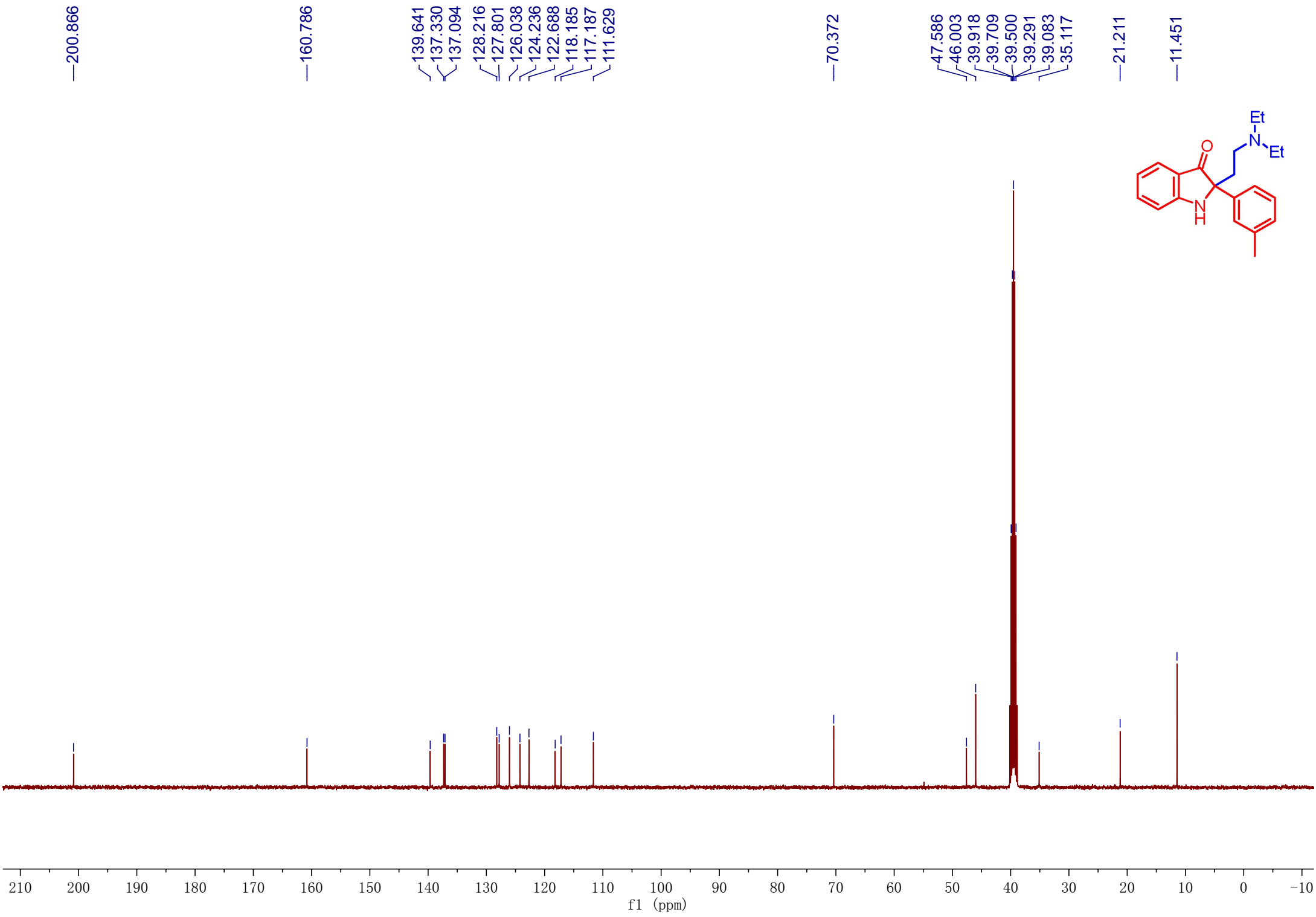
5. Copies of NMR spectra

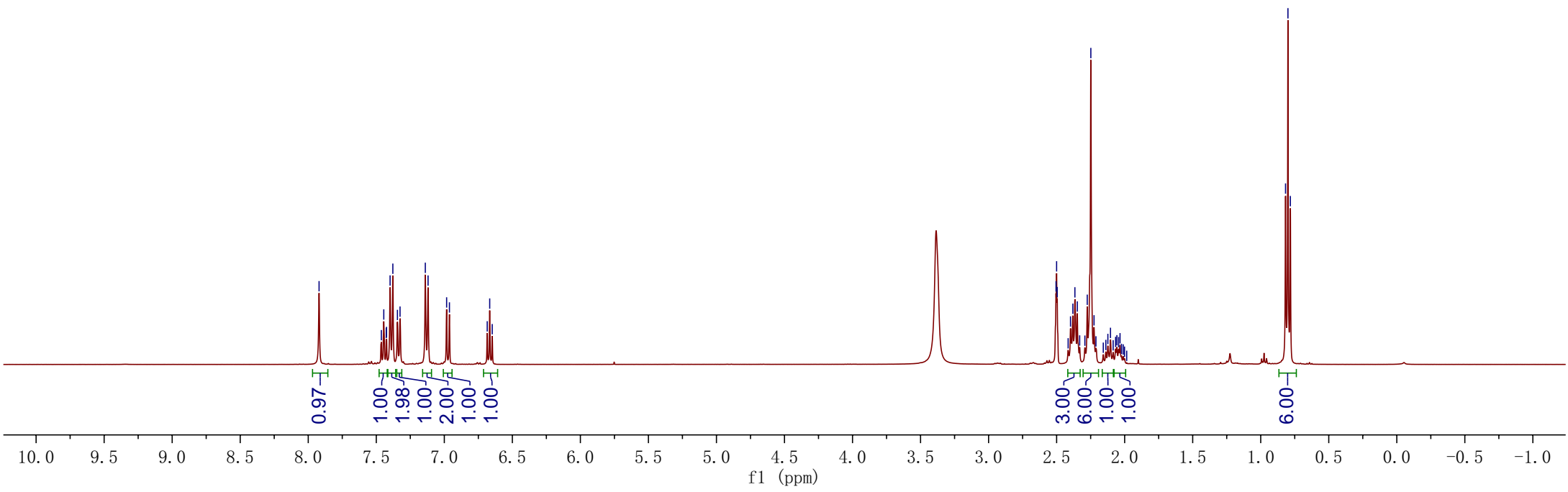
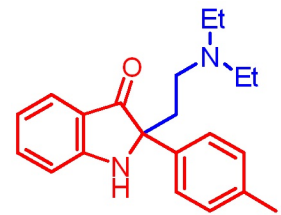
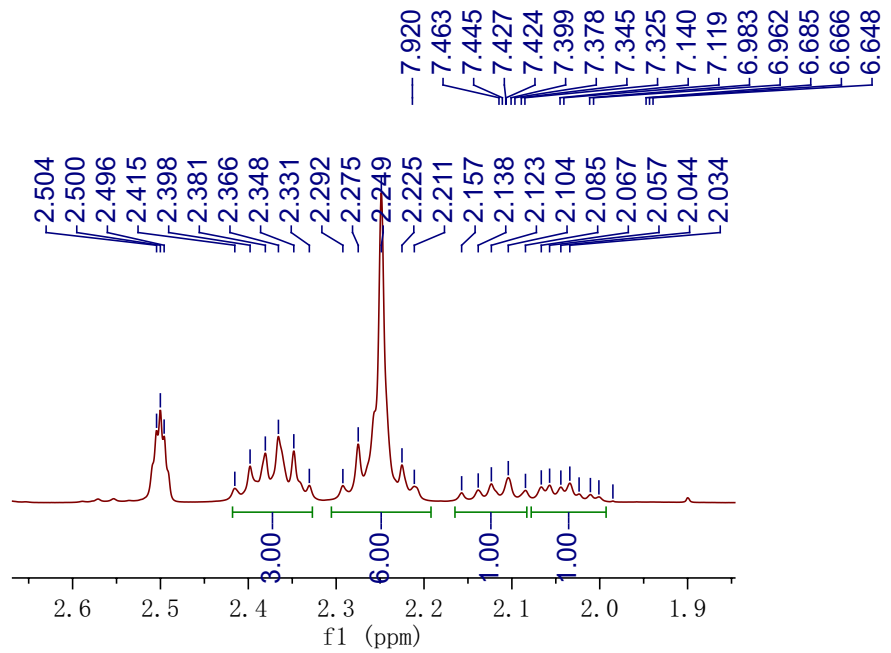


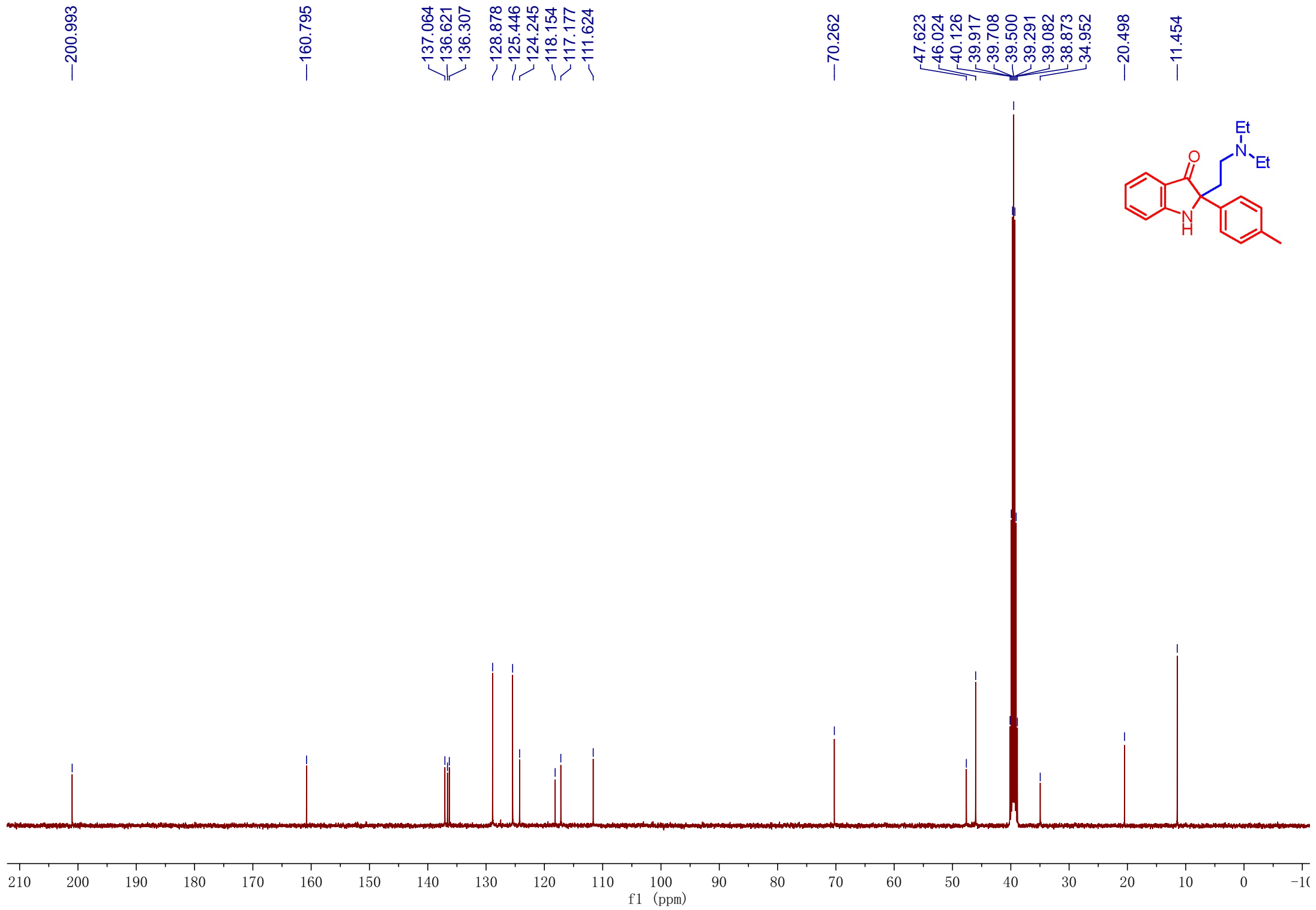


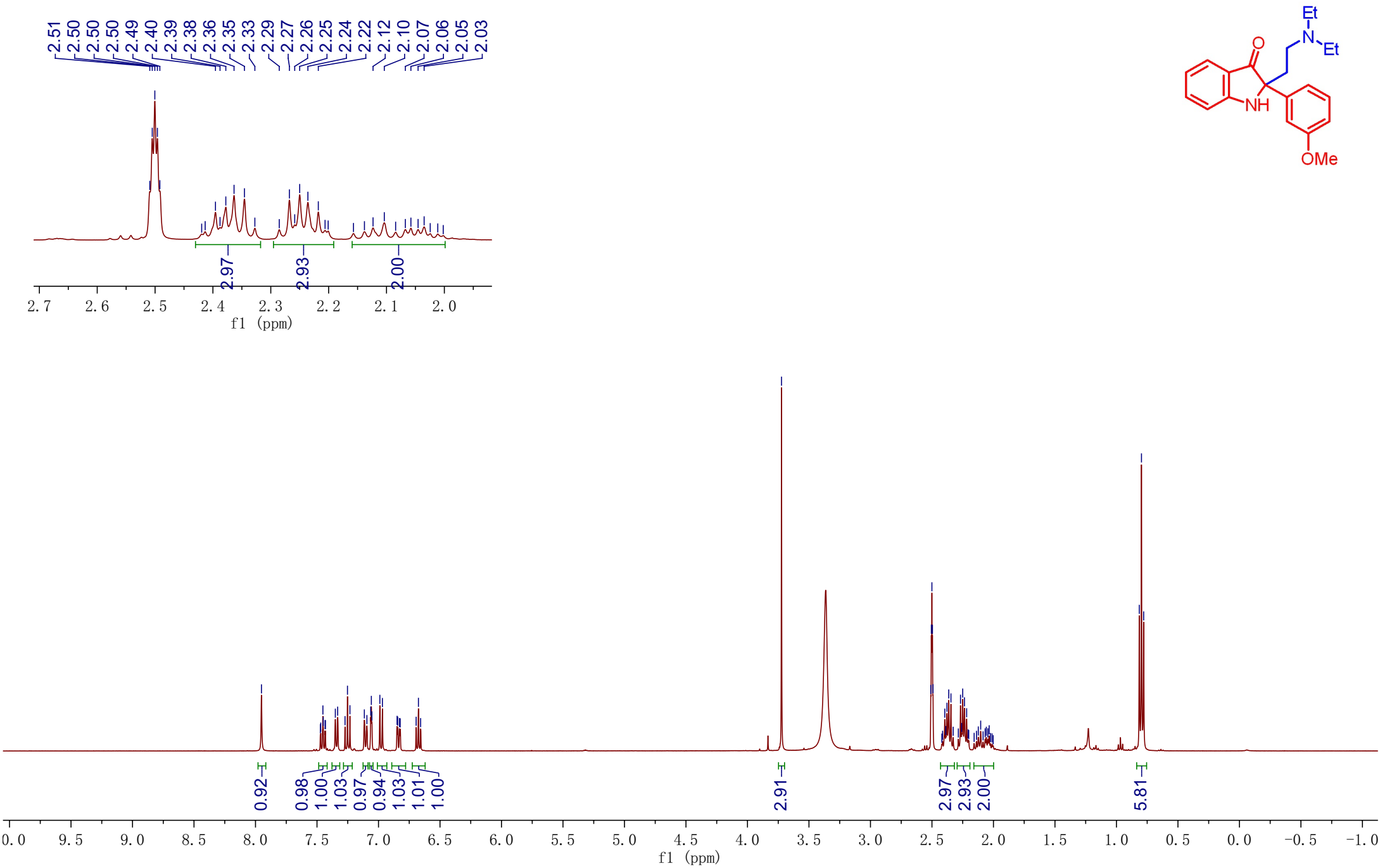






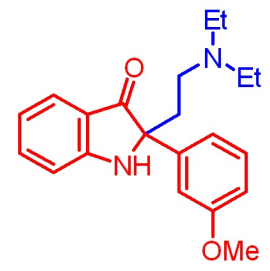


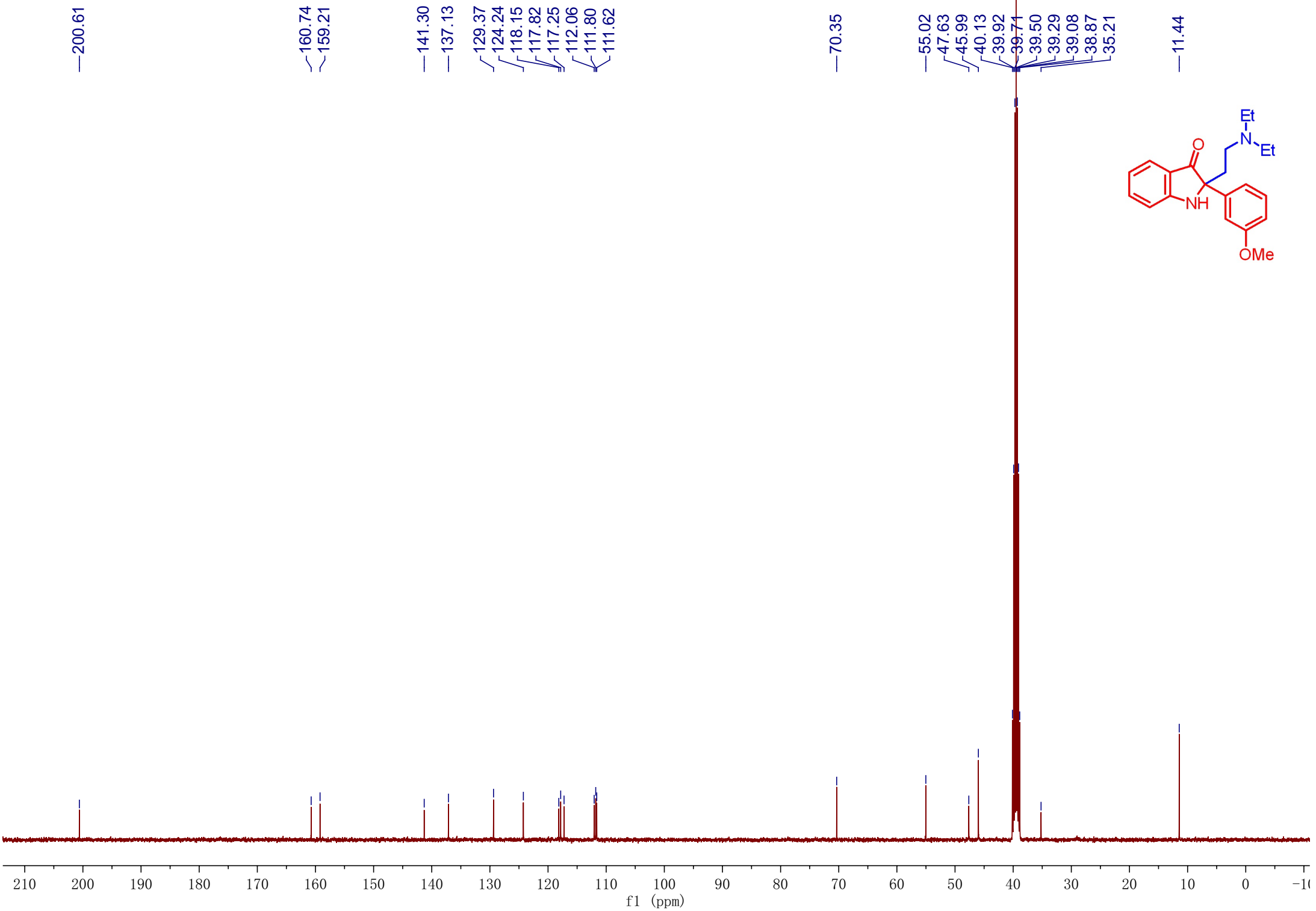


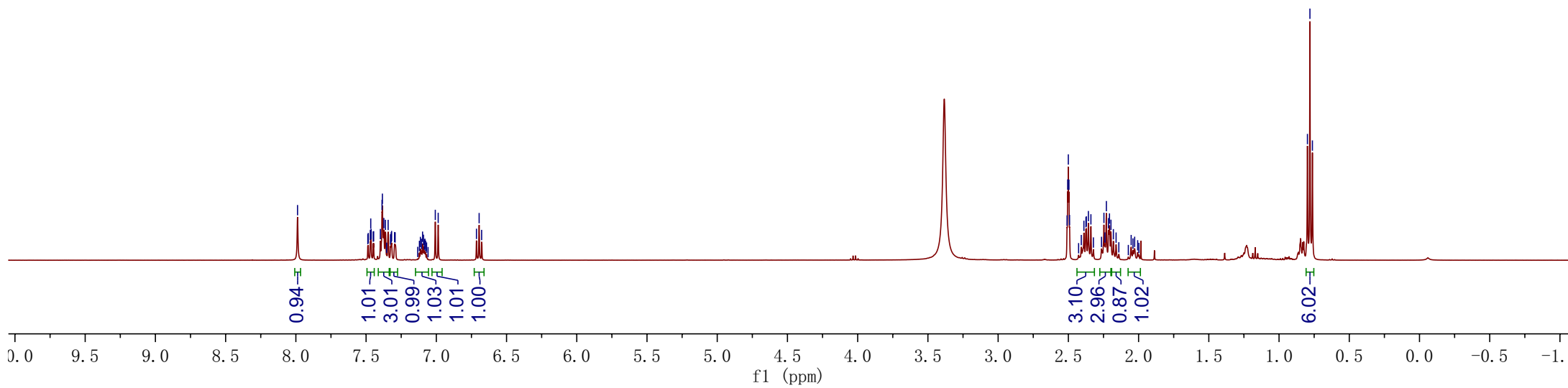
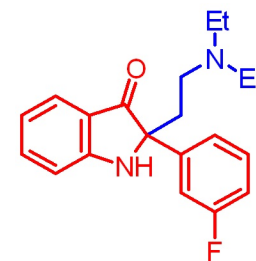
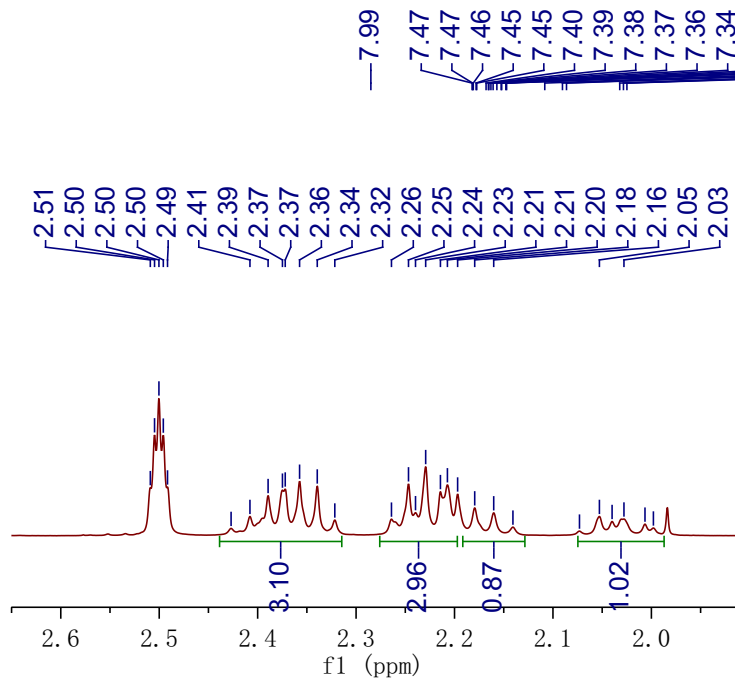


