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Support Information

L-amino acid ester as biomimetic reducing agent for the reduction of unsaturated C=C bonds

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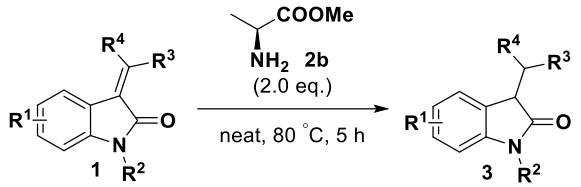
1. General experimental information

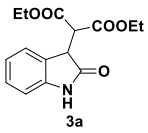
Unless otherwise noted, all the substrates and reagents were purchased from commercial suppliers and used without further purification, which were known compounds. ^{1}H NMR spectra were recorded at 500 MHz. The chemical shifts were recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. ^{13}C NMR data were collected at 125 MHz with complete proton decoupling. Chemical shifts were reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. High resolution mass spectroscopy (HRMS) was performed on Thermo Q Exactive Plus (FTMS ESI) mass spectrometer and acetonitrile was used to dissolve the sample. Column chromatography was carried out on silica gel (200-300 mesh). All the disubstituted methyleneindolinones were prepared according to the reported protocol.[1] All the substituted isoindigos were prepared according to the reported protocol.[2] All the tetrasubstituted olefins were prepared according to the reported protocol.[3]

2. Experimental procedures and characterization data

2.1. General procedure for the synthesis of 3-substituted 2-oxindoles 3

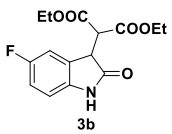


Disubstituted methyleneindolinones **1** (0.2 mmol, 1.0 equiv.) and L-alanine methyl ester **2b** (0.40 mmol, 2.0 equiv.) were heated at 80 ℃ for 5 h. After completion of the reaction, the reaction mixture was purified by flash column chromatography on silica gel (EtOAc/PE = 10%-20%) to yield 3-substituted 2-oxindoles **3**.



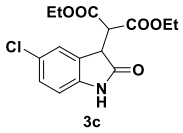
3a

3-Substituted 2-oxindole 3a[4]: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (55.5 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.17 (s, 1H), 7.37 (d, J = 7.5 Hz, 1H), 7.21 (t, J = 7.8 Hz, 1H), 6.99 (t, J = 7.3 Hz, 1H), 6.89 (d, J = 8.0 Hz, 1H), 4.25-4.33 (m, 2H), 4.23 (d, J = 4.0 Hz, 1H), 3.96-4.07 (m, 3H), 1.29 (t, J = 7.0 Hz, 3H), 1.01 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.0, 168.1, 167.0, 142.1, 128.6, 126.2, 125.2, 122.5, 109.9, 62.0, 61.8, 52.2, 45.2, 14.0, 13.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{18}NO_5^+$ 292.1179, found 292.1171.



3b

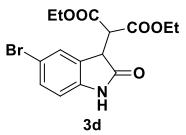
3-Substituted 2-oxindole 3b: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (59.3 mg, 96% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.03 (s, 1H), 7.19 (dd, J = 8.5, 1.5 Hz, 1H), 6.91-6.95 (m, 1H), 6.82 (dd, J = 8.5, 4.5 Hz, 1H), 4.27-4.35 (m, 2H), 4.22 (d, J = 4.0 Hz, 1H), 4.00-4.08 (m, 3H), 1.32 (t, J = 7.0 Hz, 3H), 1.04 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.7, 168.0, 166.7, 158.9 (d, $^1J_{C\text{-}F}$ = 238.8 Hz), 138.0, 127.8 (d, $^3J_{C\text{-}F}$ = 8.8 Hz), 115.0 (d, $^2J_{C\text{-}F}$ = 22.5 Hz), 113.5 (d, $^2J_{C\text{-}F}$ = 26.3 Hz), 110.3 (d, $^3J_{C\text{-}F}$ = 8.8 Hz), 62.1, 61.9, 52.1, 45.6, 14.0, 13.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}FNO_5^+$ 310.1085, found 310.1084.



3c

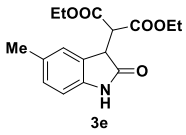
3-Substituted 2-oxindole 3c: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (61.9 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.06 (*br* s, 1H), 7.40 (s, 1H), 7.20 (dd, J = 8.0, 2.0 Hz, 1H), 6.82 (d, J = 8.0 Hz, 1H), 4.26-

4.35 (m, 2H), 4.21 (d, J = 3.5 Hz, 1H), 4.02-4.09 (m, 3H), 1.32 (t, J = 7.0 Hz, 3H), 1.05 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.4, 167.9, 166.7, 140.6, 128.6, 127.9(1), 127.8(6), 125.7, 110.8, 62.2, 62.0, 52.1, 45.3, 14.0, 13.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}ClNO_5^+$ 326.0790, found 326.0788.



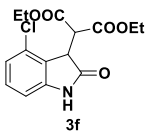
3d

3-Substituted 2-oxindole 3d: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (71.6 mg, 97% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.92 (*br* s, 1H), 7.53 (s, 1H), 7.34-7.37 (m, 1H), 6.77 (d, J = 8.5 Hz, 1H), 4.26-4.35 (m, 2H), 4.21 (d, J = 3.5 Hz, 1H), 4.02-4.09 (m, 3H), 1.32 (t, J = 7.0 Hz, 3H), 1.05 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.2, 167.9, 166.7, 141.1, 131.6, 128.5, 128.3, 115.1, 111.2, 62.2, 62.0, 52.1, 45.2, 14.0, 13.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}BrNO_5^+$ 370.0285, found 370.0283.



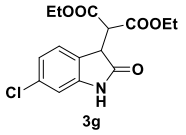
3e

3-Substituted 2-oxindole 3e: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (56.9 mg, 93% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.19 (s, 1H), 7.01 (d, J = 8.0 Hz, 1H), 6.77 (d, J = 8.0 Hz, 1H), 4.26-4.33 (m, 2H), 4.21 (d, J = 3.5 Hz, 1H), 3.99-4.07 (m, 3H), 2.29 (s, 3H), 1.30 (t, J = 7.0 Hz, 3H), 1.01 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.9, 168.2, 167.0, 139.6, 131.9, 128.9, 126.2, 125.9, 109.5, 61.9, 61.7, 52.2, 45.3, 21.2, 14.0, 13.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{16}H_{20}NO_5^+$ 306.1336, found 306.1333.



3f

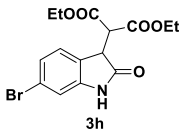
3-Substituted 2-oxindole 3f: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (59.9 mg, 92% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.60 (s, 1H), 7.17 (td, J = 8.0, 1.0 Hz, 1H), 6.95 (dd, J = 8.0, 1.0 Hz, 1H), 6.78 (d, J = 7.5 Hz, 1H), 4.72 (d, J = 4.5 Hz, 1H), 4.31-4.40 (m, 2H), 4.22 (d, J = 4.5 Hz, 1H), 4.10-4.17 (m, 1H), 3.98-4.04 (m, 1H), 1.35 (t, J = 7.0 Hz, 3H), 1.11 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.5, 167.6, 166.8, 143.9, 130.4, 130.1, 123.6, 122.8, 108.6, 62.2, 61.6, 50.3, 45.3, 14.0, 13.8; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}ClNO_5^+$ 326.0790, found 326.0787.



3g

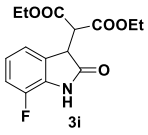
3-Substituted 2-oxindole 3g: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (63.8 mg, 98% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.94

(s, 1H), 7.32 (d, J = 8.0 Hz, 1H), 6.98 (dd, J = 8.0, 2.0 Hz, 1H), 6.90 (d, J = 2.0 Hz, 1H), 4.25-4.34 (m, 2H), 4.21 (d, J = 3.5 Hz, 1H), 4.01-4.09 (m, 3H), 1.31 (t, J = 7.0 Hz, 3H), 1.06 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.7, 168.0, 166.8, 143.1, 134.4, 126.2, 124.6, 122.5, 110.5, 62.1, 62.0, 52.1, 44.8, 14.0, 13.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}ClNO_5^+$ 326.0790, found 326.0789.