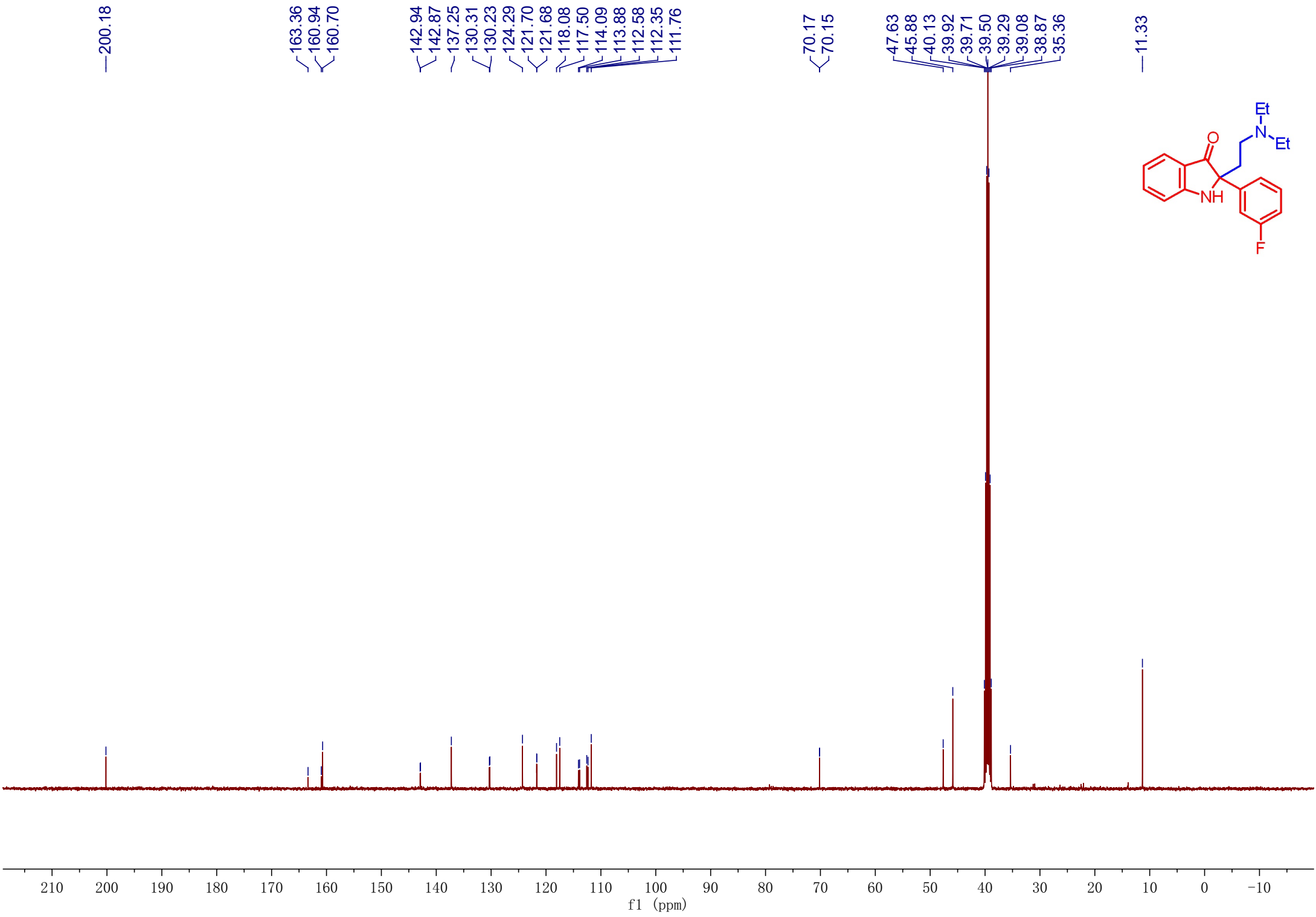
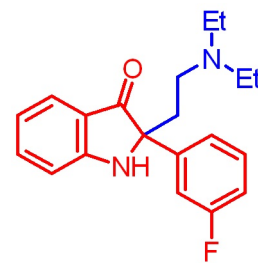
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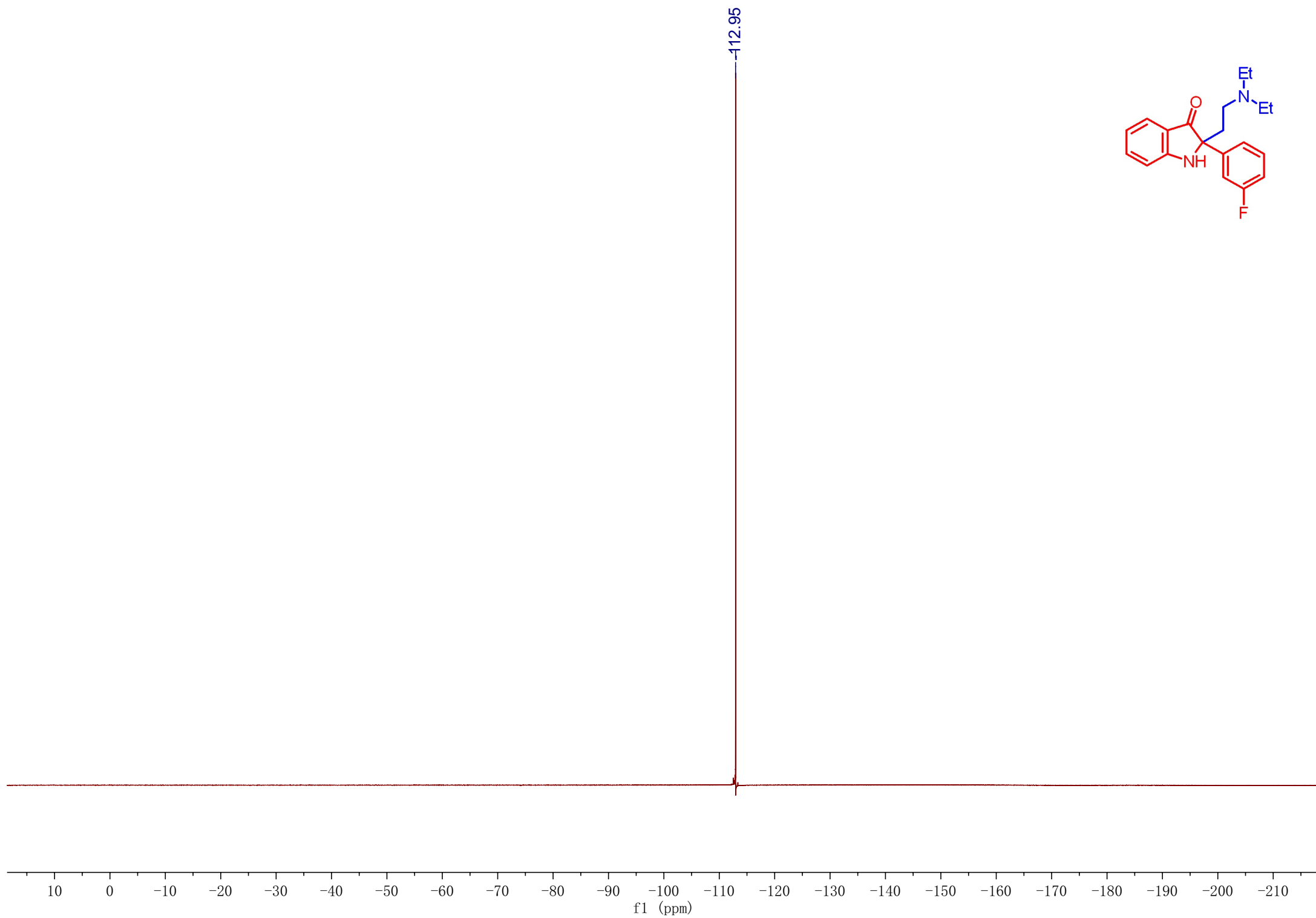
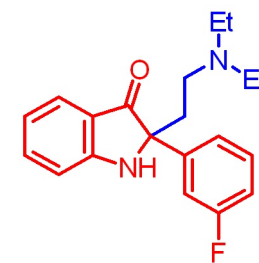
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0.80
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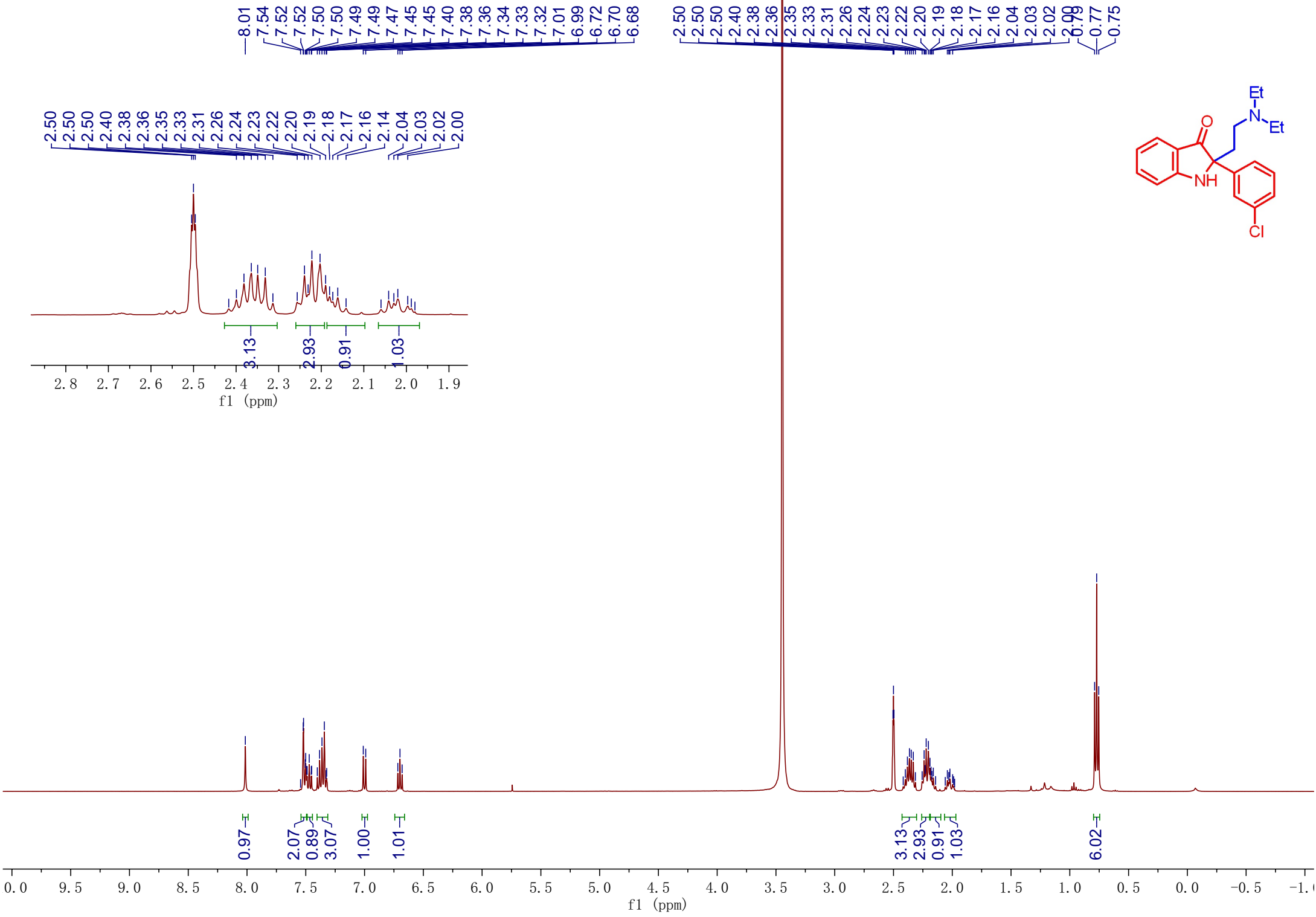


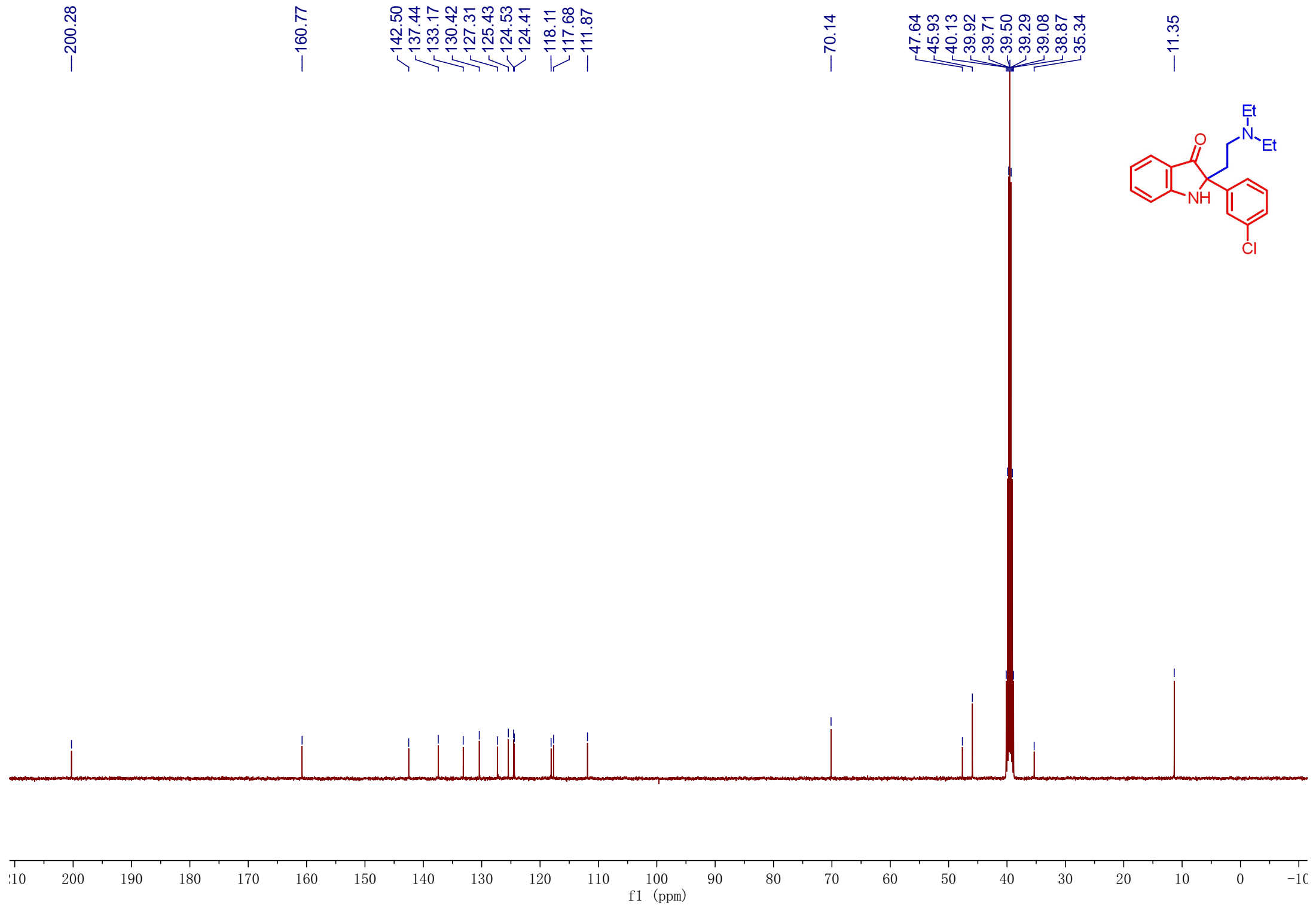


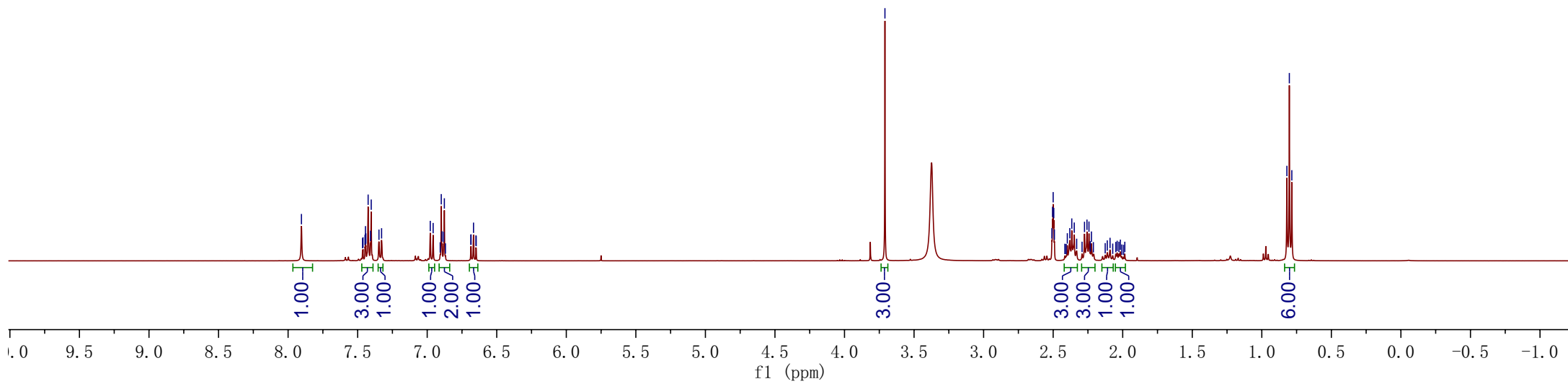
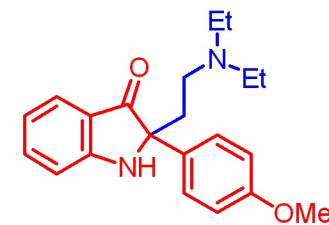
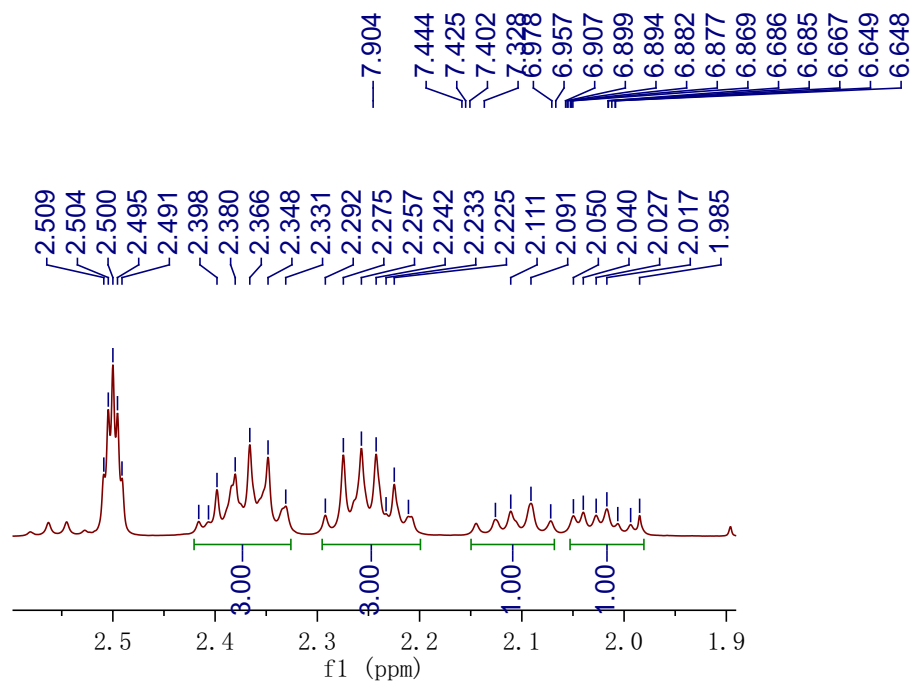


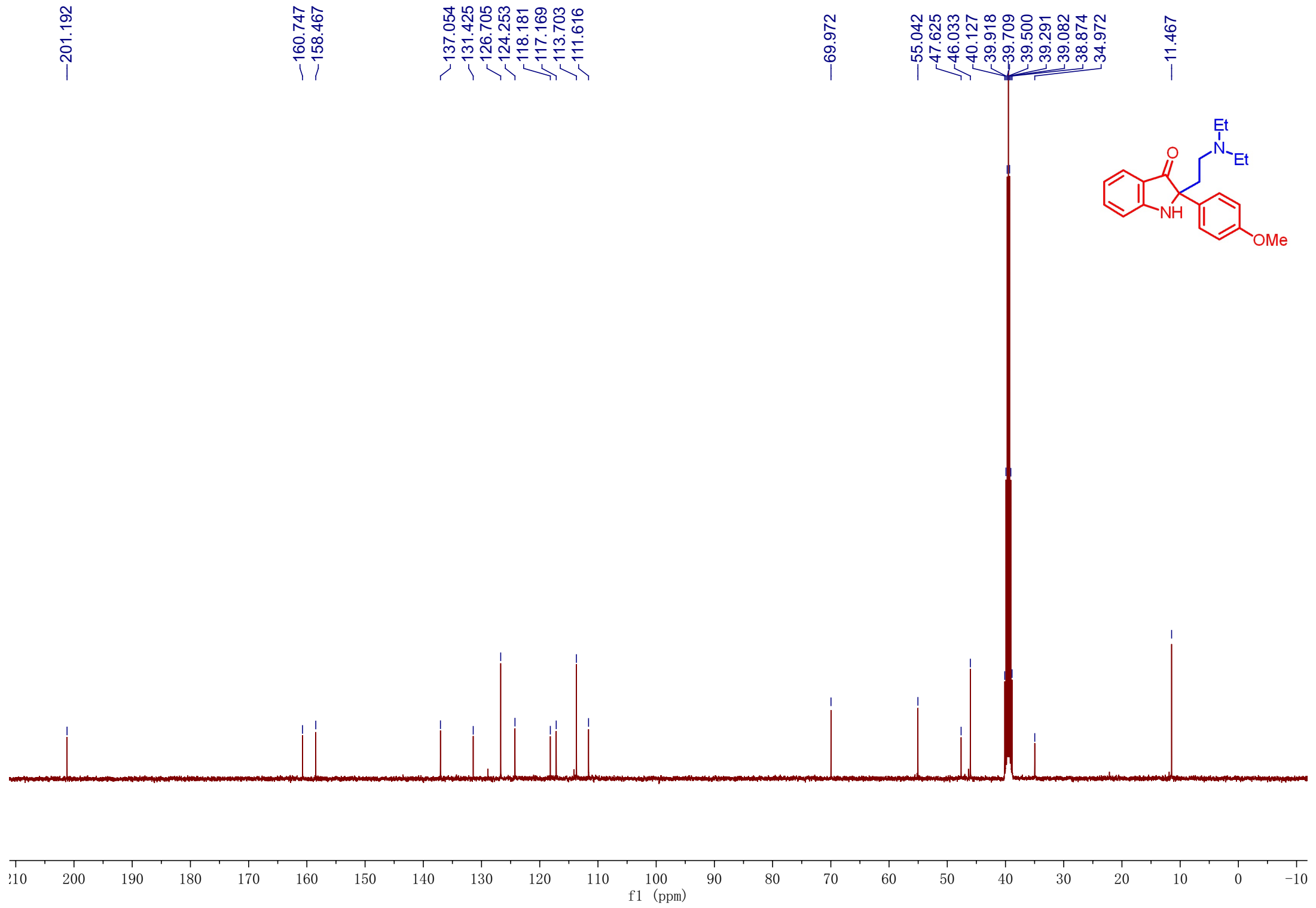


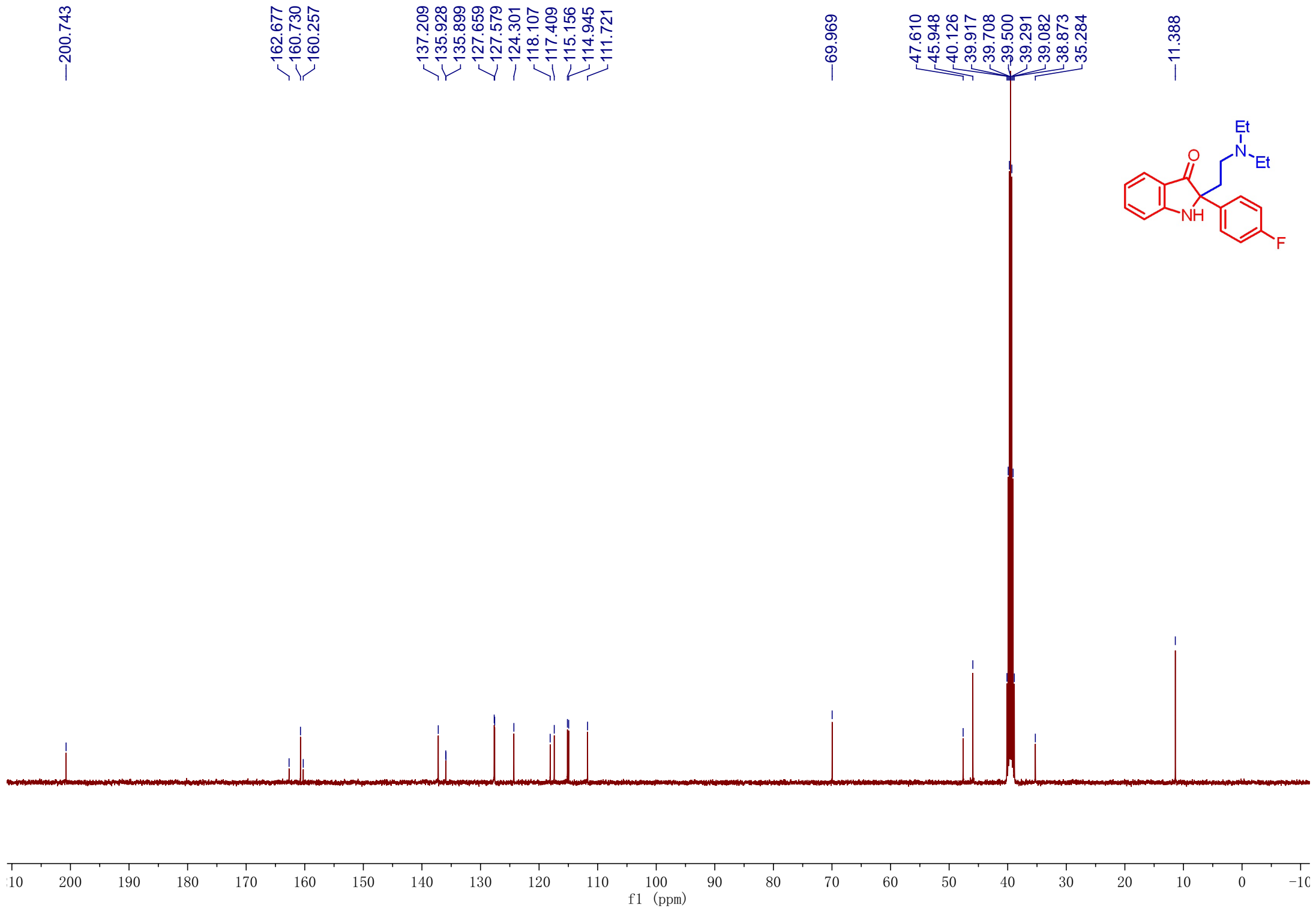


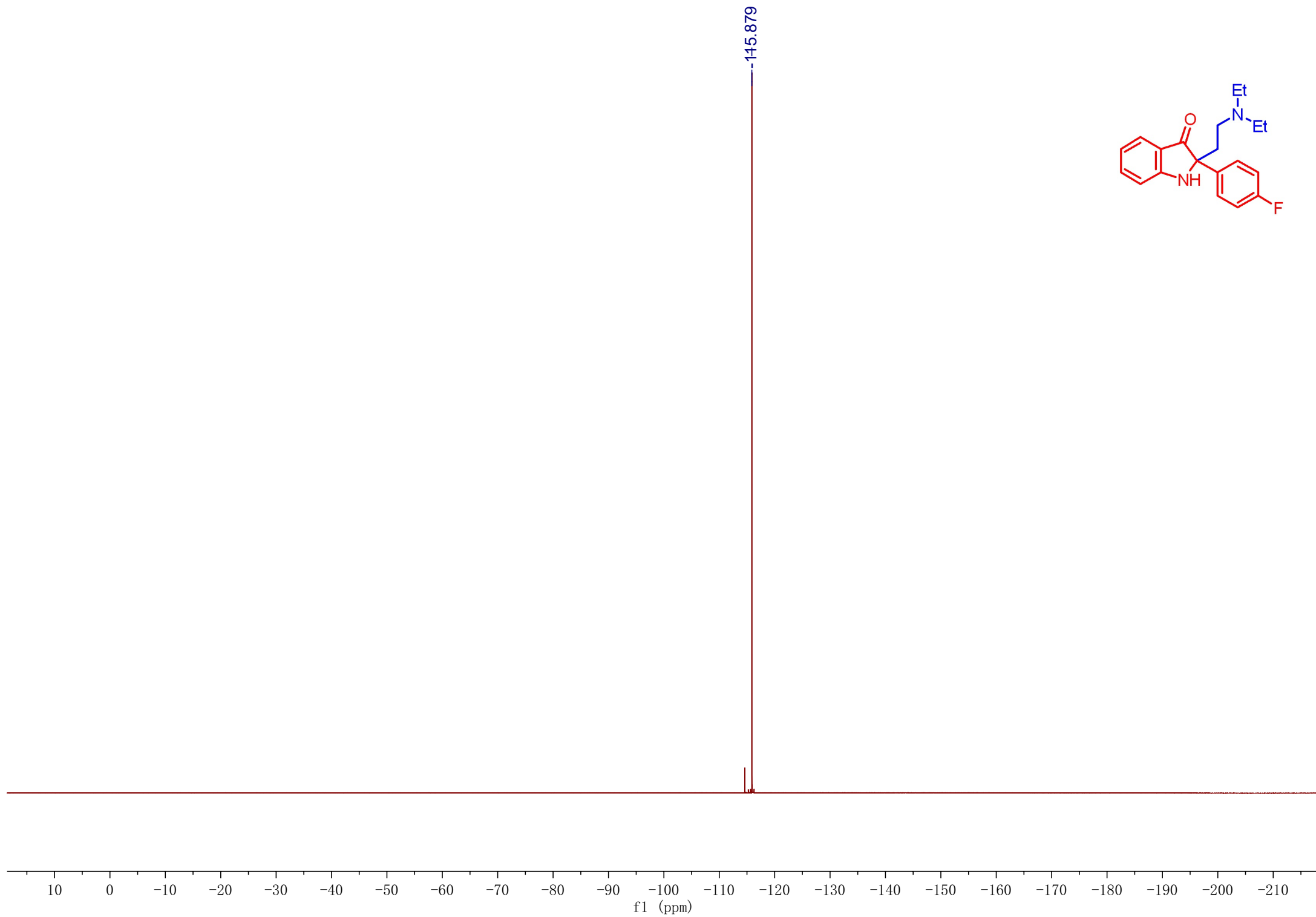
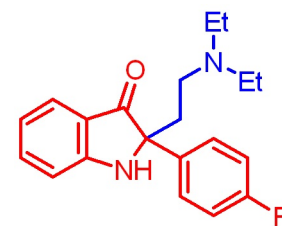