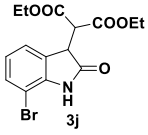
3h

3-Substituted 2-oxindole 3h: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (70.5 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.05 (s, 1H), 7.26 (d, J = 8.5 Hz, 1H), 7.13 (dd, J = 8.0, 2.0 Hz, 1H), 7.06 (d, J = 1.5 Hz, 1H), 4.25-4.34 (m, 2H), 4.21 (d, J = 3.5 Hz, 1H), 4.01-4.09 (m, 2H), 3.99 (d, J = 3.0 Hz, 1H), 1.31 (t, J = 7.0 Hz, 3H), 1.06 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.6, 168.0, 166.8, 143.3, 126.6, 125.4, 125.1, 122.2, 113.3, 62.1, 62.0, 52.0, 44.9, 14.0, 13.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}BrNO_5^+$ 370.0285, found 370.0283.



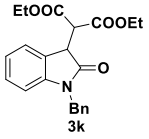
3i

3-Substituted 2-oxindole 3i: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (58.1 mg, 94% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.19 (d, J = 7.5 Hz, 1H), 7.00-7.03 (m, 1H), 6.94-6.98 (m, 1H), 4.25-4.34 (m, 3H), 4.10 (d, J = 3.5 Hz, 1H), 3.99-4.09 (m, 2H), 1.30 (t, J = 7.0 Hz, 3H), 1.03 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.9, 167.9, 166.7, 147.0 (d, $^1J_{C\text{-}F}$ = 242.5 Hz), 129.3 (d, $^2J_{C\text{-}F}$ = 12.5 Hz), 128.9, 123.1 (d, $^3J_{C\text{-}F}$ = 6.3 Hz), 120.9 (d, $^3J_{C\text{-}F}$ = 2.5 Hz), 115.7 (d, $^2J_{C\text{-}F}$ = 16.3 Hz), 62.1, 61.9, 52.1, 45.4, 14.0, 13.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}FNO_5^+$ 310.1085, found 310.1083.

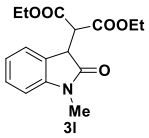


3j

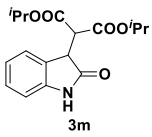
3-Substituted 2-oxindole 3j: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (69.6 mg, 94% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.34-7.37 (m, 2H), 6.90 (t, J = 8.0 Hz, 1H), 4.25-4.34 (m, 2H), 4.24 (d, J = 3.0 Hz, 1H), 4.14 (d, J = 3.5 Hz, 1H), 4.03-4.09 (m, 1H), 3.95-4.02 (m, 1H), 1.30 (t, J = 7.0 Hz, 3H), 1.00 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.9, 167.9, 166.6, 141.3, 131.3, 127.5, 124.2, 123.8, 102.7, 62.1, 61.9, 52.2, 46.3, 14.0, 13.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{15}H_{17}BrNO_5^+$ 370.0285, found 370.0282.



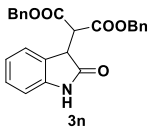
3-Substituted 2-oxindole 3k: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (72.3 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, J = 7.5 Hz, 1H), 7.36 (d, J = 7.5 Hz, 2H), 7.30-7.33 (m, 2H), 7.24-7.27 (m, 1H), 7.16 (t, J = 7.5 Hz, 1H), 6.98 (t, J = 7.5 Hz, 1H), 6.72 (d, J = 8.0 Hz, 1H), 4.93 (q, J = 15.5 Hz, 2H), 4.23-4.34 (m, 3H), 4.13 (d, J = 3.5 Hz, 1H), 3.98-4.05 (m, 1H), 3.88-3.94 (m, 1H), 1.28 (t, J = 7.0 Hz, 3H), 0.87 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.4, 168.2, 167.0, 143.8, 135.8, 128.7, 128.5, 127.7, 127.6, 125.7, 125.0, 122.6, 109.0, 62.0, 61.7, 52.3, 44.7, 44.1, 14.1, 13.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{24}NO_5^+$ 382.1649, found 382.1643.



3-Substituted 2-oxindole 3l: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (59.2 mg, 97% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, J = 7.5 Hz, 1H), 7.22 (t, J = 7.8 Hz, 1H), 6.96 (t, J = 7.5 Hz, 1H), 6.75 (d, J = 8.0 Hz, 1H), 4.16-4.25 (m, 2H), 4.14 (d, J = 4.0 Hz, 1H), 3.88-3.96 (m, 3H), 3.16 (s, 3H), 1.22 (t, J = 7.0 Hz, 3H), 0.92 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.3, 167.0, 165.9, 143.7, 127.6, 124.6, 123.9, 121.5, 106.9, 60.9, 60.6, 51.3, 43.6, 25.4, 13.0, 12.7; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{16}H_{20}NO_5^+$ 306.1336, found 306.1331.

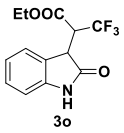


3-Substituted 2-oxindole 3m: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (59.9 mg, 94% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.82 (s, 1H), 7.43 (d, J = 7.5 Hz, 1H), 7.21 (t, J = 7.5 Hz, 1H), 6.99 (td, J = 7.5, 0.5 Hz, 1H), 6.87 (d, J = 7.5 Hz, 1H), 5.12-5.17 (m, 1H), 4.83-4.88 (m, 1H), 4.19 (d, J = 3.5 Hz, 1H), 4.04 (d, J = 3.0 Hz, 1H), 1.28 (t, J = 6.3 Hz, 6H), 1.06 (d, J = 6.5 Hz, 3H), 0.92 (d, J = 6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.9, 167.7, 166.5, 142.1, 128.6, 126.3, 125.5, 122.5, 109.7, 69.7, 69.6, 52.5, 45.2, 21.7, 21.5, 21.4, 21.0; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{17}H_{22}NO_5^+$ 320.1492, found 320.1490.



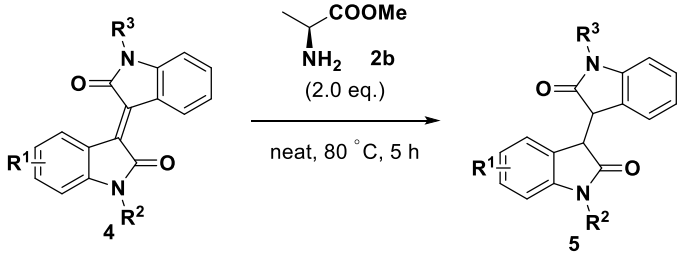
3-Substituted 2-oxindole 3n: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (78.9 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (s, 1H), 7.28-7.32 (m, 5H), 7.23-7.26 (m, 4H), 7.16 (t, J = 7.5 Hz, 1H), 7.10-7.12 (m, 2H), 6.92 (td, J = 7.5, 1.0 Hz, 1H), 6.73 (d, J = 7.5 Hz, 1H), 5.19-5.25 (m, 2H), 4.95-5.01 (m, 2H), 4.32 (d, J = 4.0 Hz, 1H), 4.07 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.2,

167.7, 166.7, 141.8, 135.0, 134.8, 128.7, 128.6, 128.4(9), 128.4(7), 128.4, 128.3, 125.9, 125.1, 122.5, 109.9, 67.8, 67.6, 52.2, 45.1; HRMS (FTMS-ESI) m/z: $[M+Na]^+$ calcd for $C_{25}H_{21}NNaO_5^+$ 438.1312, found 438.1310.