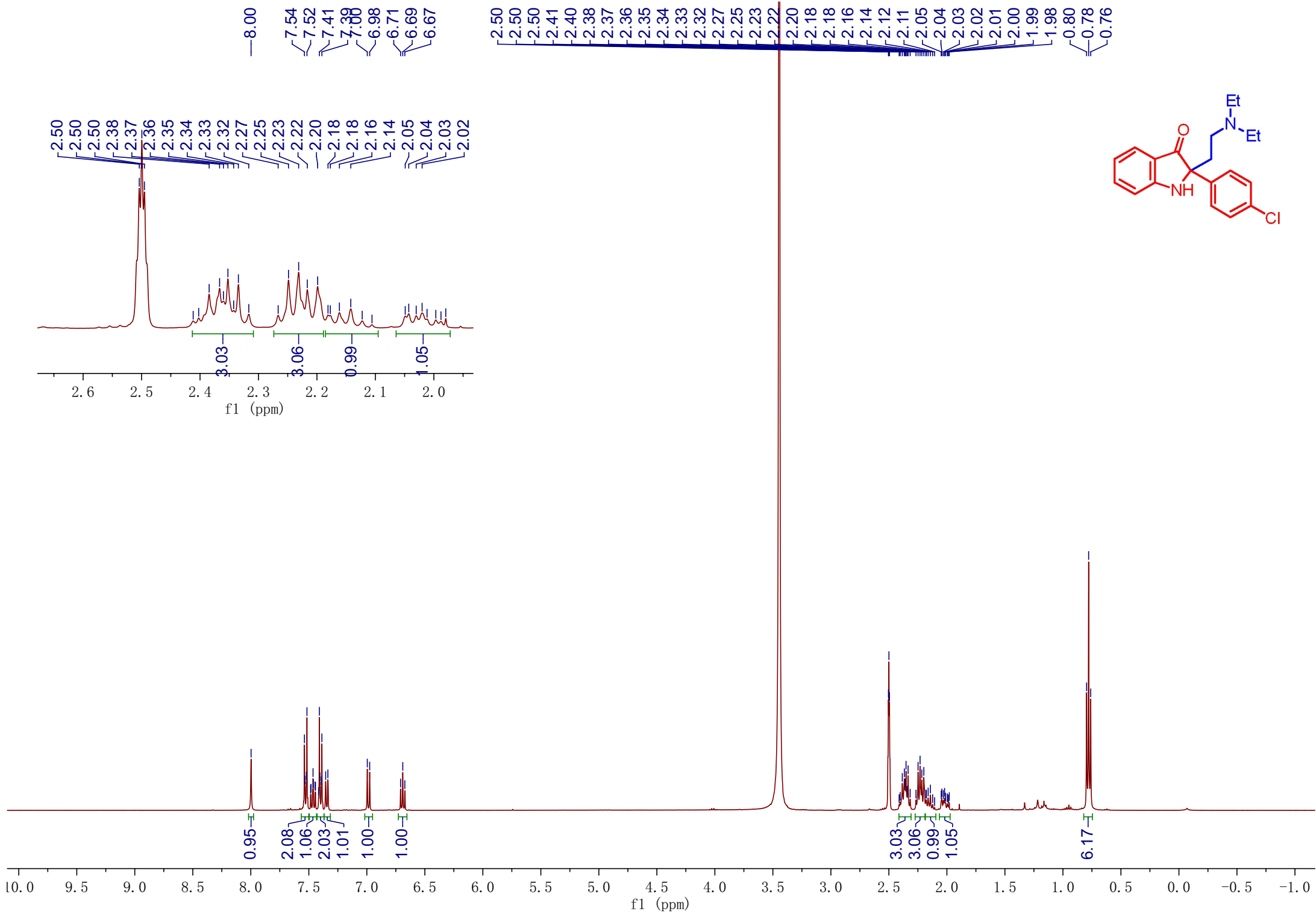


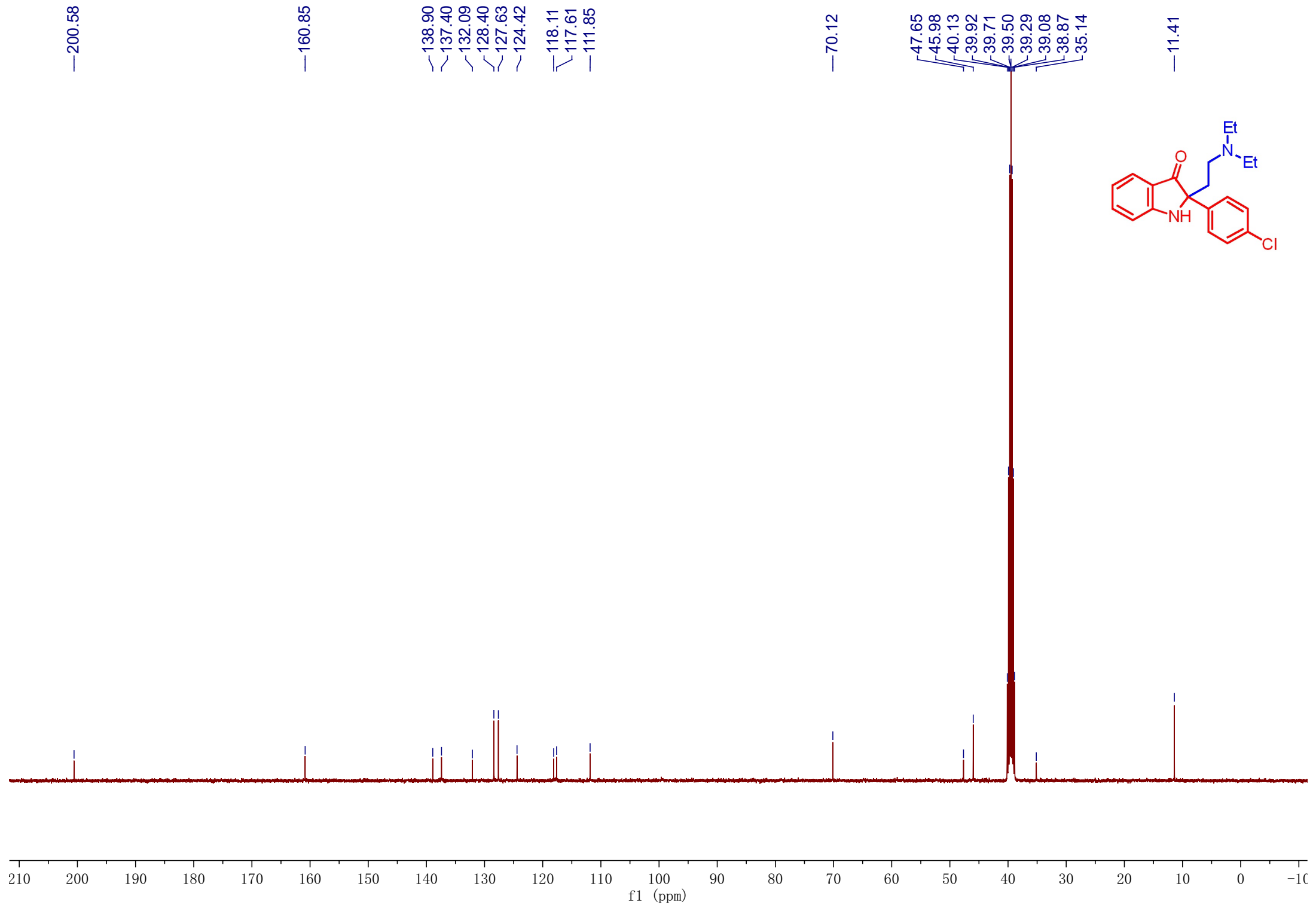


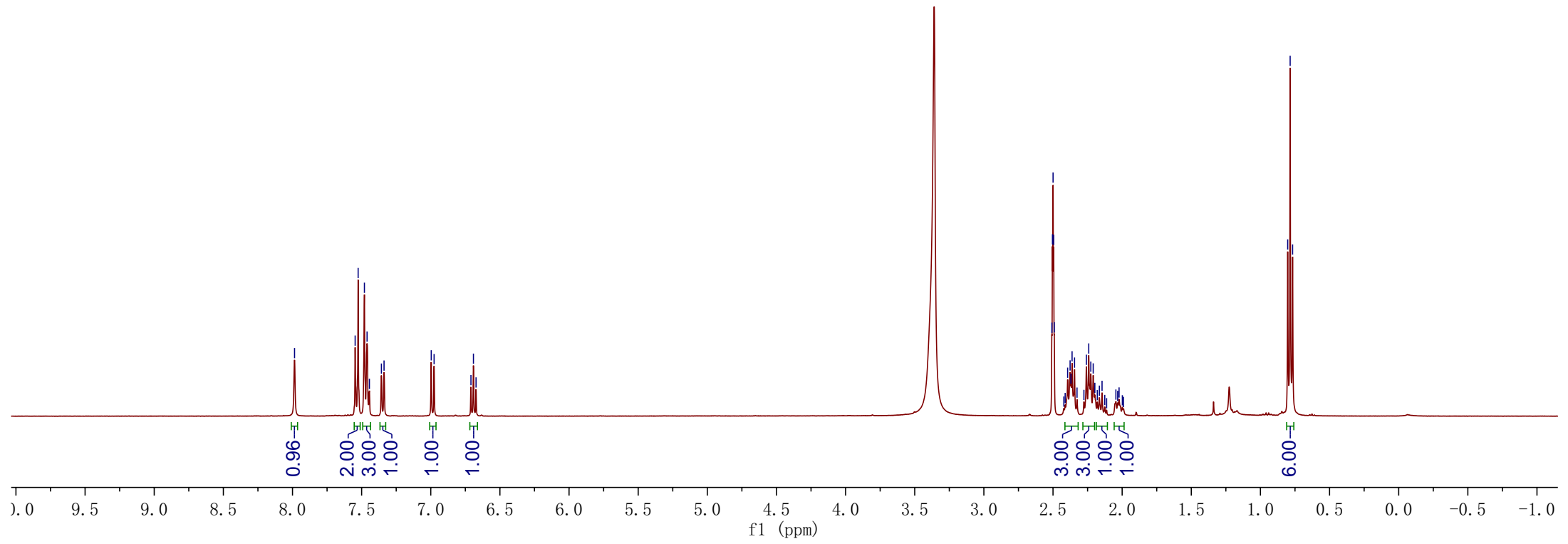
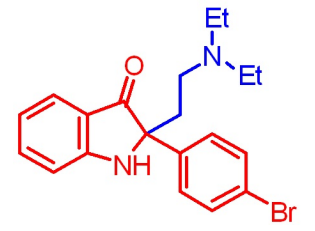
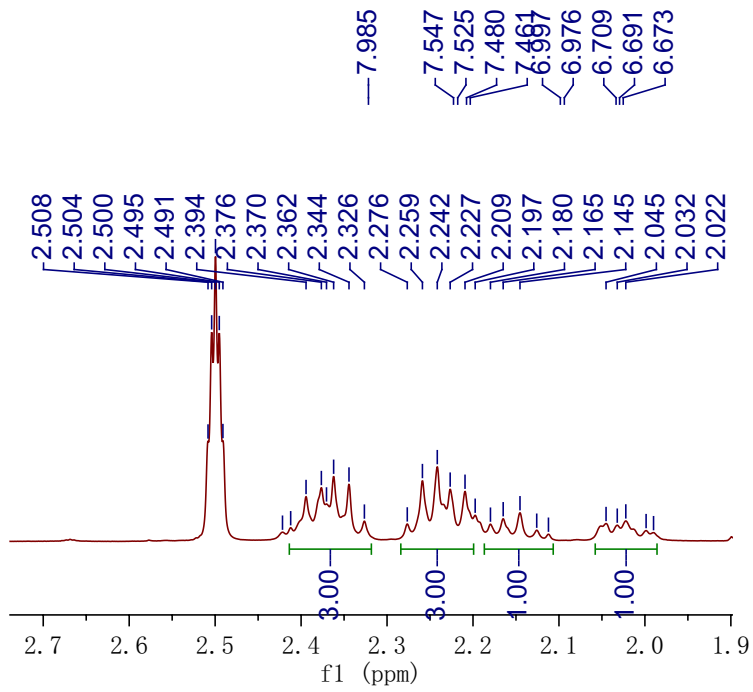


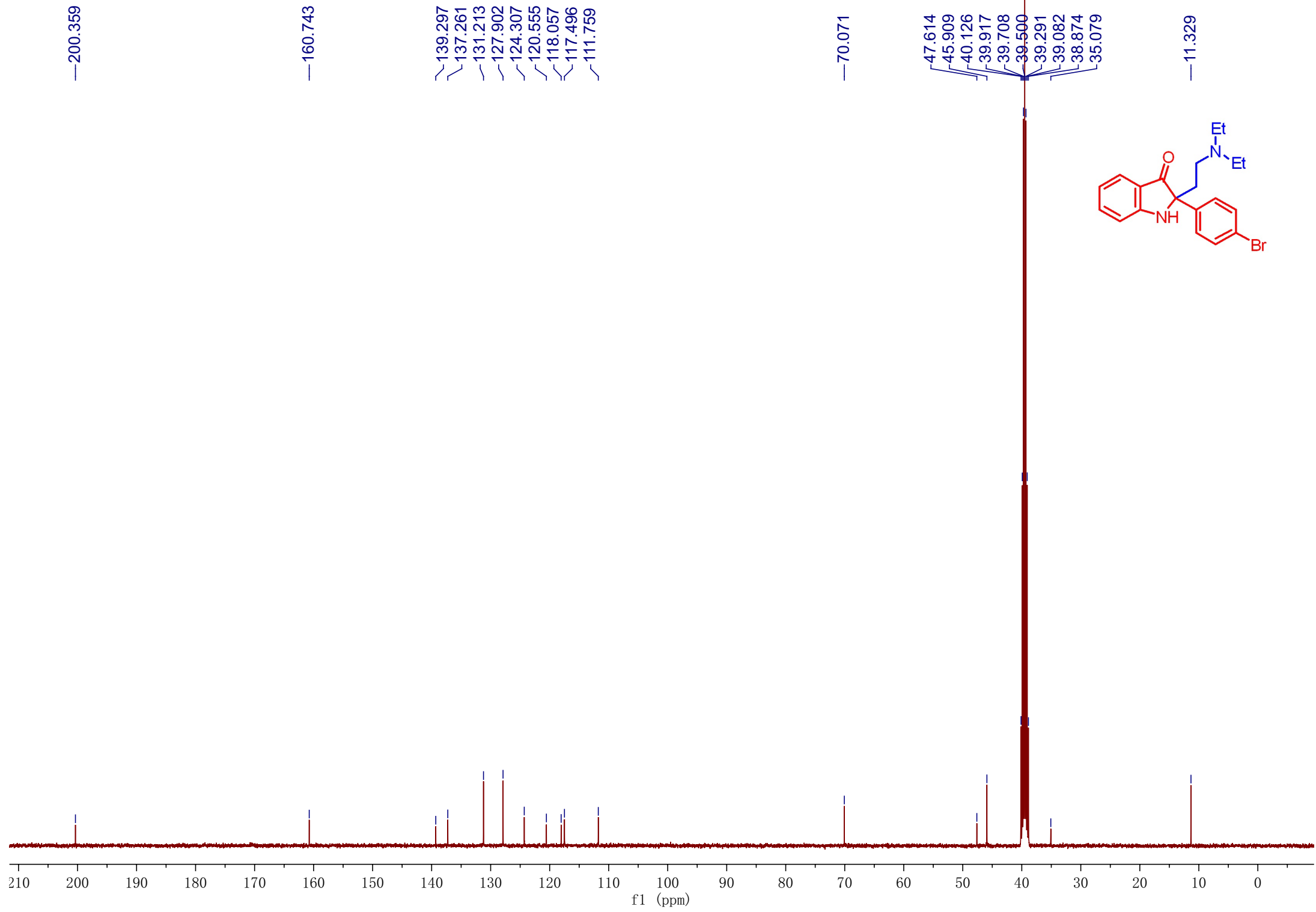


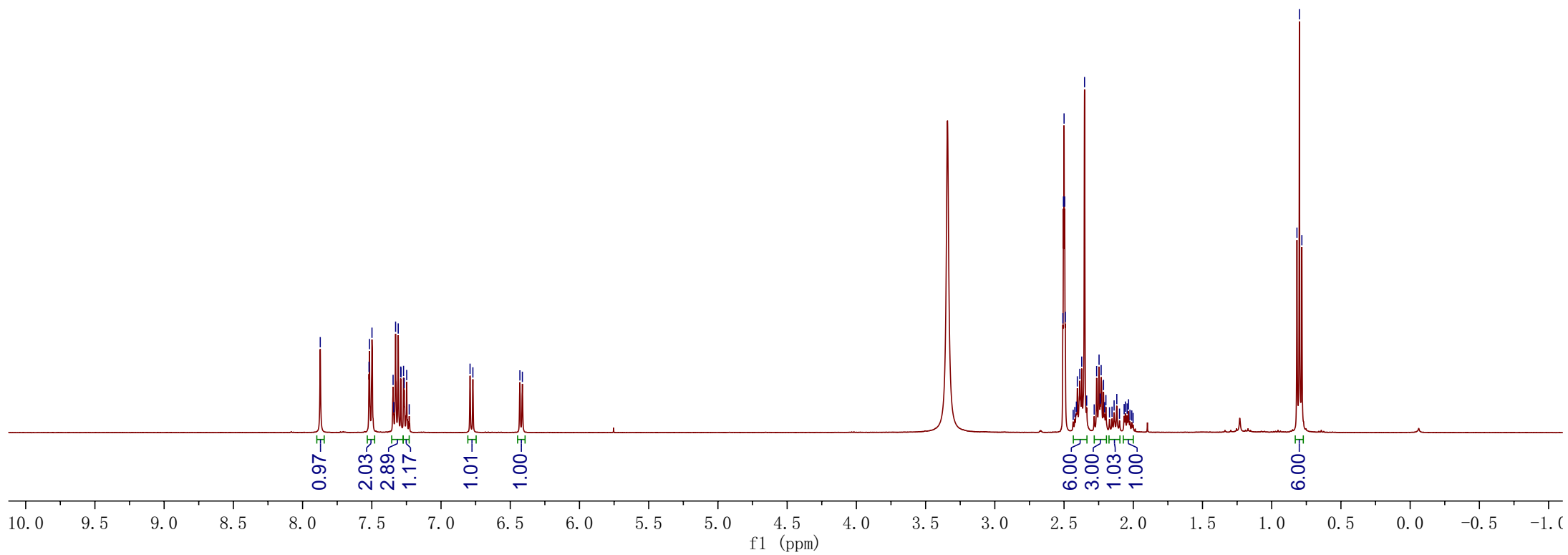
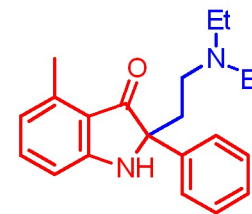
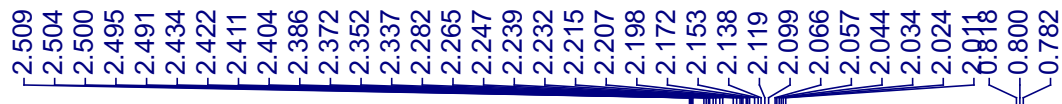
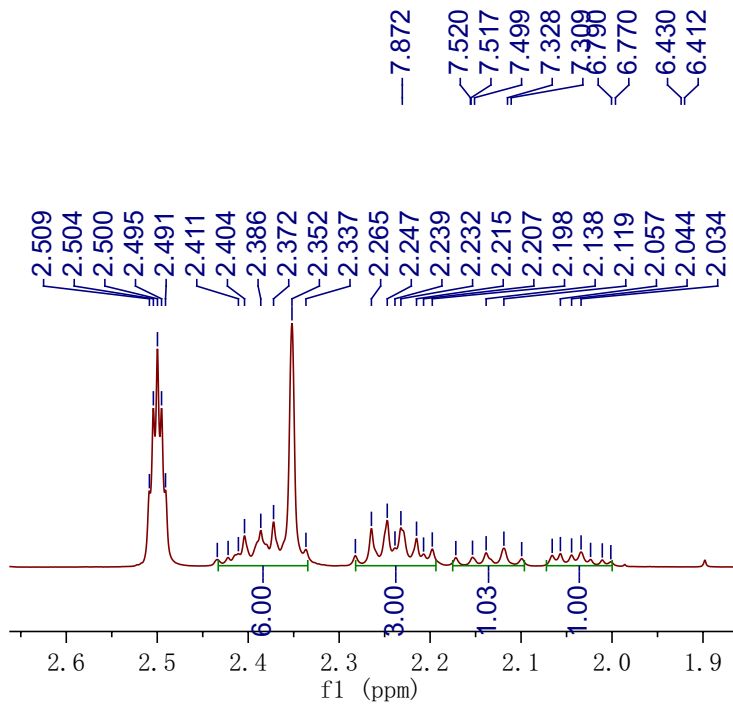


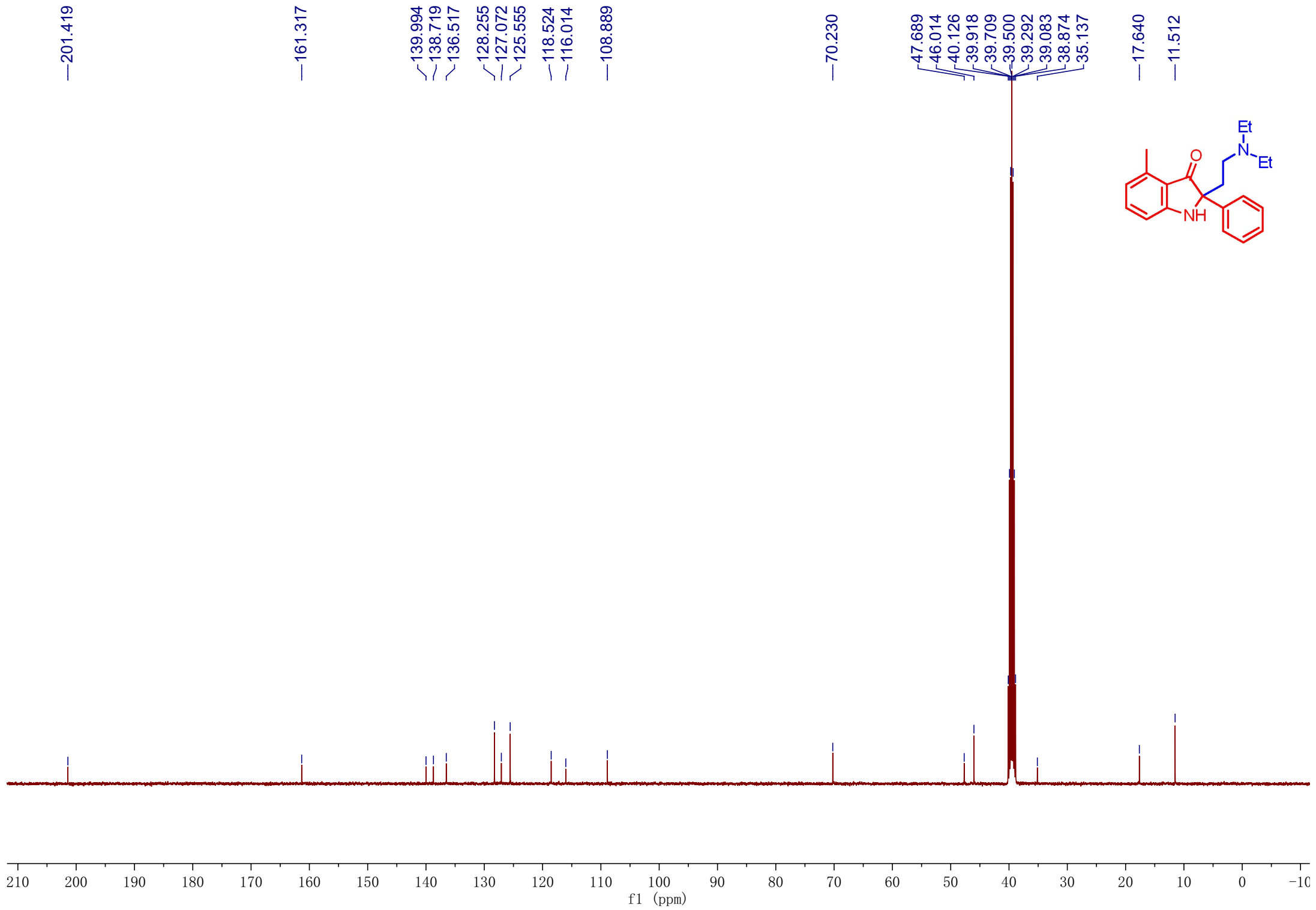


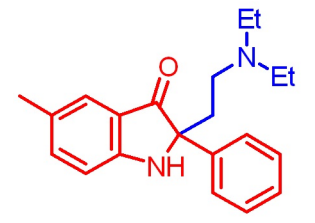
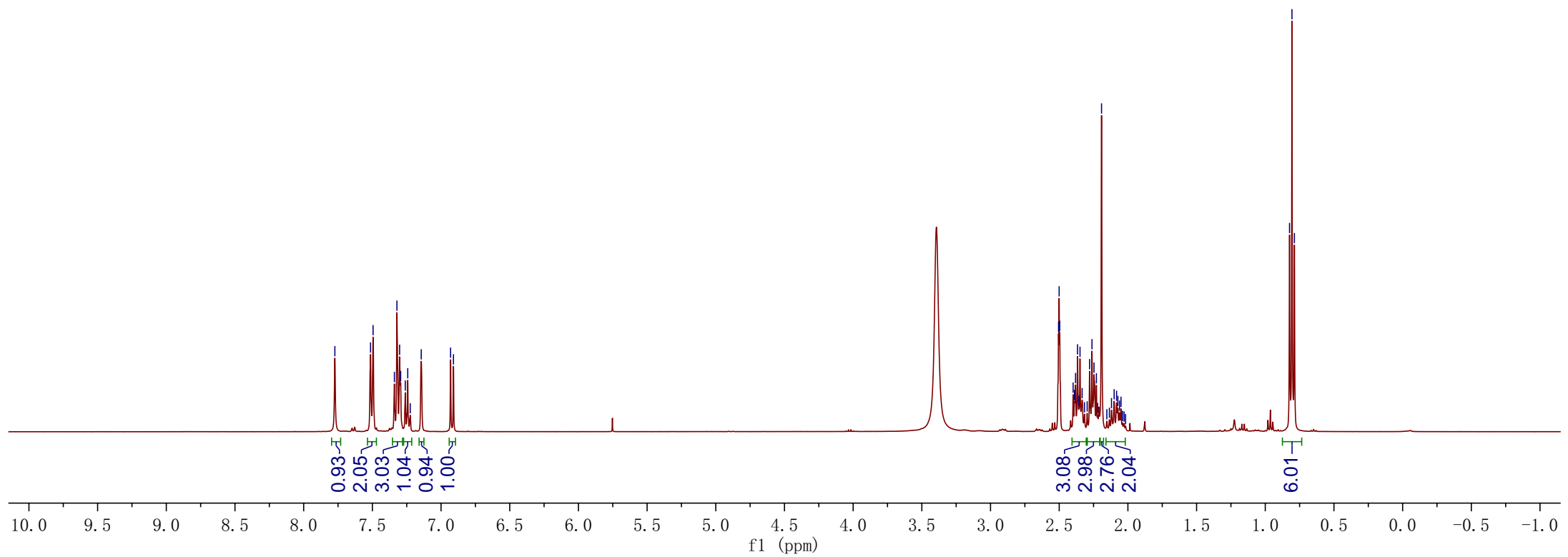
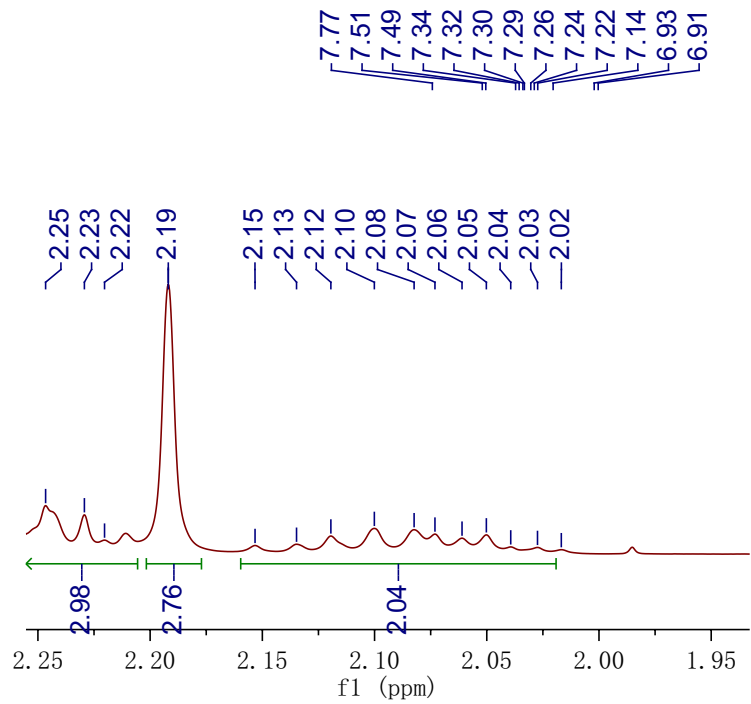


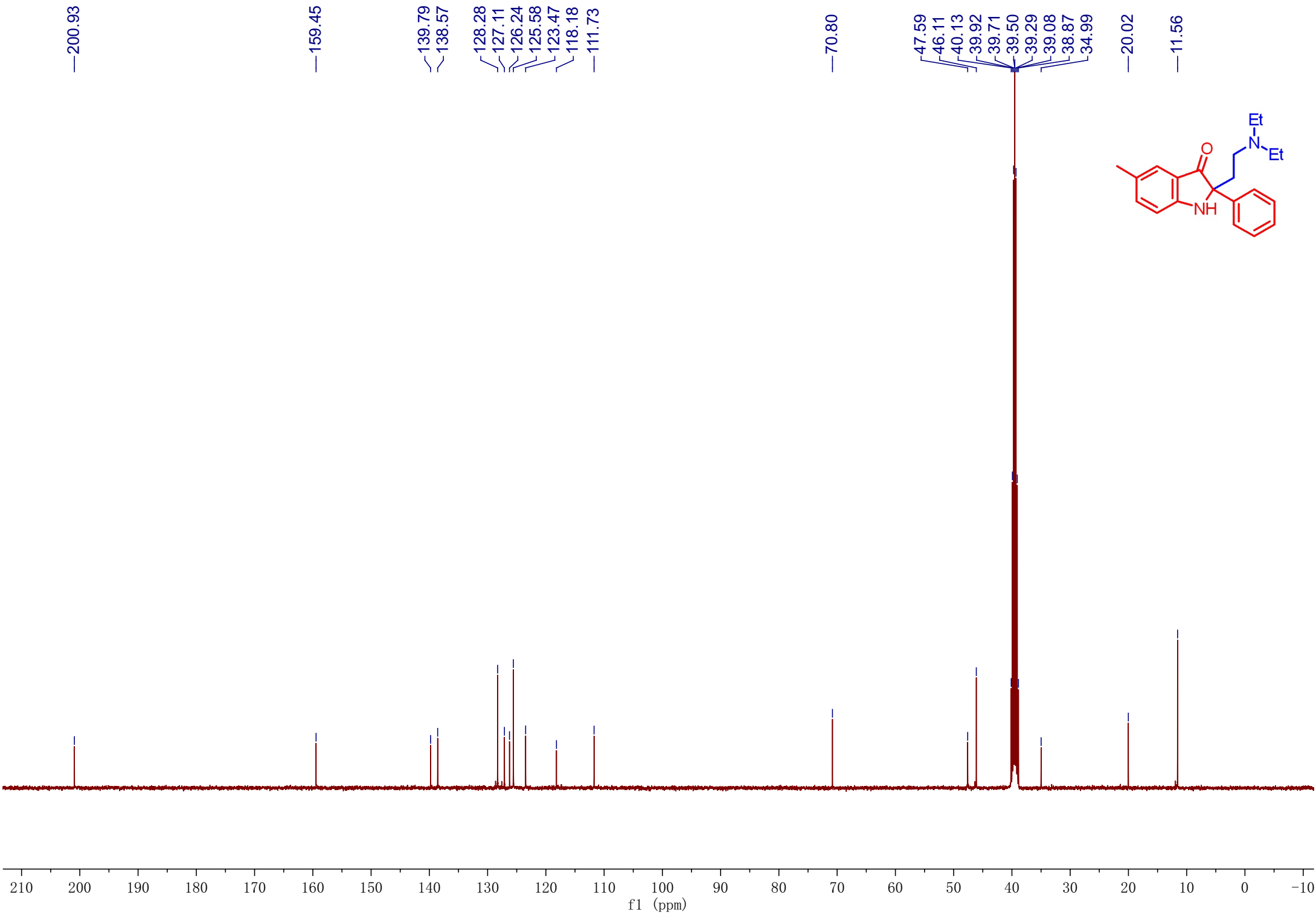


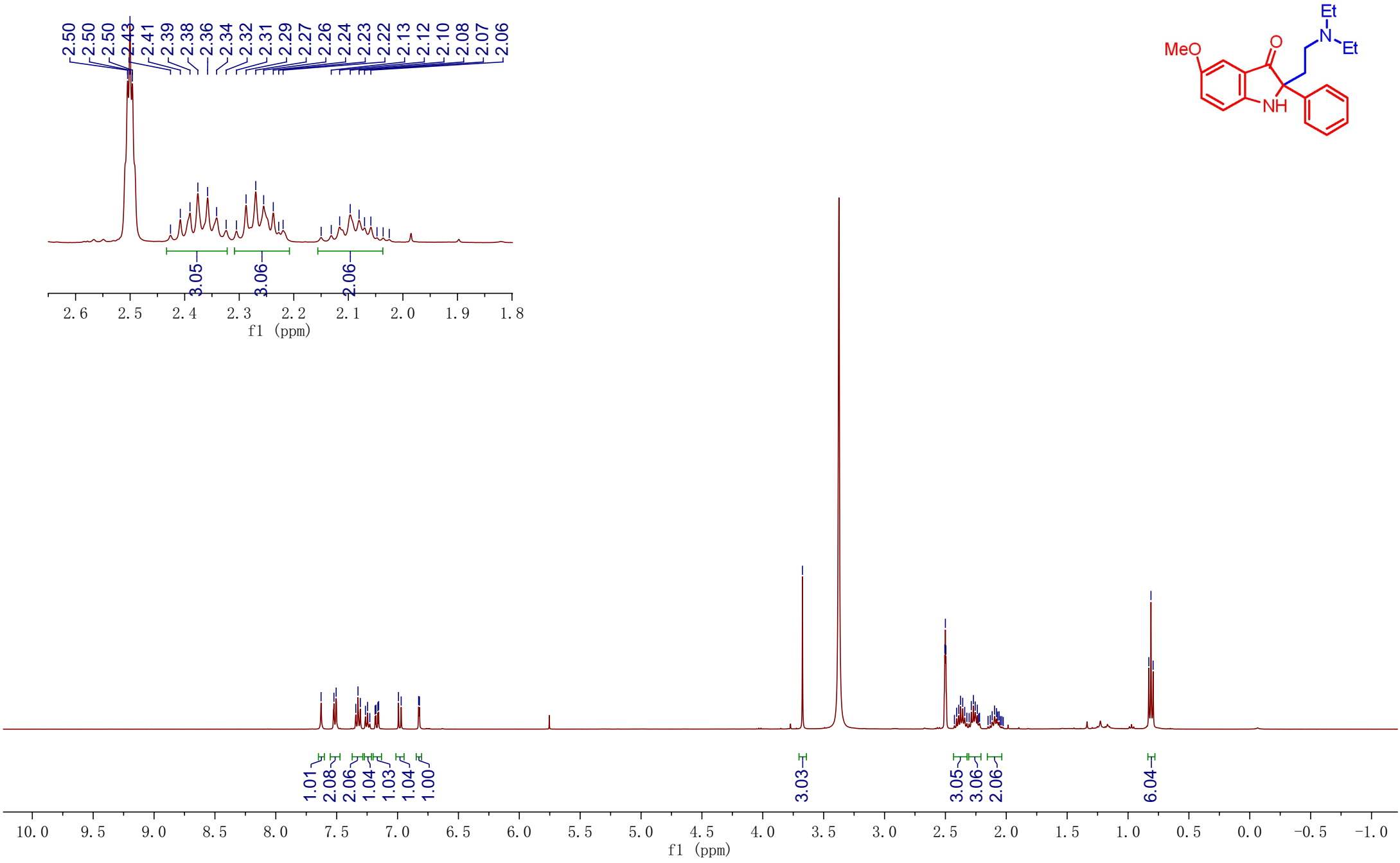


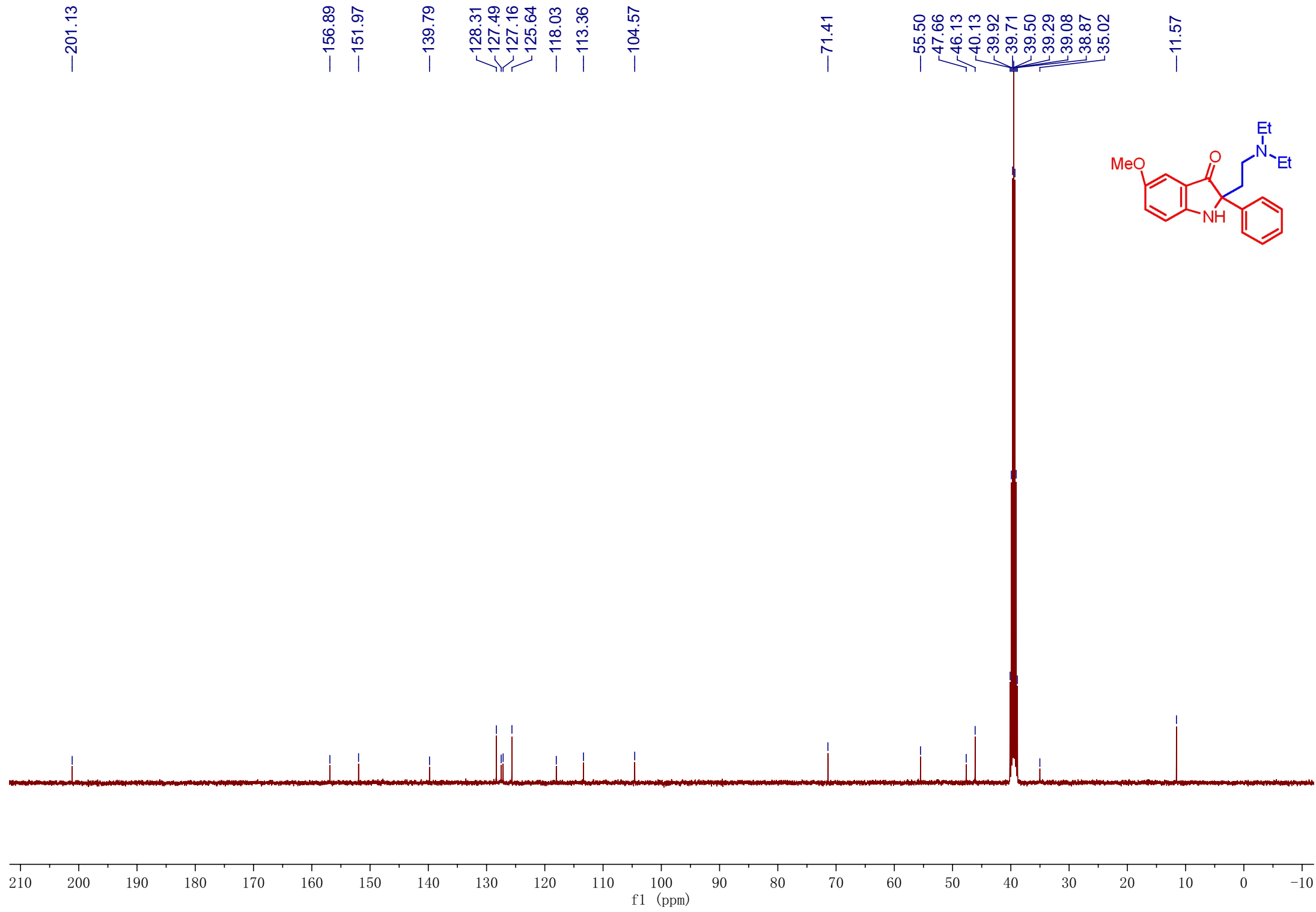








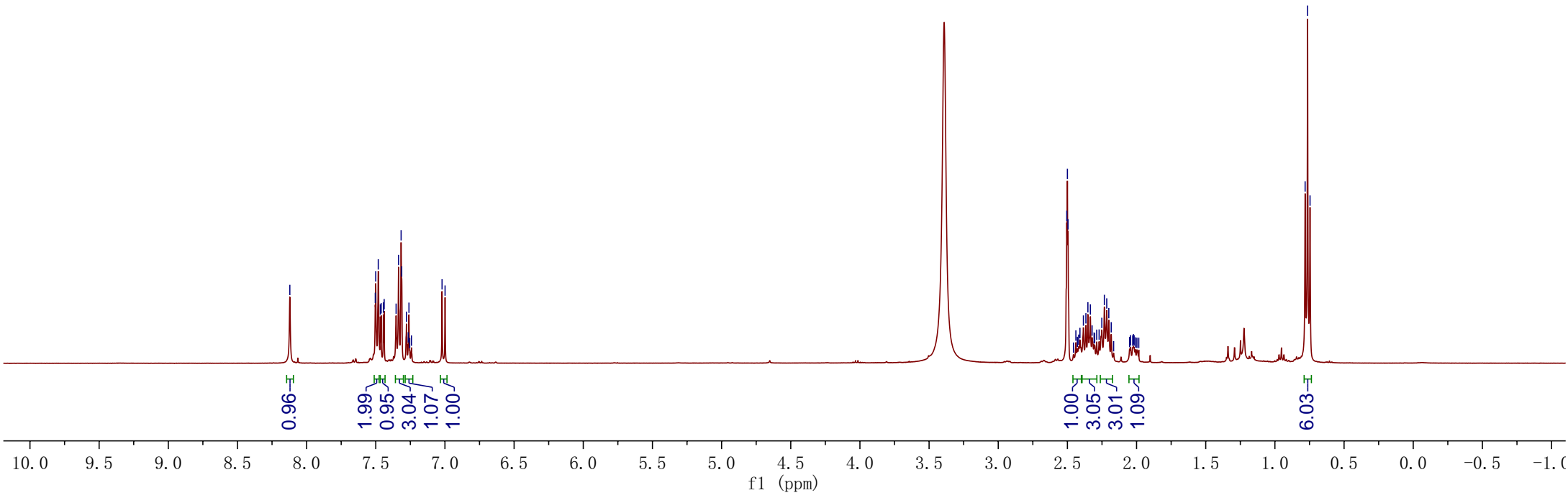
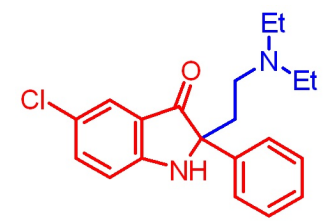
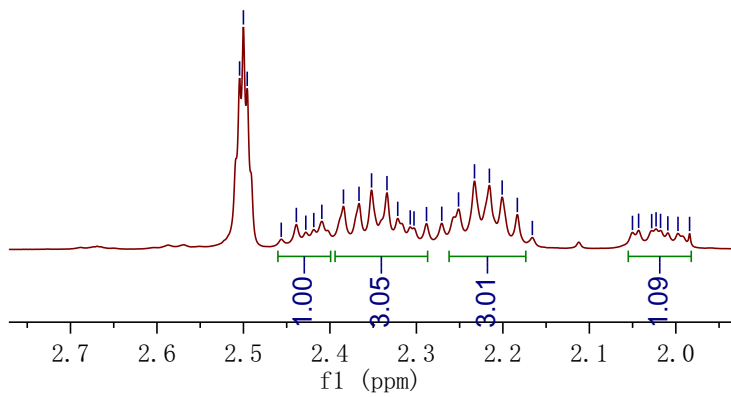