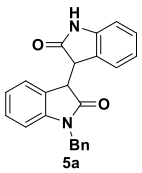


3-Substituted 2-oxindole 3o: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 10%-20% (v/v); Colorless oil (52.7 mg, 92% yield); ^{1}H NMR (500 MHz, $CDCl_3$, *mixture of two diastereomers*) δ 9.24 (s, 2.3H), 9.13 (s, 1.0H), 7.34-7.38 (m, 3.4H), 7.27 (t, J = 8.0 Hz, 3.5H), 7.02-7.06 (m, 3.4H), 6.94 (d, J = 8.0 Hz, 3.4H), 4.35-4.42 (m, 1.9H), 4.09-4.15 (m, 4.4H), 3.90-3.97 (m, 2.5H), 3.81-3.88 (m, 4.8H), 1.37 (t, J = 7.0 Hz, 3.0H), 0.83 (t, J = 7.0 Hz, 7.3H); ^{13}C NMR (125 MHz, $CDCl_3$, *major diastereomer*) δ 177.3, 164.1 (d, $^3J_{C\text{-}F}$ = 1.3 Hz), 142.1, 129.2, 124.5 (q, $^1J_{C\text{-}F}$ = 276.3 Hz), 125.5, 124.5, 122.9, 110.1, 61.8, 49.7 (q, $^2J_{C\text{-}F}$ = 28.8 Hz), 43.7 (d, $^3J_{C\text{-}F}$ = 1.3 Hz), 13.3; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{13}H_{13}F_3NO_3^+$ 288.0842, found 288.0846.

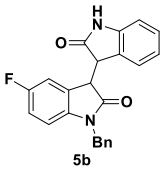
2.2. General procedure for the synthesis of dihydroisoindigos 5



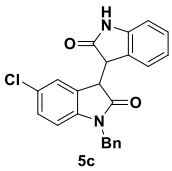
Isoindigos **4** (0.2 mmol, 1.0 equiv.) and L-alanine methyl ester **2b** (0.40 mmol, 2.0 equiv.) were heated at 80 ℃ for 5 h. After completion of the reaction, the precipitated solid was filtered, washed with dichloromethane and *n*-hexane, and then dried *in vacuo* to yield dihydroisoindigos **5**.



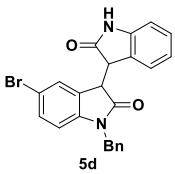
Dihydroisoindigo 5a: Obtained through direct single-run filtration; White solid (65.9 mg, 93% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.41 (s, 1H), 7.19-7.24 (m, 4H), 7.16-7.19 (m, 1H), 7.08-7.10 (m, 2H), 6.93 (d, J = 4.5 Hz, 2H), 6.81-6.84 (m, 2H), 6.74 (d, J = 7.5 Hz, 1H), 6.68 (d, J = 7.0 Hz, 1H), 4.93 (d, J = 16.0 Hz, 1H), 4.67 (d, J = 16.0 Hz, 1H), 4.39 (d, J = 3.5 Hz, 1H), 4.31 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.6, 175.0, 144.1, 143.9, 136.5, 128.9, 128.8, 127.6, 127.4, 127.3, 126.9, 124.1, 123.9, 122.5, 121.8, 110.0, 109.5, 46.2, 45.9, 43.1; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{19}N_2O_2^+$ 355.1441, found 355.1442.



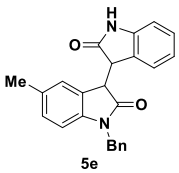
Dihydroisoindigo 5b: Obtained through direct single-run filtration; White solid (68.4 mg, 92% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.47 (s, 1H), 7.24 (t, J = 7.8 Hz, 1H), 7.20-7.21 (m, 3H), 7.04-7.07 (m, 3H), 6.82-6.88 (m, 3H), 6.74 (dd, J = 8.8, 4.3 Hz, 1H), 6.63 (d, J = 7.0 Hz, 1H), 4.92 (d, J = 16.0 Hz, 1H), 4.65 (d, J = 16.0 Hz, 1H), 4.44 (d, J = 3.0 Hz, 1H), 4.35 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.5, 174.7, 158.6 (d, $^{1}J_{C\text{-}F}$ = 236.3 Hz), 143.9, 140.4, 136.2, 129.0, 128.9, 128.8, 127.6, 127.4, 126.9, 124.0, 121.8, 115.0 (d, $^{2}J_{C\text{-}F}$ = 22.5 Hz), 112.0 (d, $^{2}J_{C\text{-}F}$ = 25.0 Hz), 110.3 (d, $^{3}J_{C\text{-}F}$ = 8.8 Hz), 110.0, 46.3, 45.9, 43.1; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}FN_2O_2^+$ 373.1347, found 373.1340.



Dihydroisoindigo 5c: Obtained through direct single-run filtration; White solid (73.3 mg, 94% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.47 (s, 1H), 7.23-7.27 (m, 2H), 7.19(8)-7.20(4) (m, 3H), 7.04(6)-7.05(3) (m, 2H), 6.99 (*br* s, 1H), 6.85 (t, J = 8.8 Hz, 2H), 6.75 (d, J = 8.0 Hz, 1H), 6.68 (d, J = 7.0 Hz, 1H), 4.93 (d, J = 16.0 Hz, 1H), 4.65 (d, J = 16.0 Hz, 1H), 4.45 (d, J = 3.0 Hz, 1H), 4.35 (d, J = 3.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.5, 174.6, 143.8, 143.0, 136.0, 129.1(0), 129.0(5), 128.9, 128.6, 127.7, 127.4, 126.9, 126.6, 124.2, 124.0, 121.9, 110.9, 110.1, 46.0, 45.9, 43.1; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}ClN_2O_2^+$ 389.1051, found 389.1047.

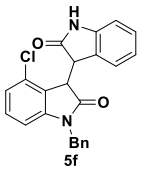


Dihydroisoindigo 5d: Obtained through direct single-run filtration; White solid (81.2 mg, 94% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.47 (s, 1H), 7.39 (dd, J = 8.0, 1.0 Hz, 1H), 7.25 (t, J = 8.0 Hz, 1H), 7.20-7.21 (m, 3H), 7.11 (*br* s, 1H), 7.05(5)-7.06(1) (m, 2H), 6.84-6.86 (m, 2H), 6.68-6.72 (m, 2H), 4.93 (d, J = 16.0 Hz, 1H), 4.65 (d, J = 16.0 Hz, 1H), 4.47 (d, J = 3.0 Hz, 1H), 4.36 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.4, 174.5, 143.8, 143.5, 136.1, 131.4, 129.5, 129.0, 128.9, 127.7, 127.4, 127.0, 126.9, 124.1, 121.9, 114.4, 111.4, 110.0, 45.9, 43.1; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}BrN_2O_2^+$ 433.0546, found 433.0544.



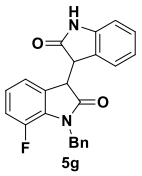
Dihydroisoindigo 5e: Obtained through direct single-run filtration; White solid (67.1 mg, 91% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.42 (s, 1H), 7.19-7.24 (m, 4H), 7.04-7.08 (m, 2H), 6.99 (d, J = 8.0 Hz, 1H), 6.79-6.84 (m, 3H), 6.62 (d, J = 8.0 Hz, 2H), 4.90 (d, J = 16.0 Hz, 1H), 4.62 (d, J = 16.0 Hz, 1H), 4.34 (d, J = 3.5 Hz, 1H), 4.27 (d, J = 3.5 Hz, 1H),

2.16 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.7, 174.8, 143.9, 141.8, 136.5, 131.4, 128.9, 128.8, 127.5, 127.4, 127.3, 127.0, 124.7, 124.0, 121.7, 109.9, 109.3, 46.1, 46.0, 43.0, 21.2; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{21}N_2O_2^+$ 369.1598, found 369.1596.



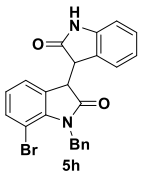
5f

Dihydroisoindigo 5f: Obtained through direct single-run filtration; White solid (69.8 mg, 90% yield); ^{1}H NMR (500 MHz, Tetrahydrofuran-d_8) δ 9.45 (*br* s, 1H), 7.09 (t, J = 8.0 Hz, 1H), 6.94-7.03 (m, 5H), 6.69-6.71 (m, 3H), 6.52 (d, J = 7.5 Hz, 1H), 6.45 (t, J = 7.5 Hz, 1H), 5.96 (d, J = 3.0 Hz, 1H), 4.80 (d, J = 15.5 Hz, 1H), 4.34 (dd, J = 17.5, 2.5 Hz, 2H), 4.29 (d, J = 15.5 Hz, 1H); ^{13}C NMR (125 MHz, Tetrahydrofuran-d_8) δ 175.9, 173.1, 146.6, 144.8, 136.3, 130.8, 130.4, 128.8, 128.7, 127.5, 127.4, 124.9, 123.9, 122.9, 121.3, 109.7, 108.0, 47.0, 43.9, 43.4; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}ClN_2O_2^+$ 389.1051, found 389.1048.