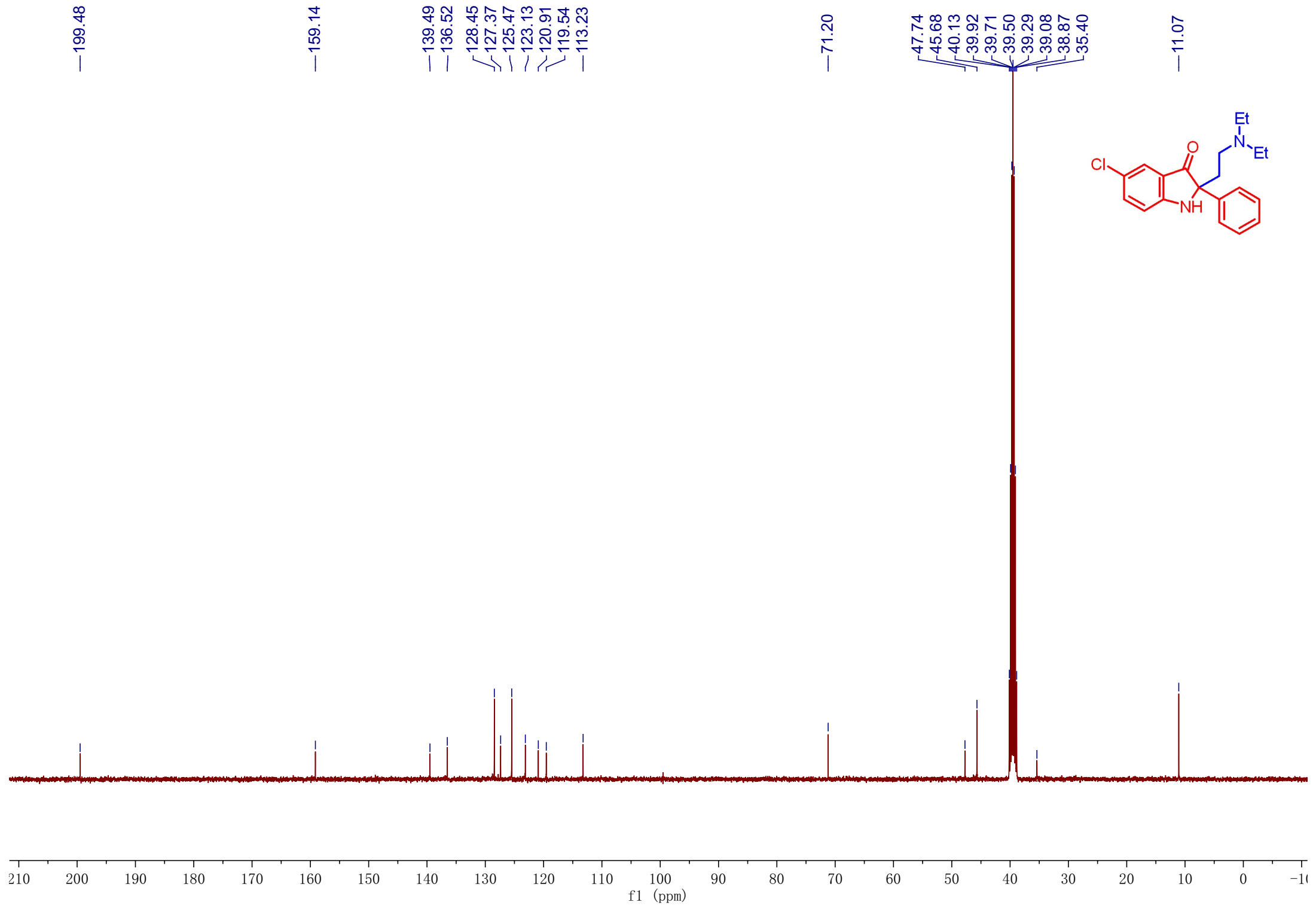


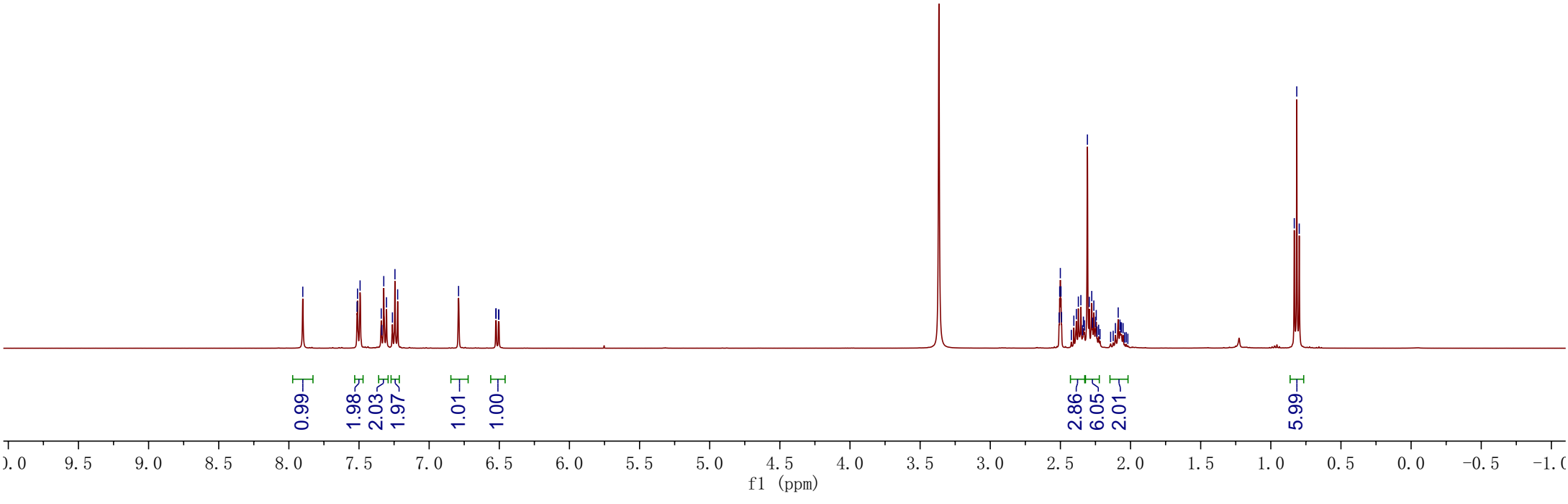
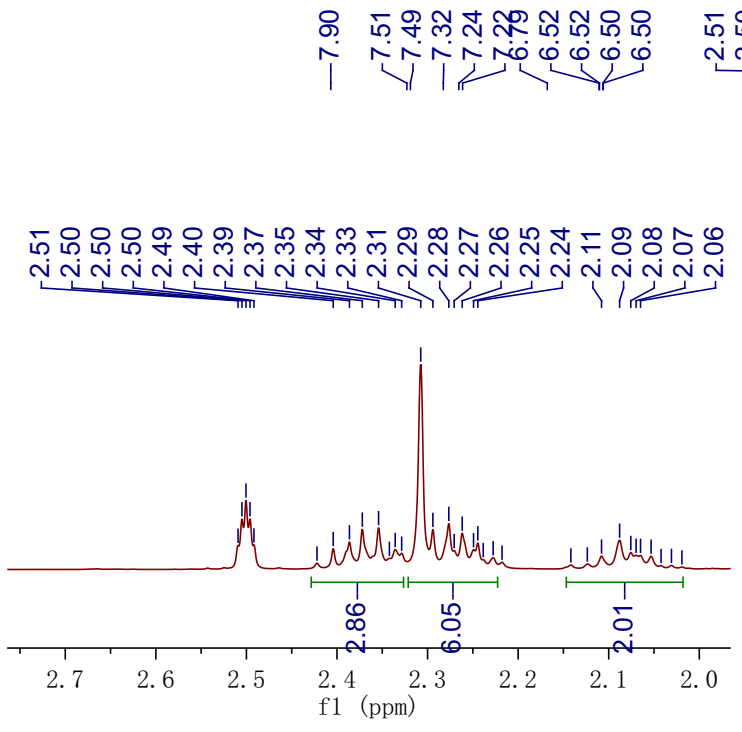
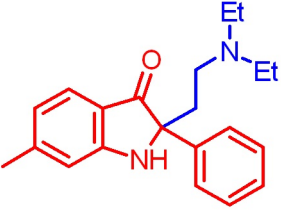
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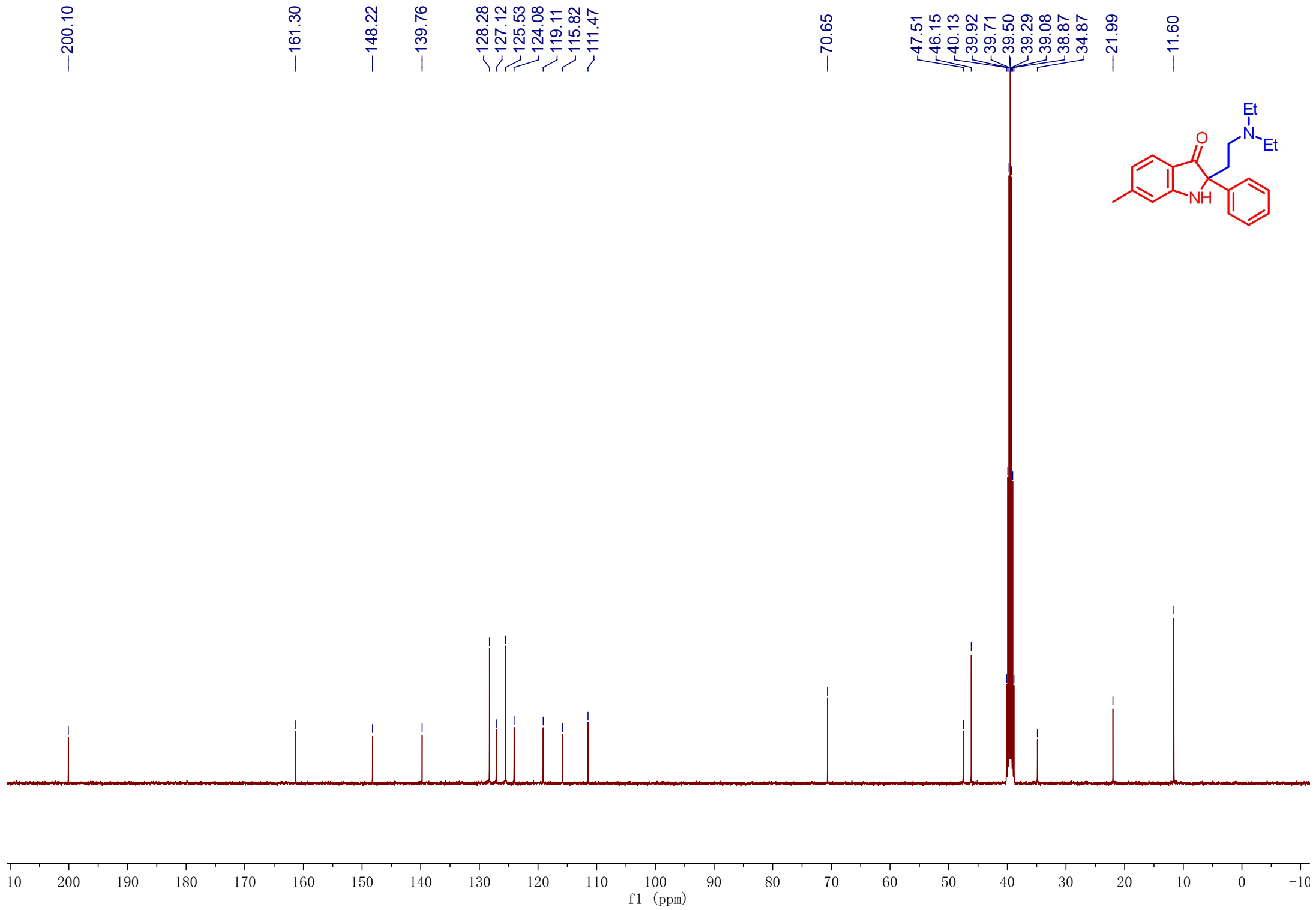
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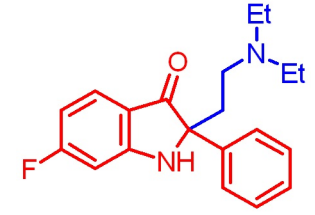
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2.02



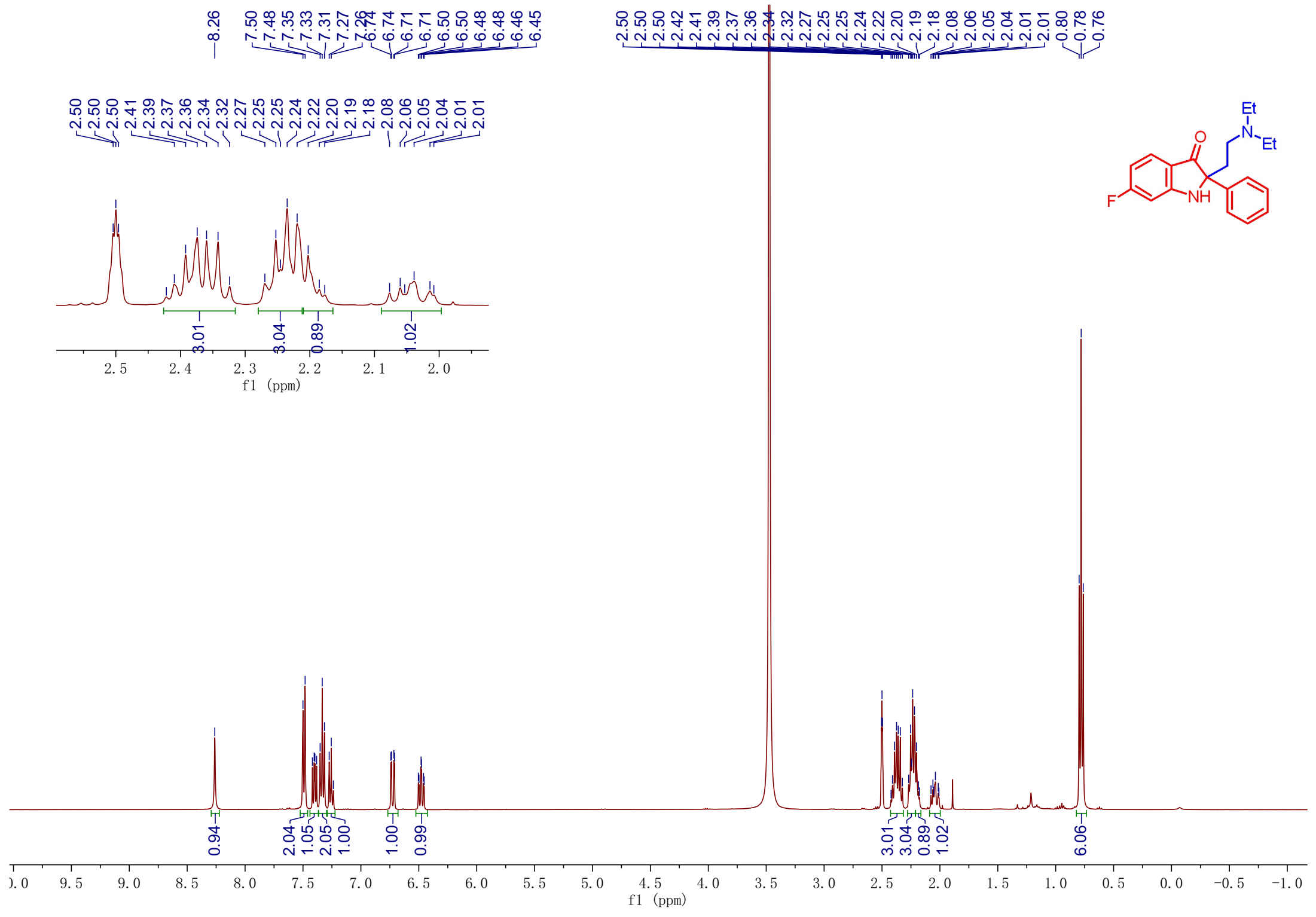
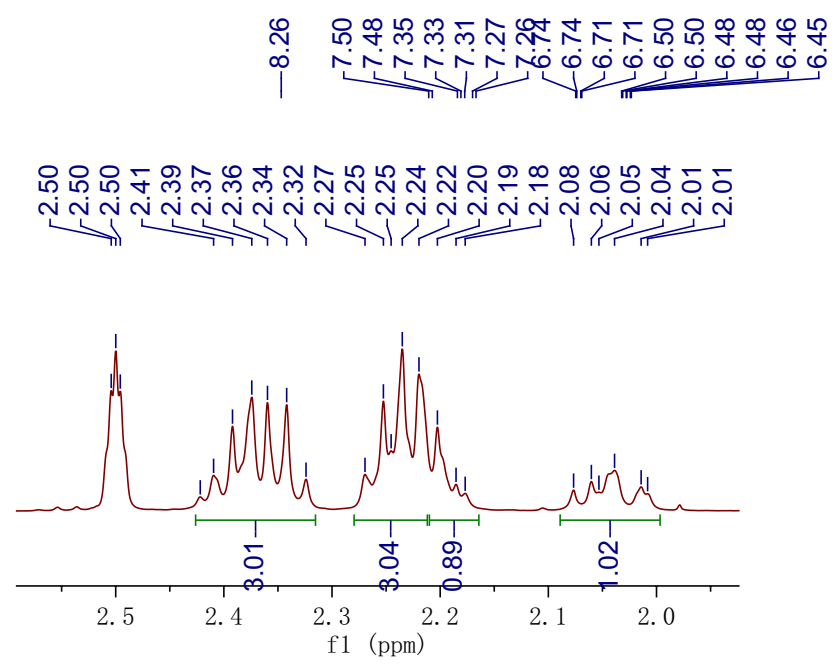


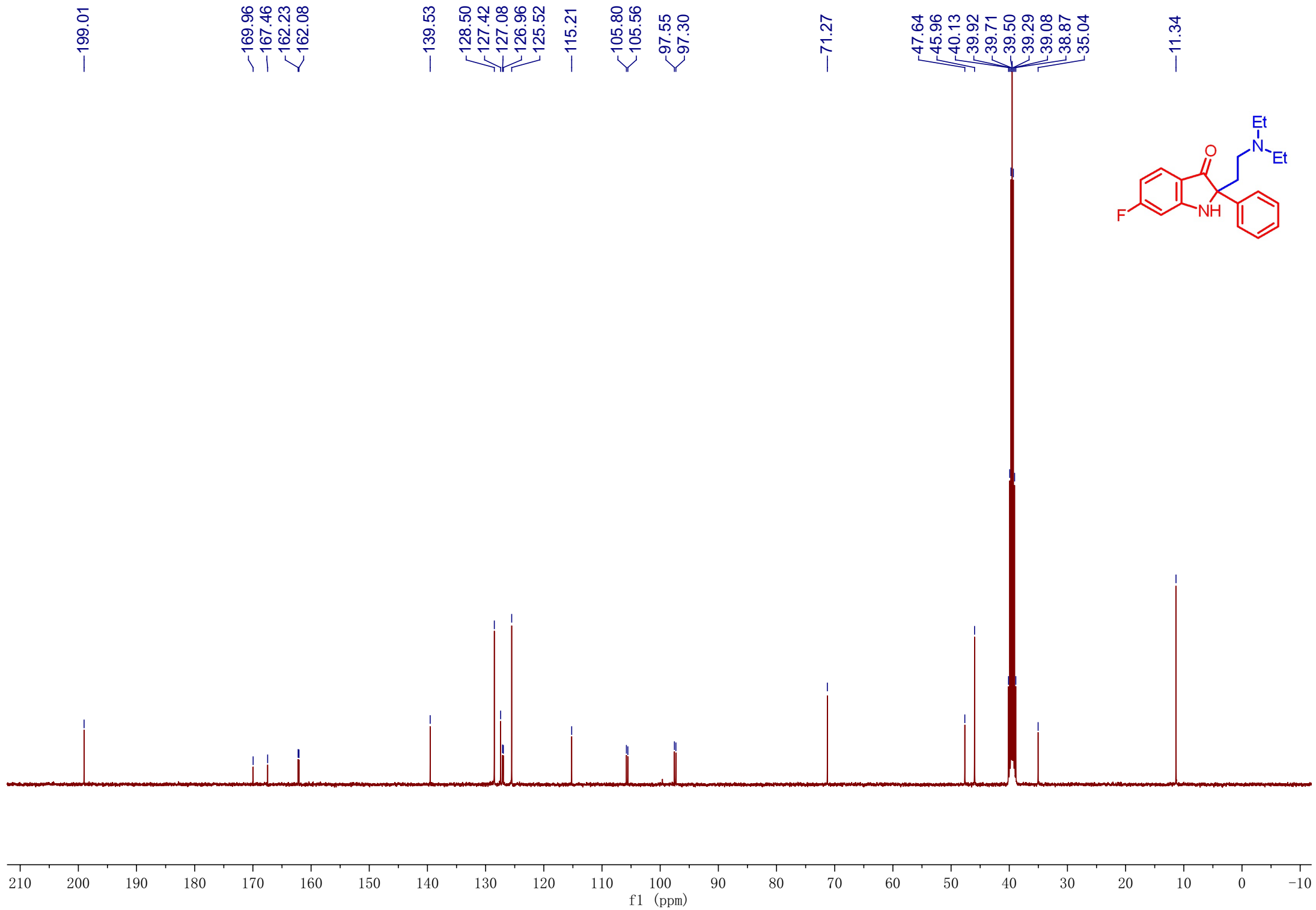


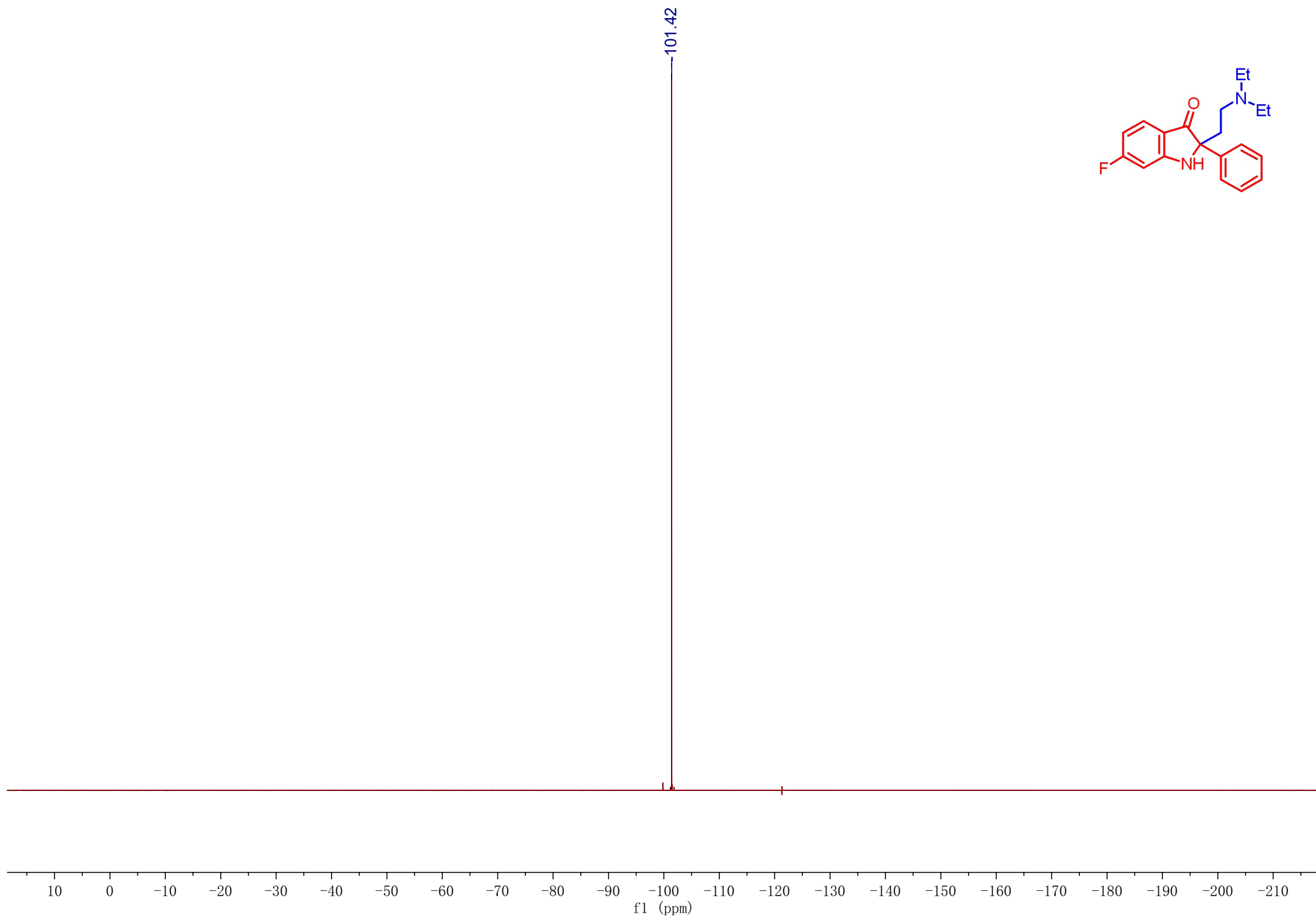
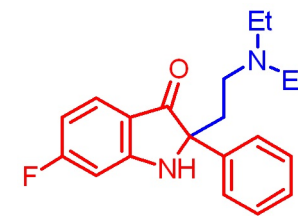