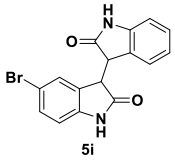
5g

Dihydroisoindigo 5g: Obtained through direct single-run filtration; White solid (68.5 mg, 92% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.46 (s, 1H), 7.25 (t, J = 7.5 Hz, 1H), 7.19-7.20 (m, 3H), 7.06-7.11 (m, 1H), 6.95-7.01 (m, 3H), 6.84-6.88 (m, 2H), 6.81 (d, J = 7.0 Hz, 1H), 6.72 (d, J = 7.0 Hz, 1H), 5.00 (d, J = 16.0 Hz, 1H), 4.76 (d, J = 16.0 Hz, 1H), 4.51 (d, J = 3.5 Hz, 1H), 4.35 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.4, 174.8, 147.0 (d, $^1J_{C\text{-}F}$ = 240.0 Hz), 143.9, 137.4, 130.6 (d, $^2J_{C\text{-}F}$ = 8.8 Hz), 130.2 (d, $^3J_{C\text{-}F}$ = 2.5 Hz), 129.0, 128.8, 127.4, 127.0, 126.8, 124.1, 123.7 (d, $^3J_{C\text{-}F}$ = 6.3 Hz), 121.9, 120.3, 116.8 (d, $^2J_{C\text{-}F}$ = 18.8 Hz), 110.1, 46.1(9), 46.1(5), 44.8; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}FN_2O_2^+$ 373.1347, found 373.1344.

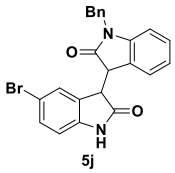


5h

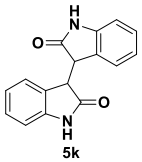
Dihydroisoindigo 5h: Obtained through direct single-run filtration; White solid (80.8 mg, 93% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.47 (s, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.25 (t, J = 8.0 Hz, 1H), 7.16-7.18 (m, 3H), 7.06 (d, J = 6.5 Hz, 1H), 6.89-6.95 (m, 4H), 6.84 (d, J = 8.0 Hz, 1H), 6.72 (d, J = 7.0 Hz, 1H), 5.16 (s, 2H), 4.52 (d, J = 3.5 Hz, 1H), 4.37 (d, J = 4.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.4, 175.8, 143.9, 141.4, 137.9, 134.3, 130.7, 129.1, 128.7, 127.0(2), 126.9(7), 126.3, 124.4, 124.1, 123.5, 121.9, 110.1, 101.9, 46.2, 45.7, 44.4; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}BrN_2O_2^+$ 433.0546, found 433.0542.



Dihydroisoindigo 5i: Obtained through direct single-run filtration; White solid (65.9 mg, 96% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.48 (s, 1H), 10.38 (s, 1H), 7.37 (dd, J = 8.5, 1.5 Hz, 1H), 7.20 (t, J = 7.8 Hz, 1H), 6.92 (*br* s, 1H), 6.88 (t, J = 7.5 Hz, 1H), 6.82 (d, J = 8.0 Hz, 1H), 6.76-6.79 (m, 2H), 4.24 (d, J = 3.5 Hz, 1H), 4.22 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 176.4, 176.1, 143.8, 143.2, 131.4, 130.3, 128.9, 127.3, 126.8, 123.9, 121.8, 113.3, 111.7, 109.9, 46.3, 46.0; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{16}H_{12}BrN_2O_2^+$ 343.0077, found 343.0075.

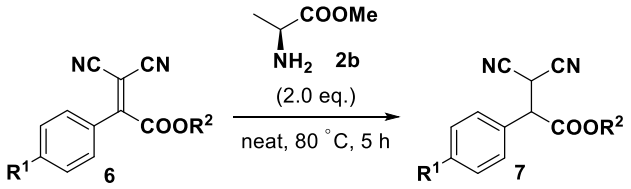


Dihydroisoindigo 5j: Obtained through direct single-run filtration; White solid (80.8 mg, 93% yield); ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.85 (s, 1H), 7.38 (d, J = 4.0 Hz, 4H), 7.28-7.31 (m, 1H), 7.26 (dd, J = 8.0, 2.0 Hz, 1H), 7.11 (t, J = 8.0 Hz, 1H), 7.02 (d, J = 7.0 Hz, 1H), 6.95 (*br* s, 1H), 6.88 (t, J = 7.5 Hz, 1H), 6.83 (d, J = 8.0 Hz, 1H), 6.72 (d, J = 8.0 Hz, 1H), 5.07 (d, J = 15.5 Hz, 1H), 4.87 (d, J = 16.0 Hz, 1H), 4.36 (d, J = 3.5 Hz, 1H), 4.31 (d, J = 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 177.1, 175.7, 143.7, 142.7, 136.6, 131.5, 129.3, 128.8, 127.9, 127.7, 126.5, 125.6, 123.5, 122.8, 113.4, 111.9, 109.8, 46.1, 45.8, 43.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{18}BrN_2O_2^+$ 433.0546, found 433.0542.



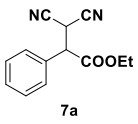
Dihydroisoindigo 5k: Obtained through direct single-run filtration; White solid (50.3 mg, 95% yield); ^{1}H NMR (500 MHz, DMSO-d_6, *mixture of two diastereomers*) δ 10.68 (s, 0.4H), 10.33 (s, 2.0H), 7.18 (t, J = 7.5 Hz, 2.0H), 7.04 (t, J = 7.5 Hz, 0.5H), 6.88 (d, J = 7.5 Hz, 0.5H), 6.85 (t, J = 7.5 Hz, 1.9H), 6.80 (d, J = 7.5 Hz, 2.0H), 6.77 (d, J = 7.5 Hz, 2.1H), 6.73 (t, J = 7.5 Hz, 0.6H), 4.17 (s, 2.0H), 4.07 (s, 0.5H); ^{13}C NMR (125 MHz, DMSO-d_6, *major diastereomer*) δ 176.6, 143.8, 128.7, 127.7, 123.9, 121.7, 109.8, 46.2; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{16}H_{13}N_2O_2^+$ 265.0972, found 265.0949.

2.3. General procedure for the synthesis of tetrasubstituted ethanes 7



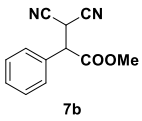
Tetrasubstituted olefins **6** (0.2 mmol, 1.0 equiv.) and L-alanine methyl ester **2b** (0.40 mmol, 2.0 equiv.) were heated at 80 ℃ for 5 h. After completion of the reaction, the

reaction mixture was purified by flash column chromatography on silica gel (EtOAc/PE = 5%-10%) to yield tetrasubstituted ethanes **7**.



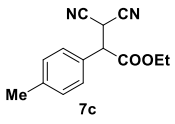
7a

Tetrasubstituted ethane 7a: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 5%-10% (v/v); Colorless oil (42.9 mg, 94% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.47 (m, 3H), 7.35-7.38 (m, 2H), 4.40 (d, J = 9.0 Hz, 1H), 4.30-4.37 (m, 1H), 4.23-4.30 (m, 1H), 4.21 (d, J = 9.0 Hz, 1H), 1.28 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.6, 131.7, 129.9, 129.7, 128.2, 111.5, 110.9, 63.0, 51.9, 27.0, 13.9; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{13}H_{13}N_2O_2^+$ 229.0972, found 229.0955.



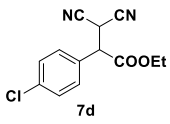
7b

Tetrasubstituted ethane 7b: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 5%-10% (v/v); Colorless oil (41.0 mg, 96% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44-7.45 (m, 3H), 7.32-7.34 (m, 2H), 4.39 (d, J = 8.5 Hz, 1H), 4.21 (d, J = 8.5 Hz, 1H), 3.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.1, 131.5, 130.0, 129.7, 128.2, 111.5, 110.8, 53.6, 51.8, 27.0; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{12}H_{11}N_2O_2^+$ 215.0815, found 215.0813.



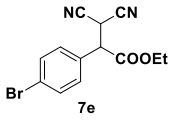
7c

Tetrasubstituted ethane 7c: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 5%-10% (v/v); Colorless oil (44.4 mg, 92% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 7.5 Hz, 2H), 4.35 (dd, J = 8.5, 1.5 Hz, 1H), 4.27-4.33 (m, 1H), 4.19-4.26 (m, 1H), 4.15 (d, J = 8.5 Hz, 1H), 2.37 (s, 3H), 1.25 (td, J = 7.3, 1.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.7, 140.0, 130.3, 128.7, 128.0, 111.7, 111.1, 62.9, 51.5, 27.1, 21.2, 13.9; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{14}H_{15}N_2O_2^+$ 243.1128, found 243.1127.



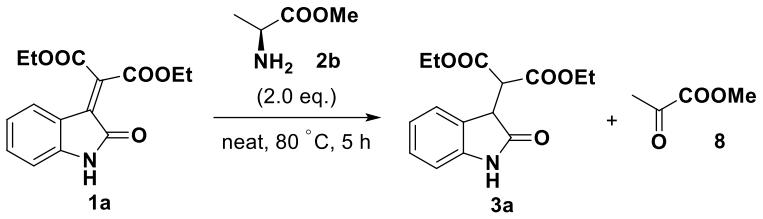
7d

Tetrasubstituted ethane 7d: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 5%-10% (v/v); Colorless oil (50.4 mg, 96% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42-7.44 (m, 2H), 7.28-7.30 (m, 2H), 4.37 (d, J = 8.5 Hz, 1H), 4.28-4.34 (m, 1H), 4.22-4.28 (m, 1H), 4.17 (d, J = 8.5 Hz, 1H), 1.26 (t, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.2, 136.2, 130.1, 129.9, 129.6, 111.3, 110.7, 63.2, 51.2, 26.9, 13.9; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{13}H_{12}ClN_2O_2^+$ 263.0582, found 263.0563.

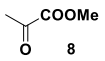


Tetrasubstituted ethane 7e: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 5%-10% (v/v); Colorless oil (58.2 mg, 95% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.0 Hz, 2H), 4.38 (d, J = 8.0 Hz, 1H), 4.28-4.34 (m, 1H), 4.21-4.27 (m, 1H), 4.16 (d, J = 8.5 Hz, 1H), 1.25 (t, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.1, 132.9, 130.6, 129.9, 124.4, 111.3, 110.8, 63.3, 51.2, 26.8, 13.9; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_{13}H_{12}BrN_2O_2^+$ 307.0077, found 307.0080.

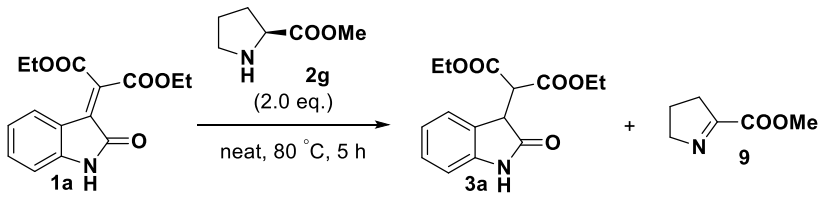
2.4. Synthetic procedure of 5 mmol scale model reaction



Disubstituted methyleneindolinone **1a** (1.45 g, 5.0 mmol, 1.0 equiv.) and L-alanine methyl ester **2b** (1.03 g, 10.0 mmol, 2.0 equiv.) were heated at 80 ℃ for 5 h. After completion of the reaction, the reaction mixture was purified by flash column chromatography on silica gel (EtOAc/PE = 2%-20%) to yield 3-substituted 2-oxindole **3a** in 93% yield (1.36 g, colorless oil) and methyl pyruvate **8** in 84% yield (0.43 g, colorless oil).

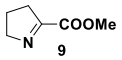


Methyl pyruvate 8[5]: Purified by flash chromatography on silica gel, eluting with ethyl acetate/petroleum ether 2% (v/v); Colorless oil (0.43 g, 84% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.88 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.6, 161.1, 52.9, 26.6; HRMS (FTMS-ESI) m/z: $[M+H]^+$ calcd for $C_4H_7O_3^+$ 103.0390, found 103.0389.



Disubstituted methyleneindolinone **1a** (1.45 g, 5.0 mmol, 1.0 equiv.) and L-proline methyl ester **2g** (1.29 g, 10.0 mmol, 2.0 equiv.) were heated at 80 ℃ for 5 h. After completion of the reaction, the reaction mixture was purified by flash column chromatography on silica gel (EtOAc/PE = 15%-20%) to yield 3-substituted 2-oxindole **3a** in 88% yield (1.28 g, colorless oil). The imine **9** was obtained by vacuum distillation

using a short-path distillation apparatus (bp: 85-90 °C at 10 mbar) as a colorless oil (0.48 g, 75% yield).



Imine 9[6]: Purified by vacuum distillation; Colorless oil (0.48 g, 75% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.08-4.12 (m, 2H), 3.87 (s, 3H), 2.81-2.84 (m, 2H), 1.95-2.02 (m, 2H).