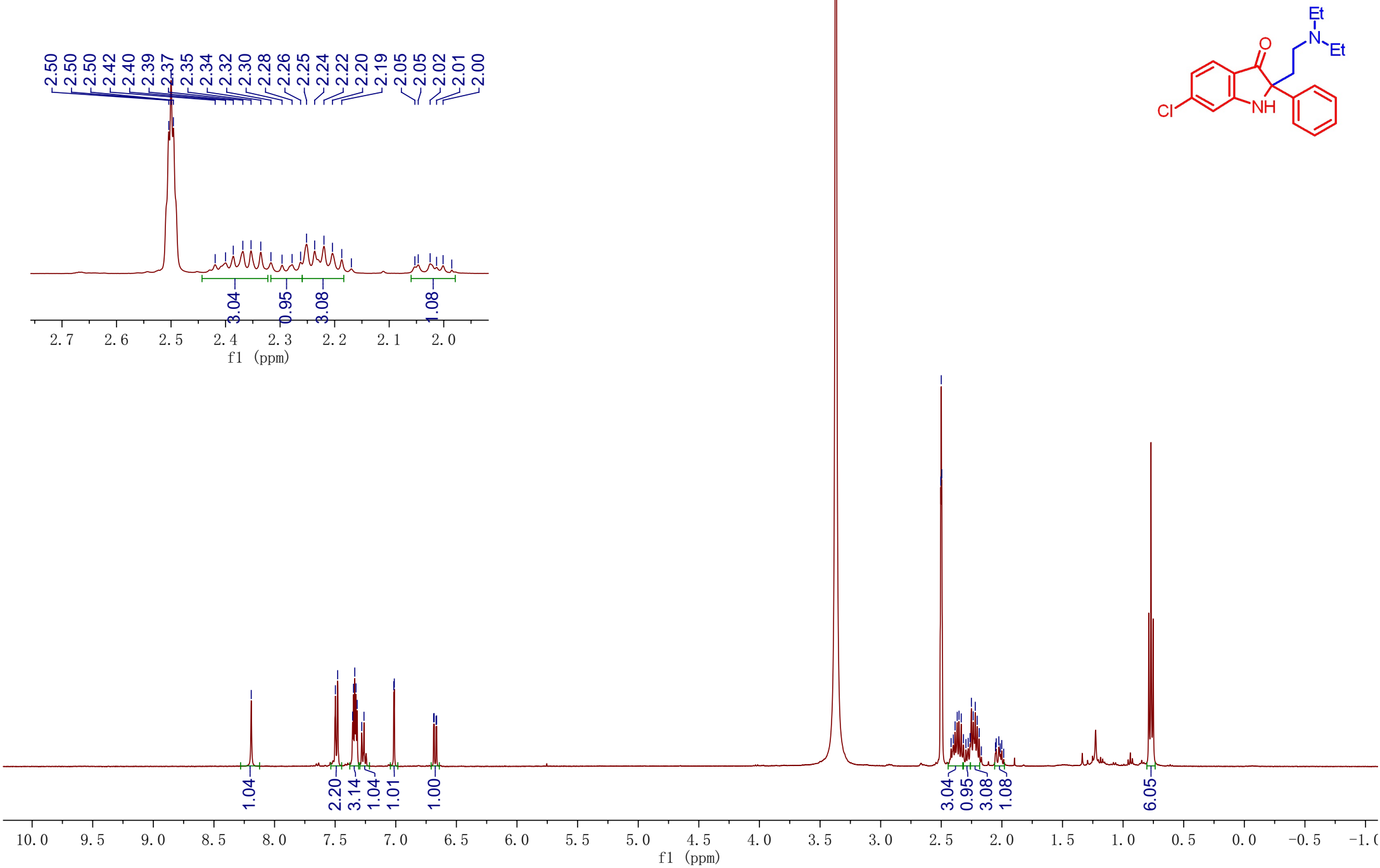


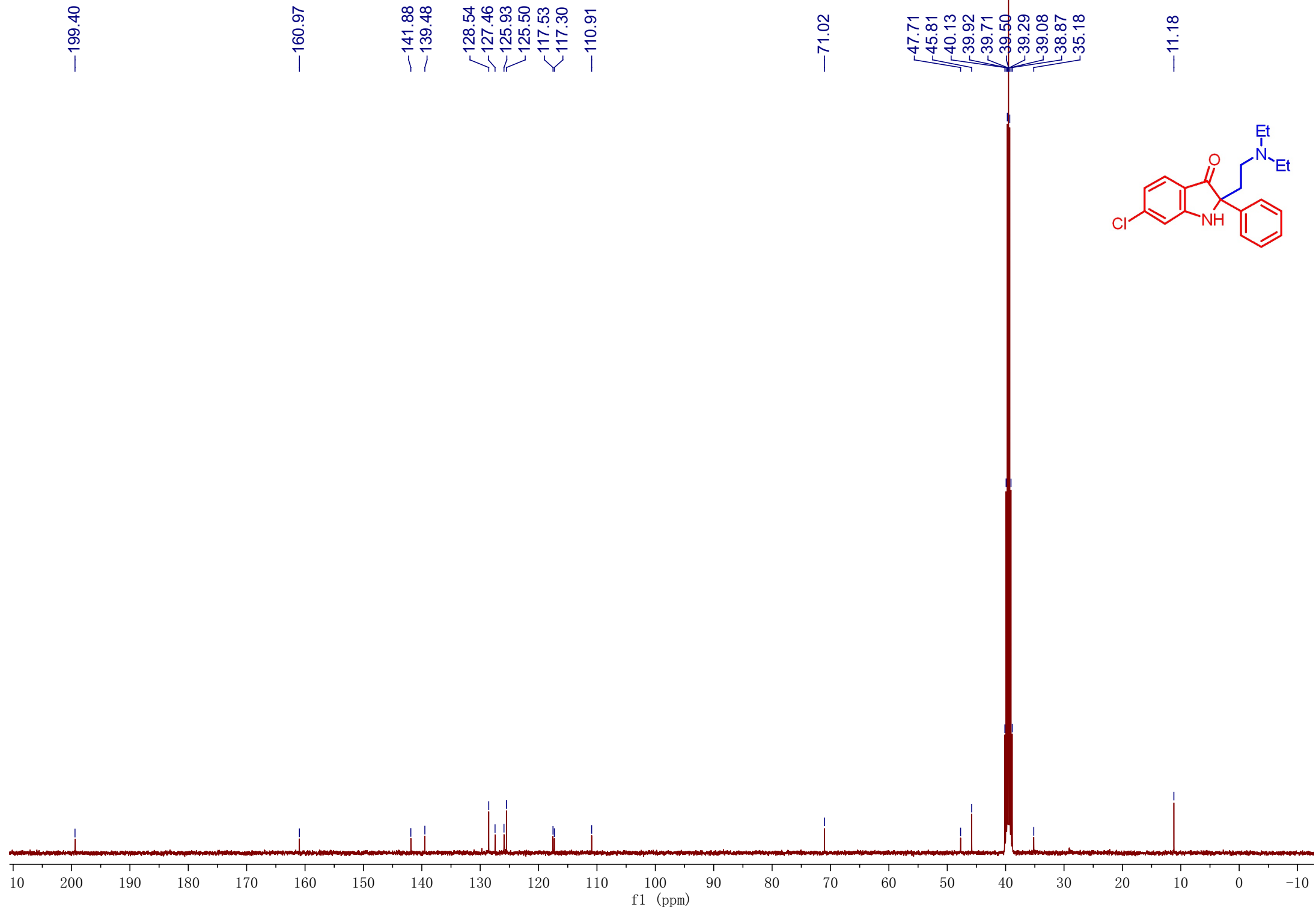
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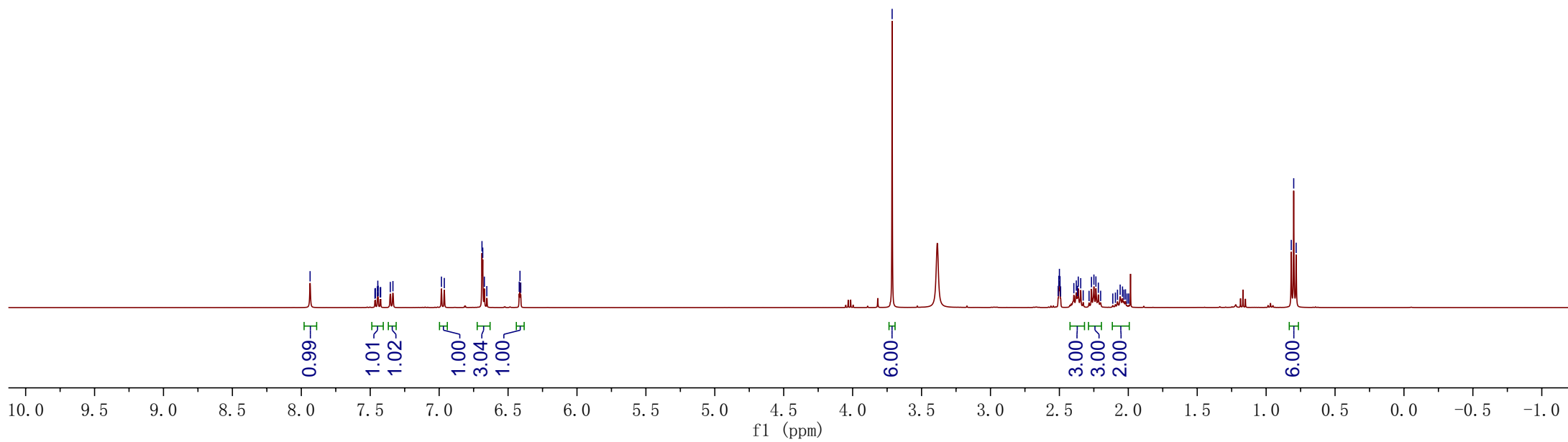
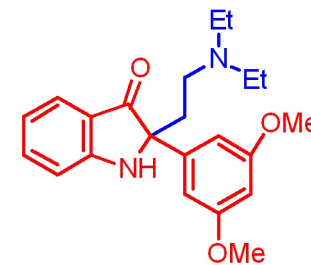
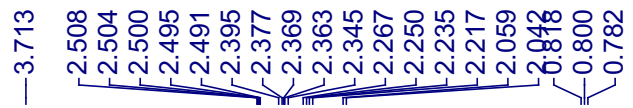
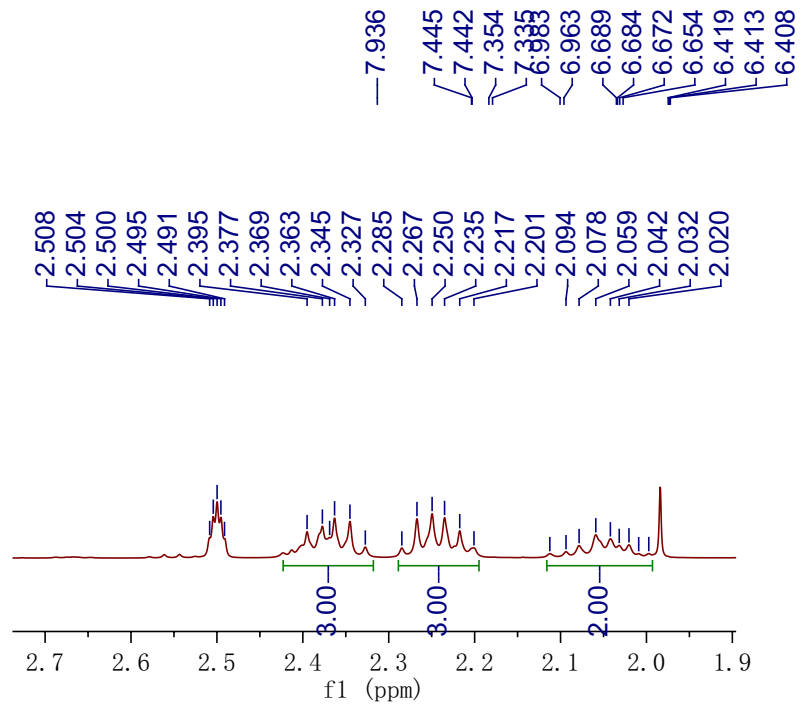


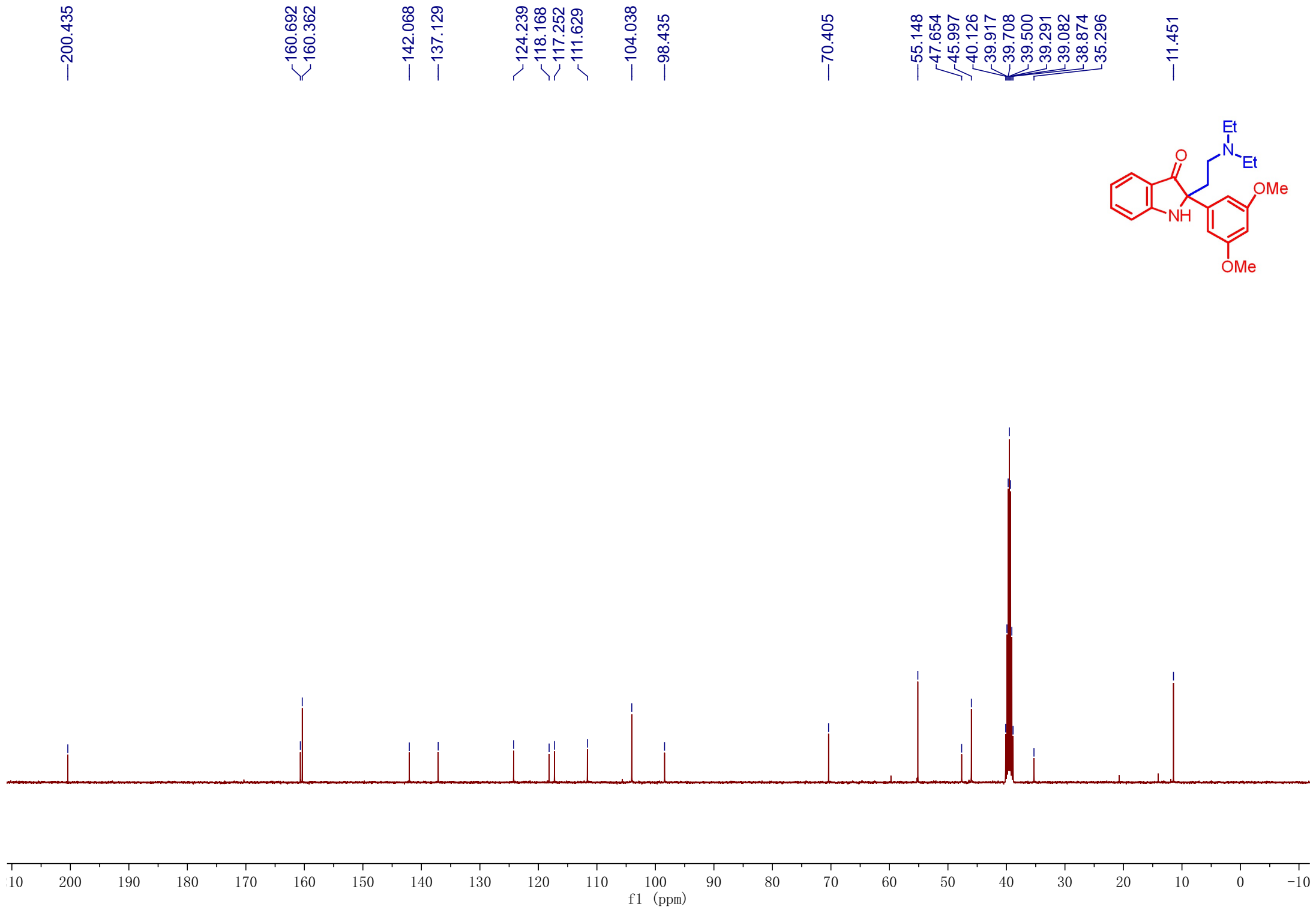


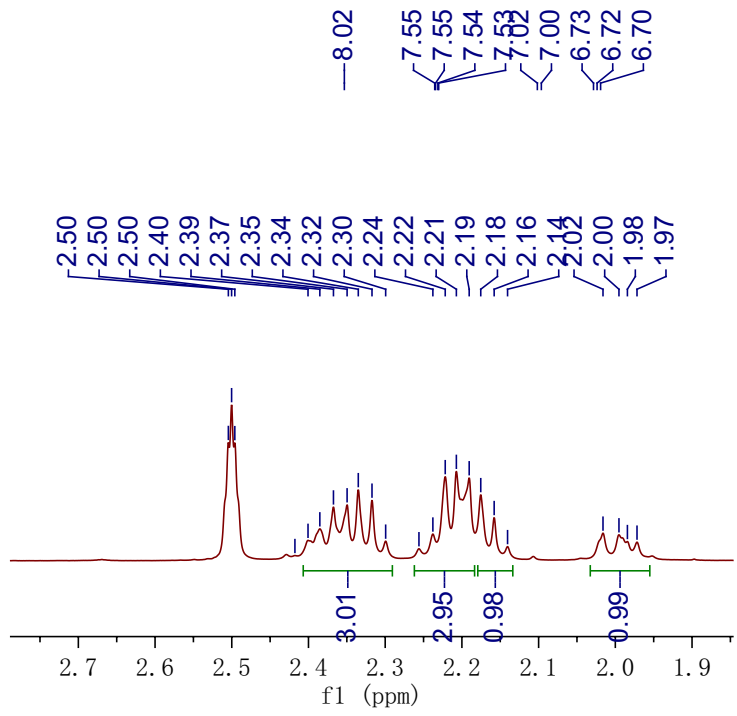






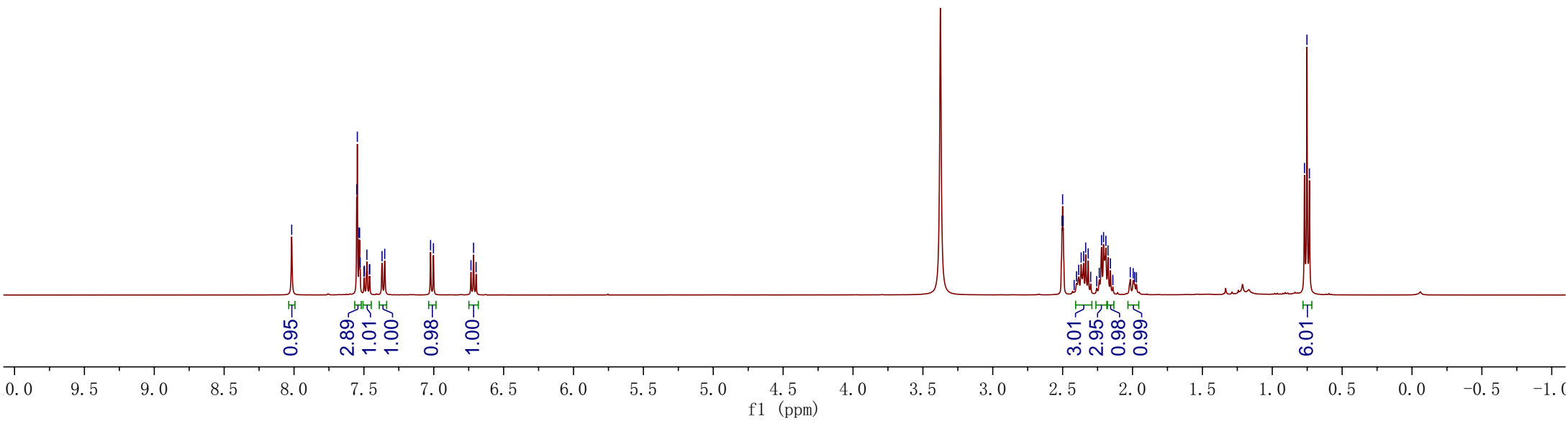
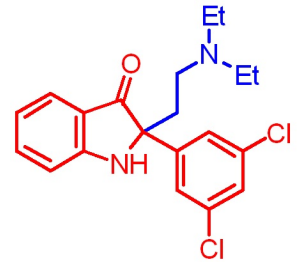