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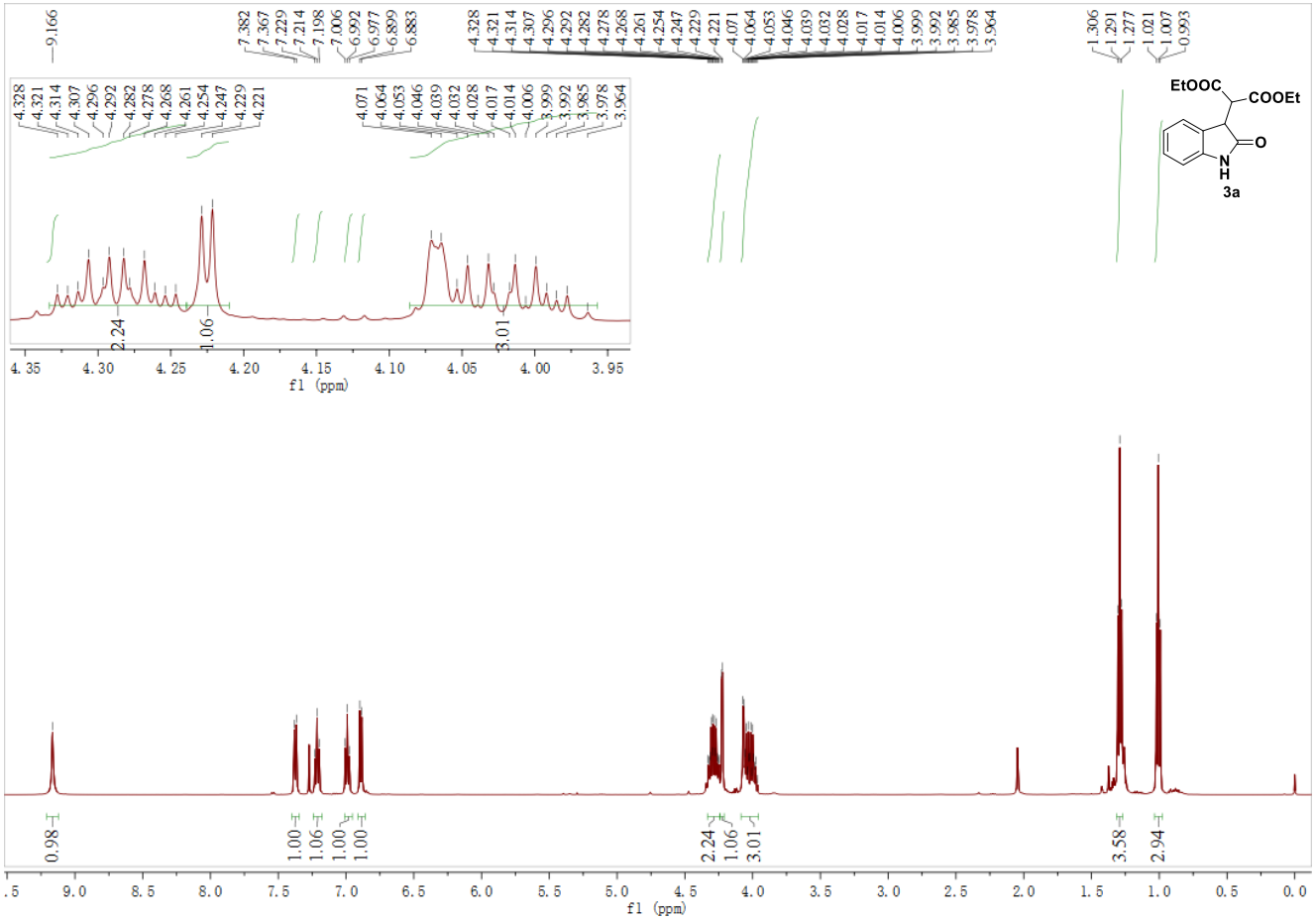
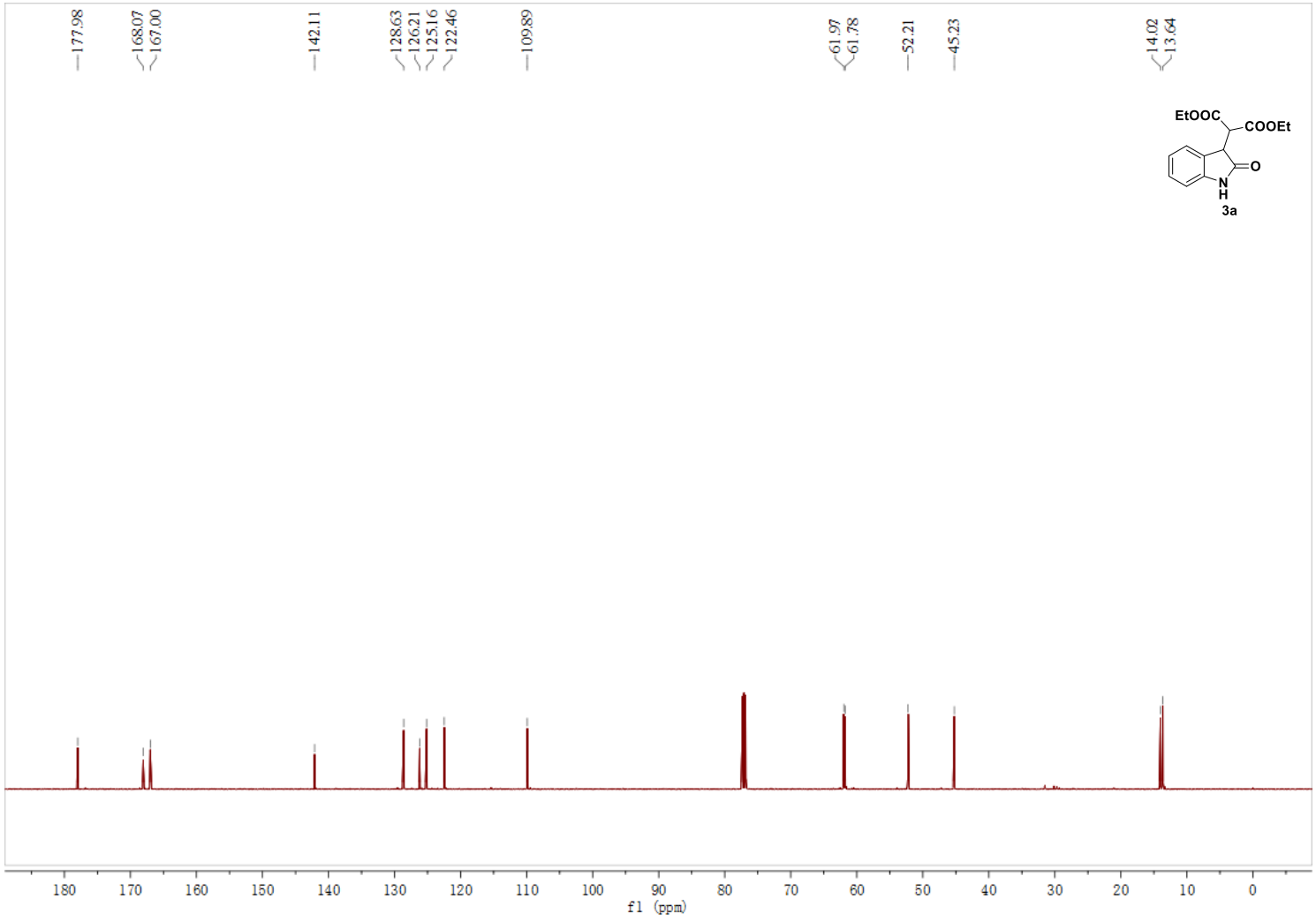
3. NMR Spectra

500 MHz, $CDCl_3$, ^1H NMR



125 MHz, $CDCl_3$, ^{13}C NMR



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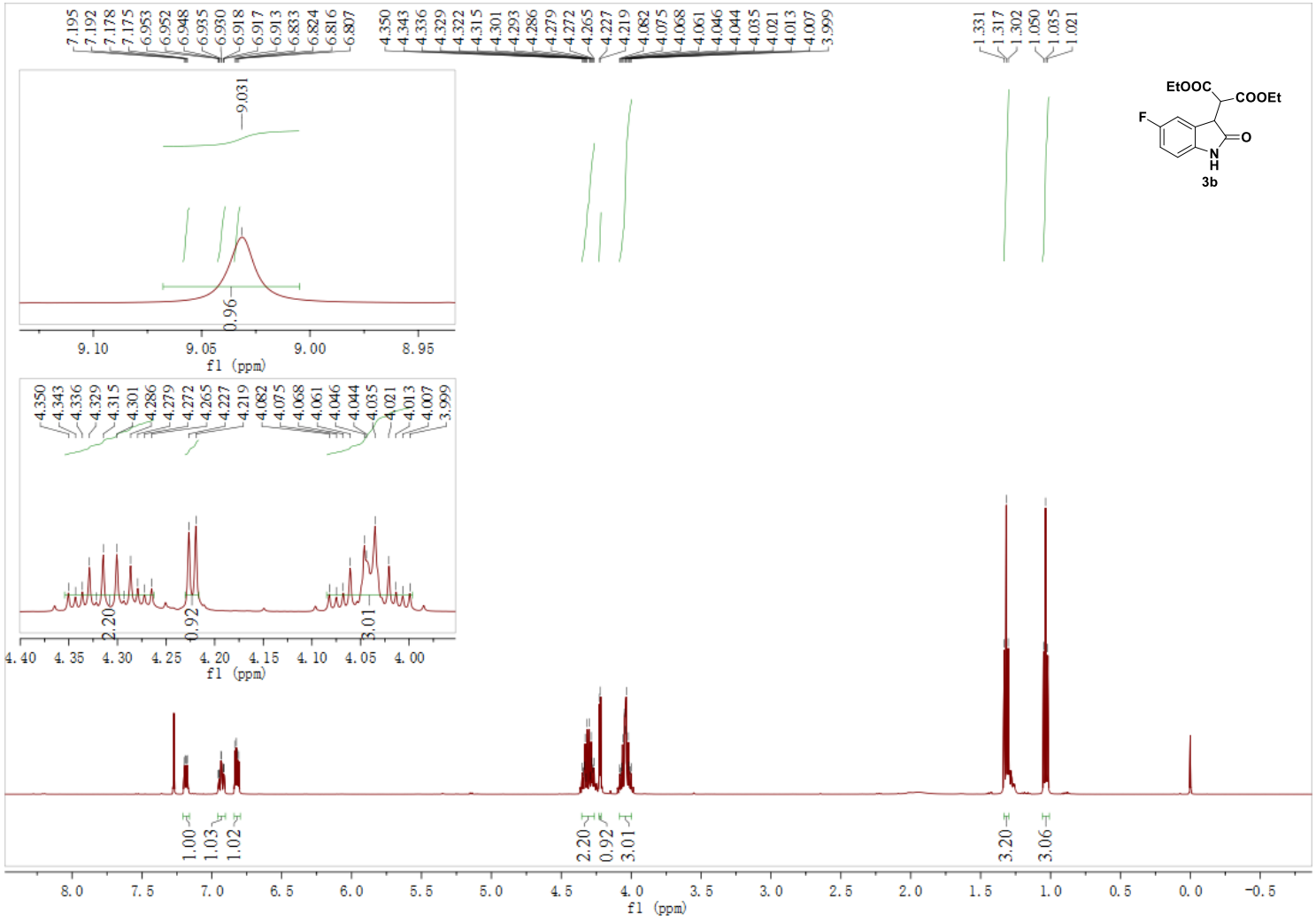

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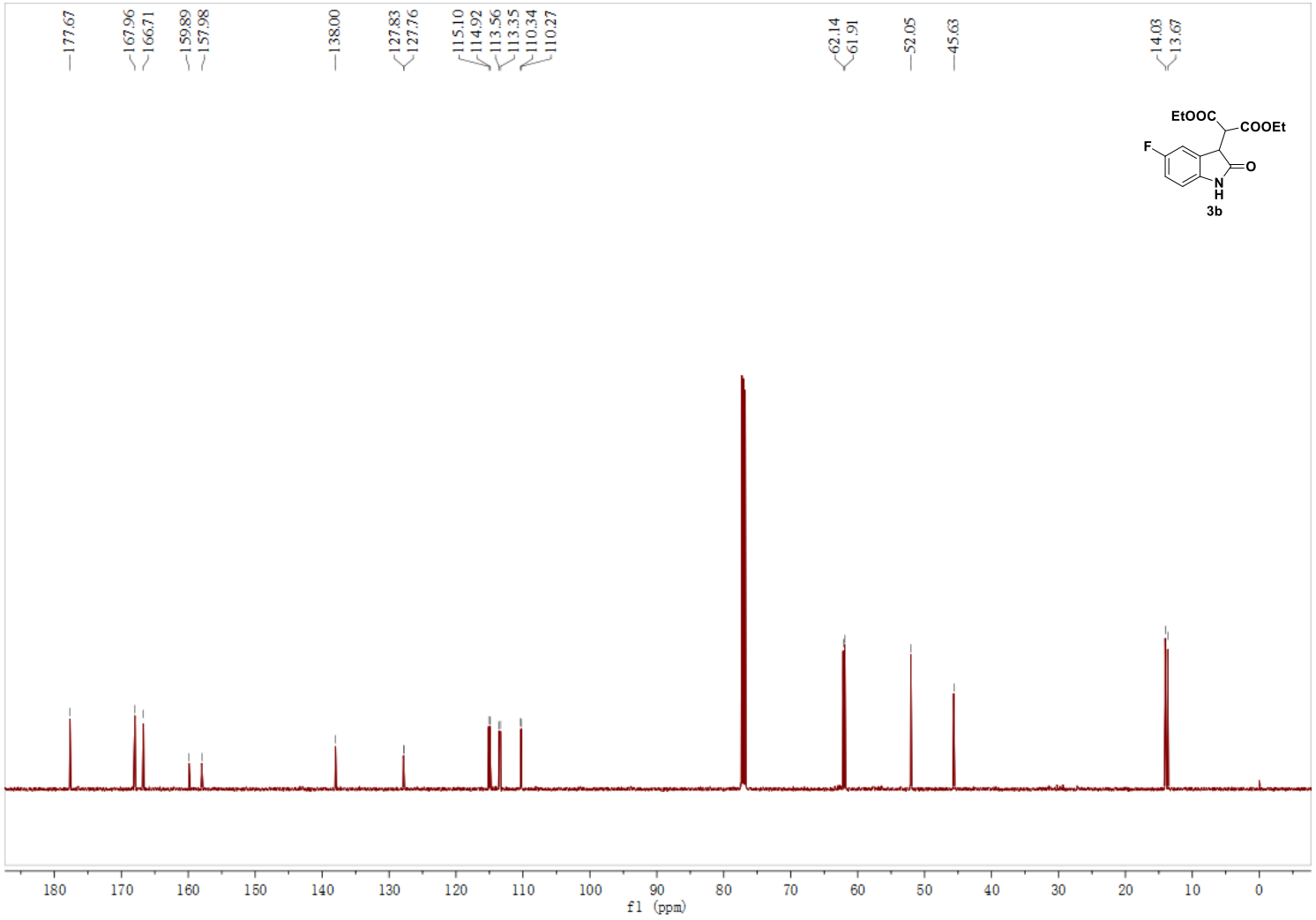

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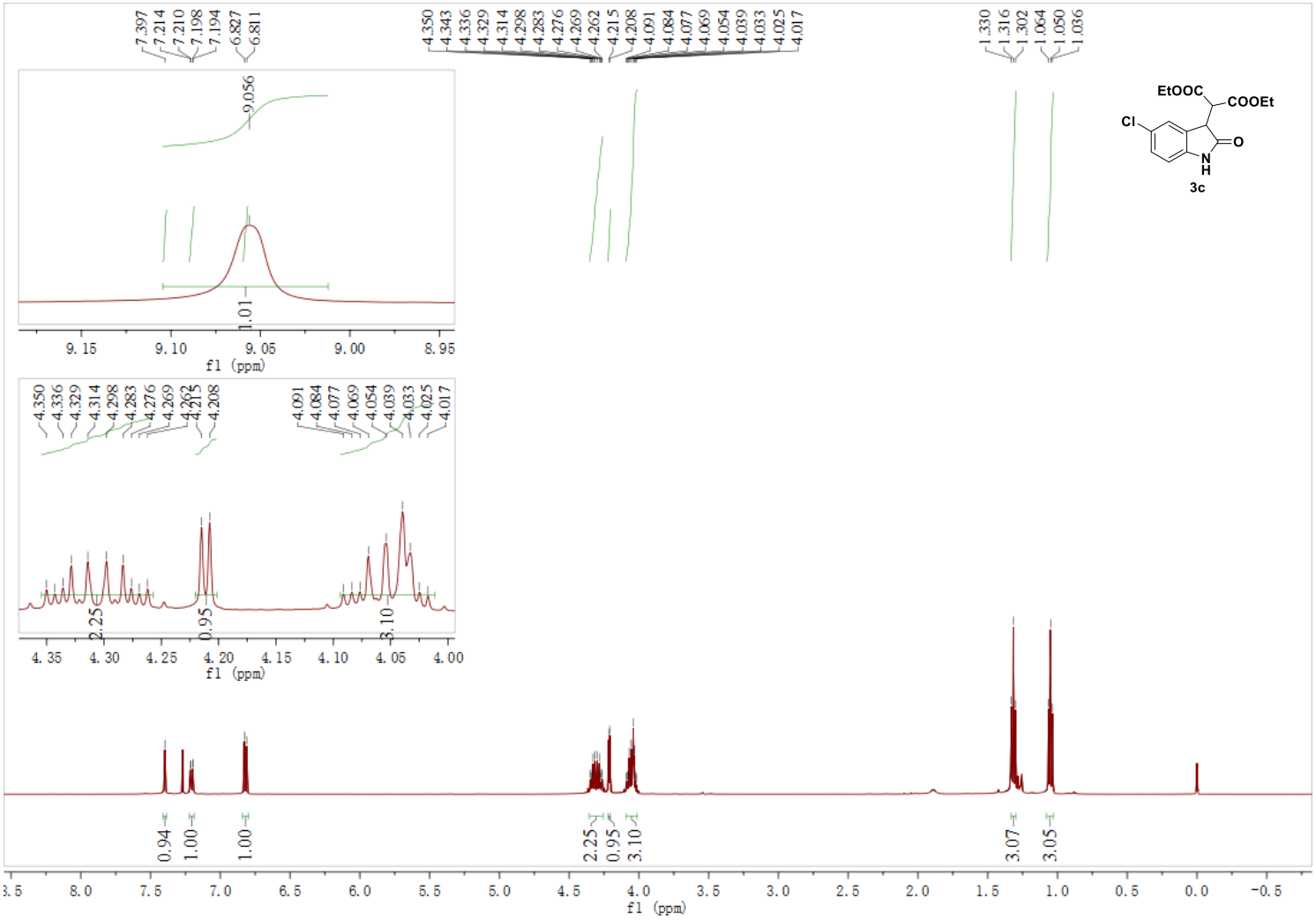