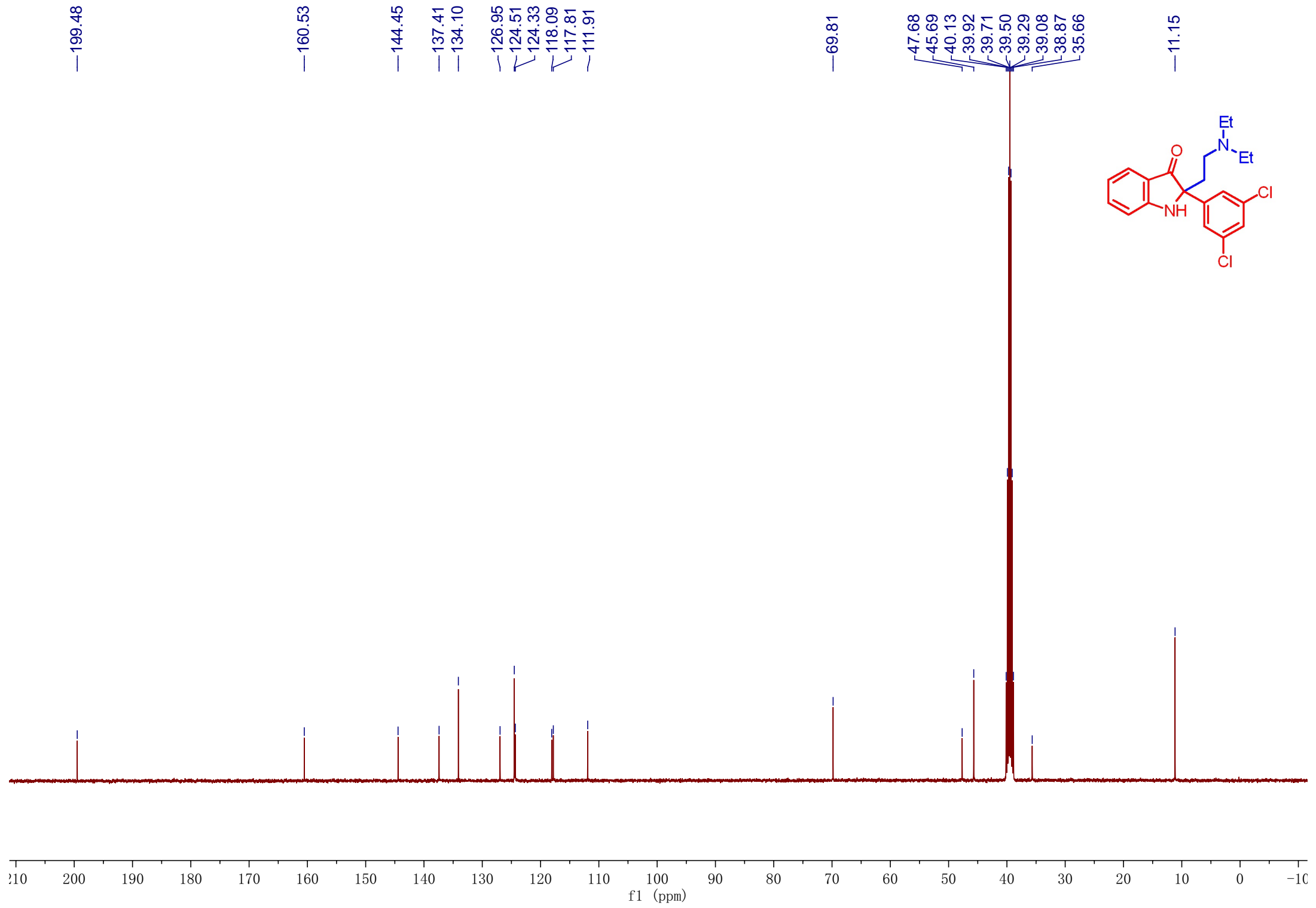




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6.70

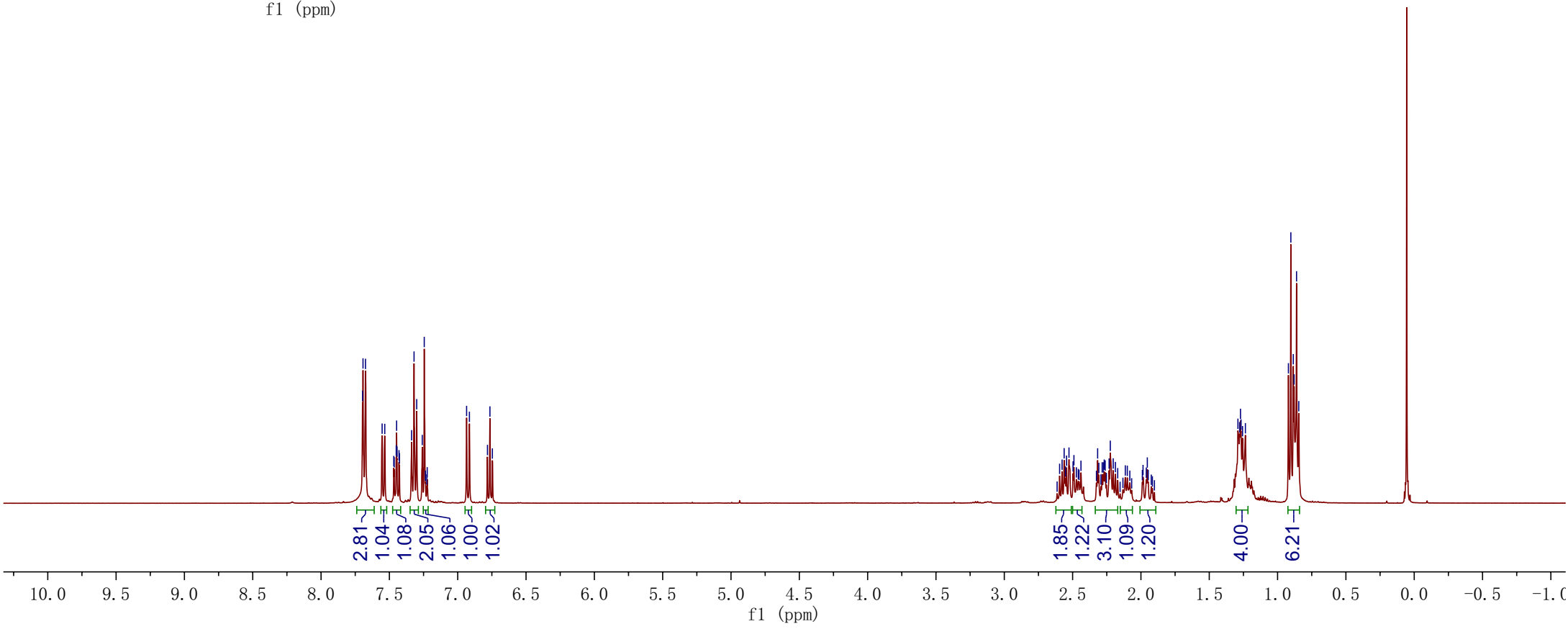
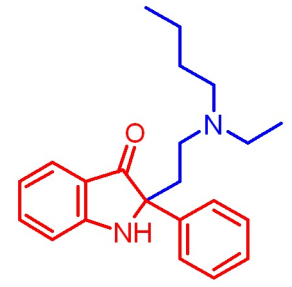
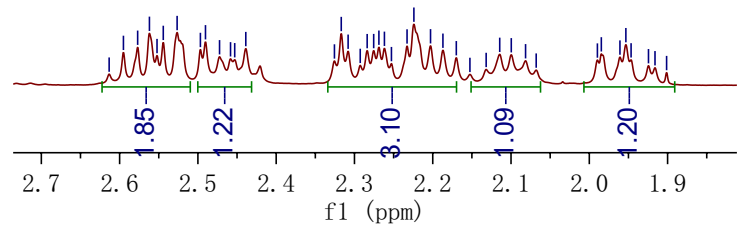
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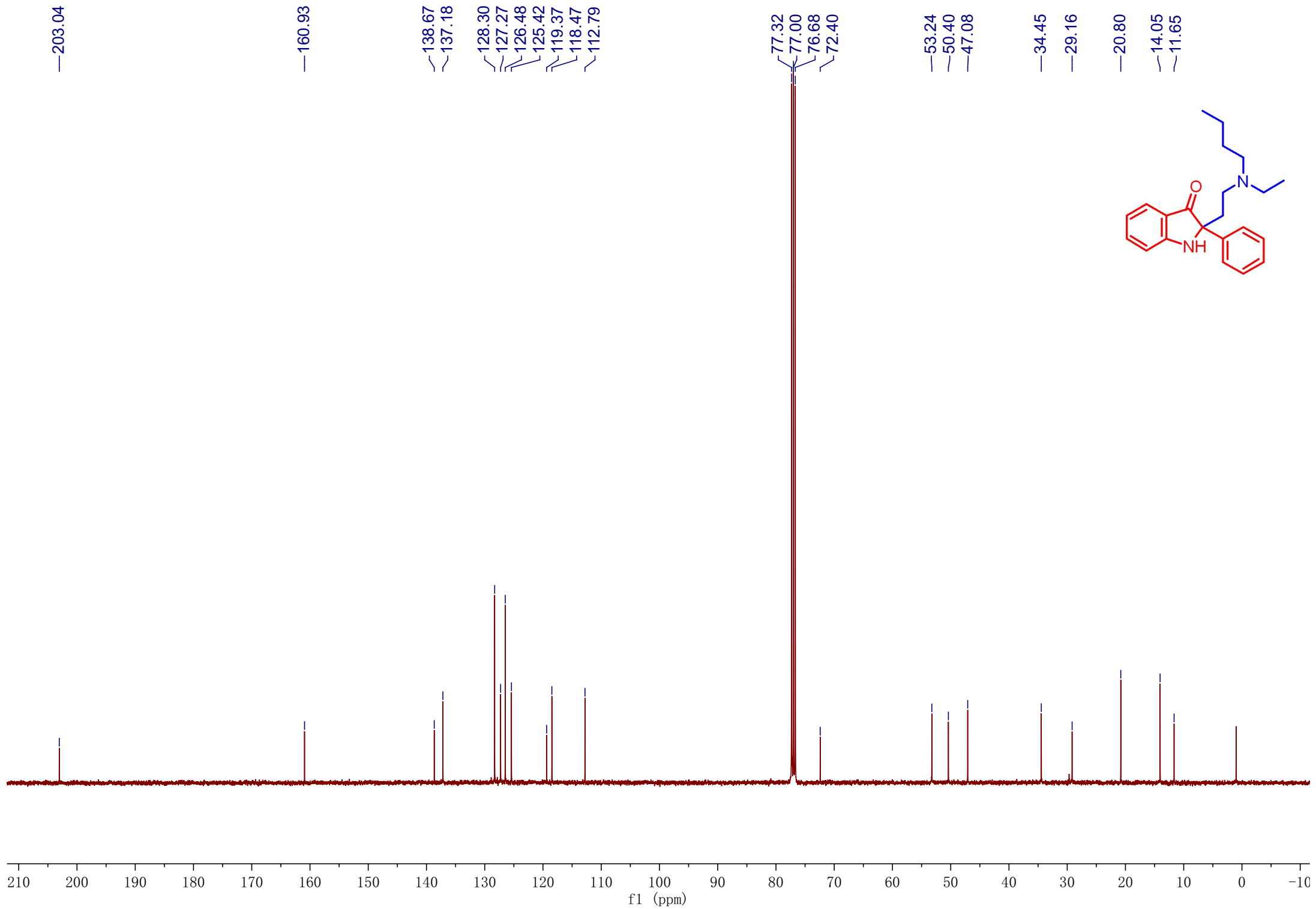


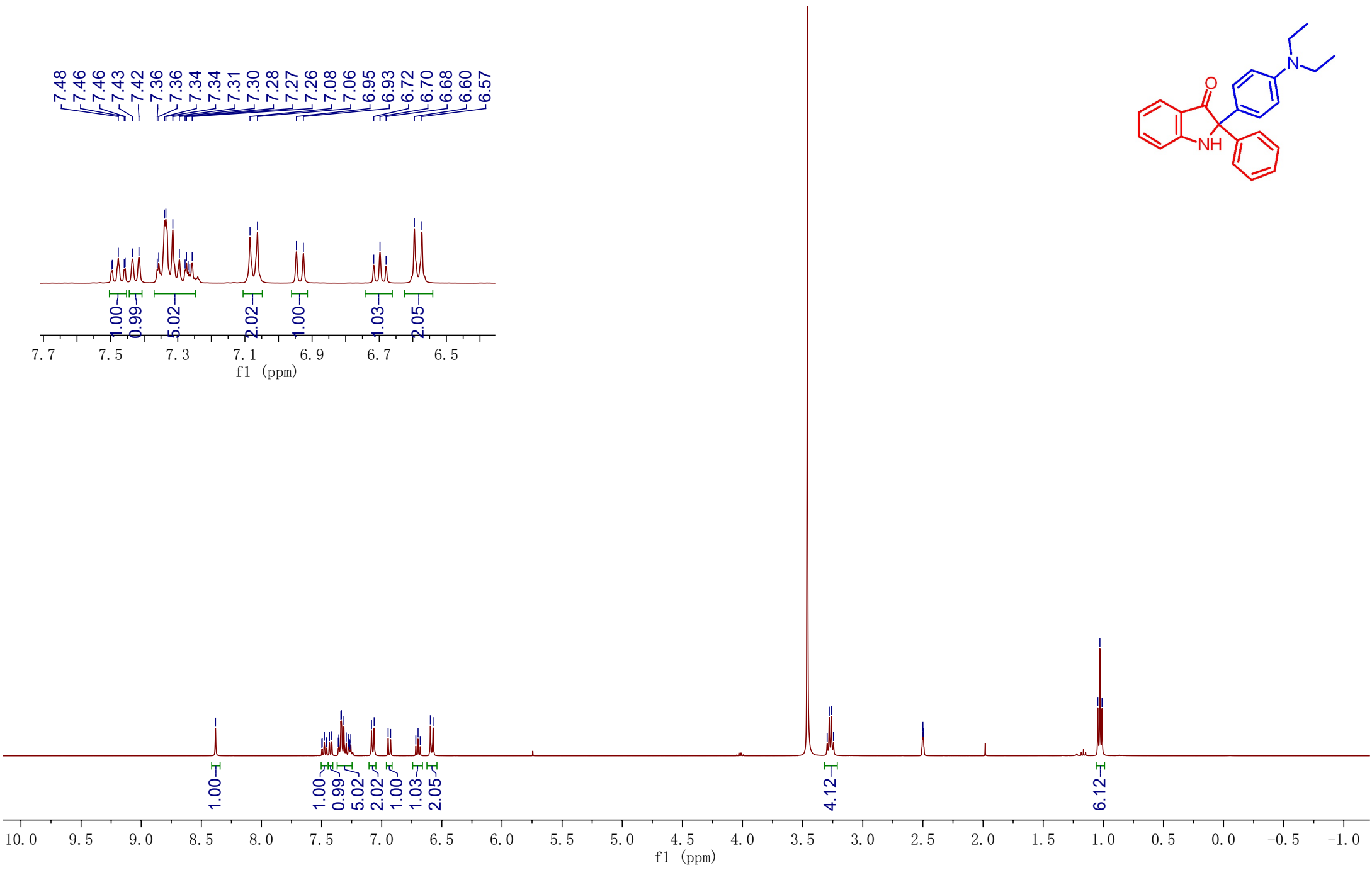


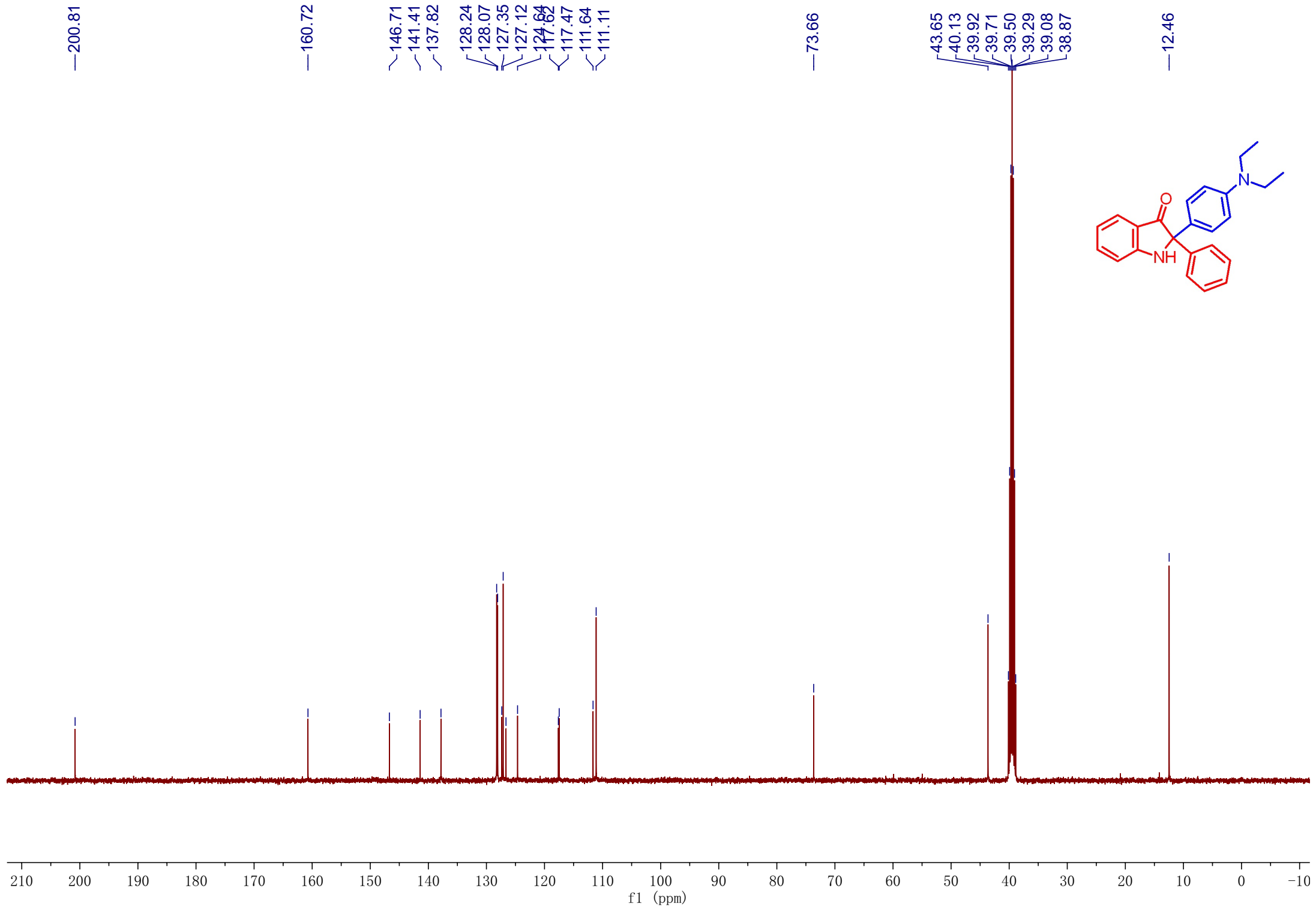
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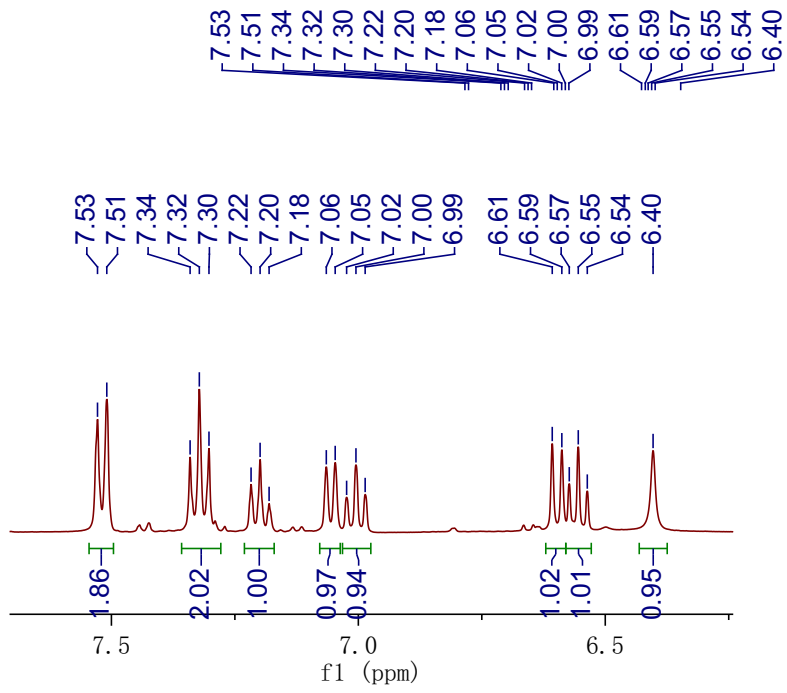
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—4.85

—3.35