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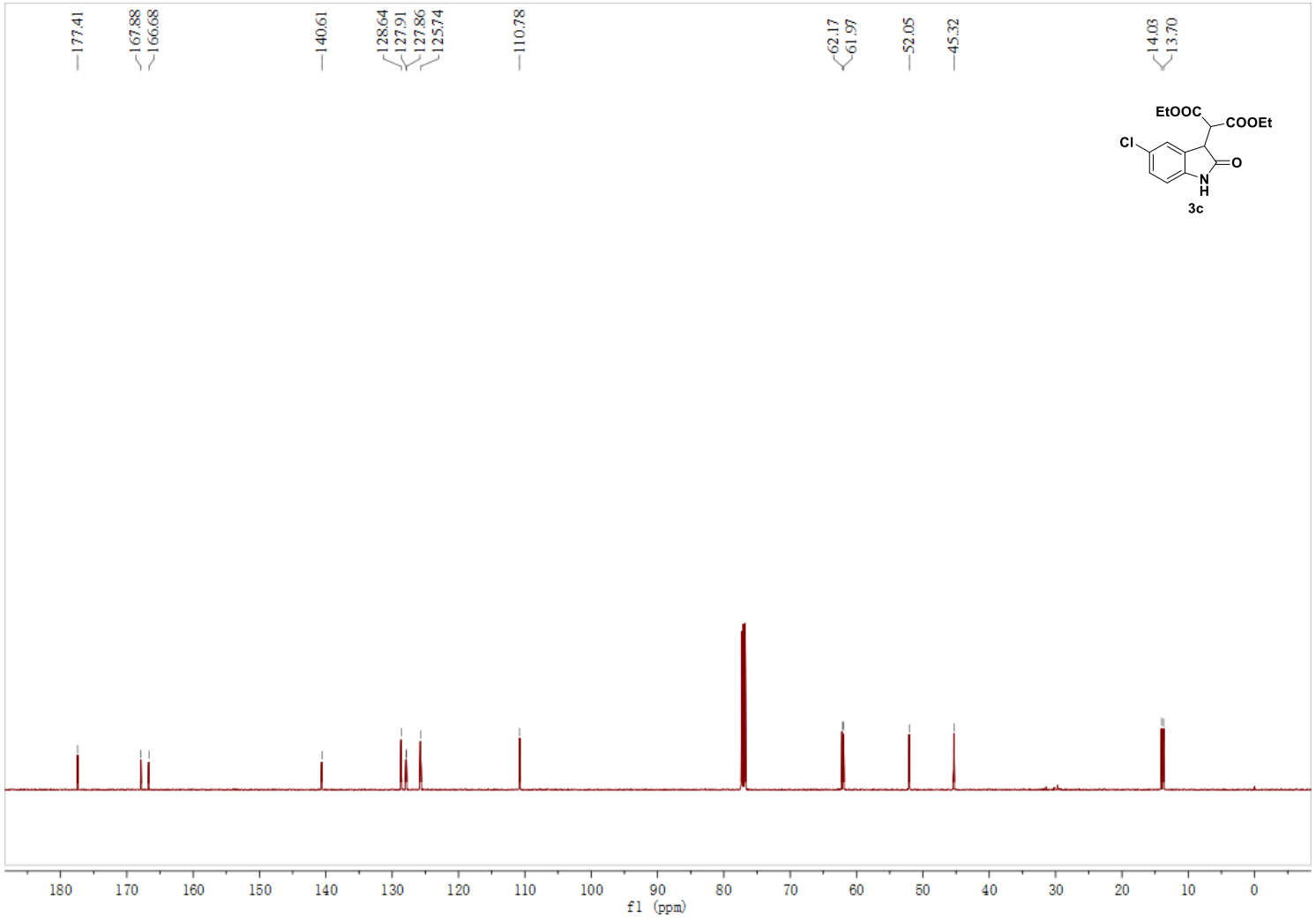

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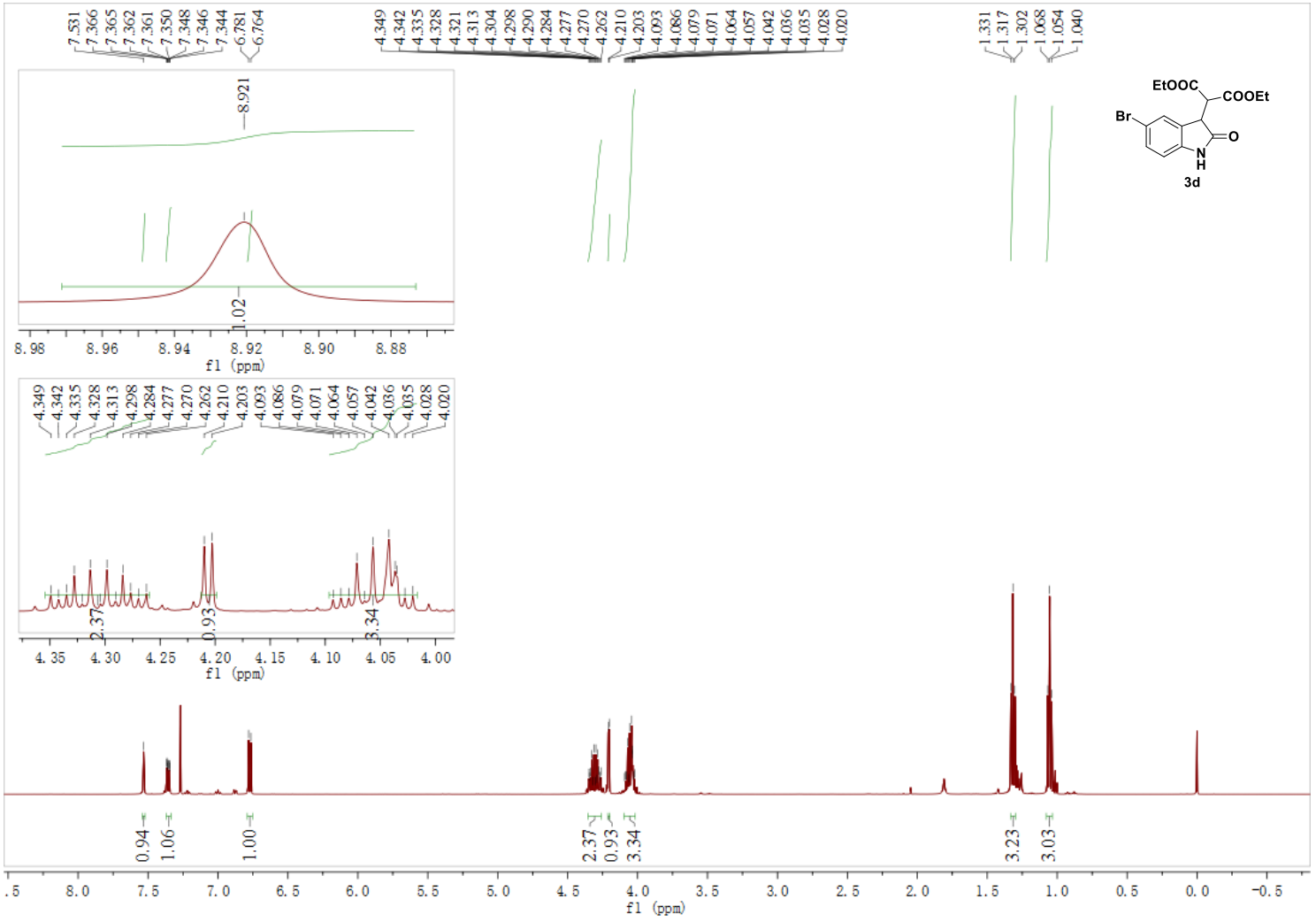

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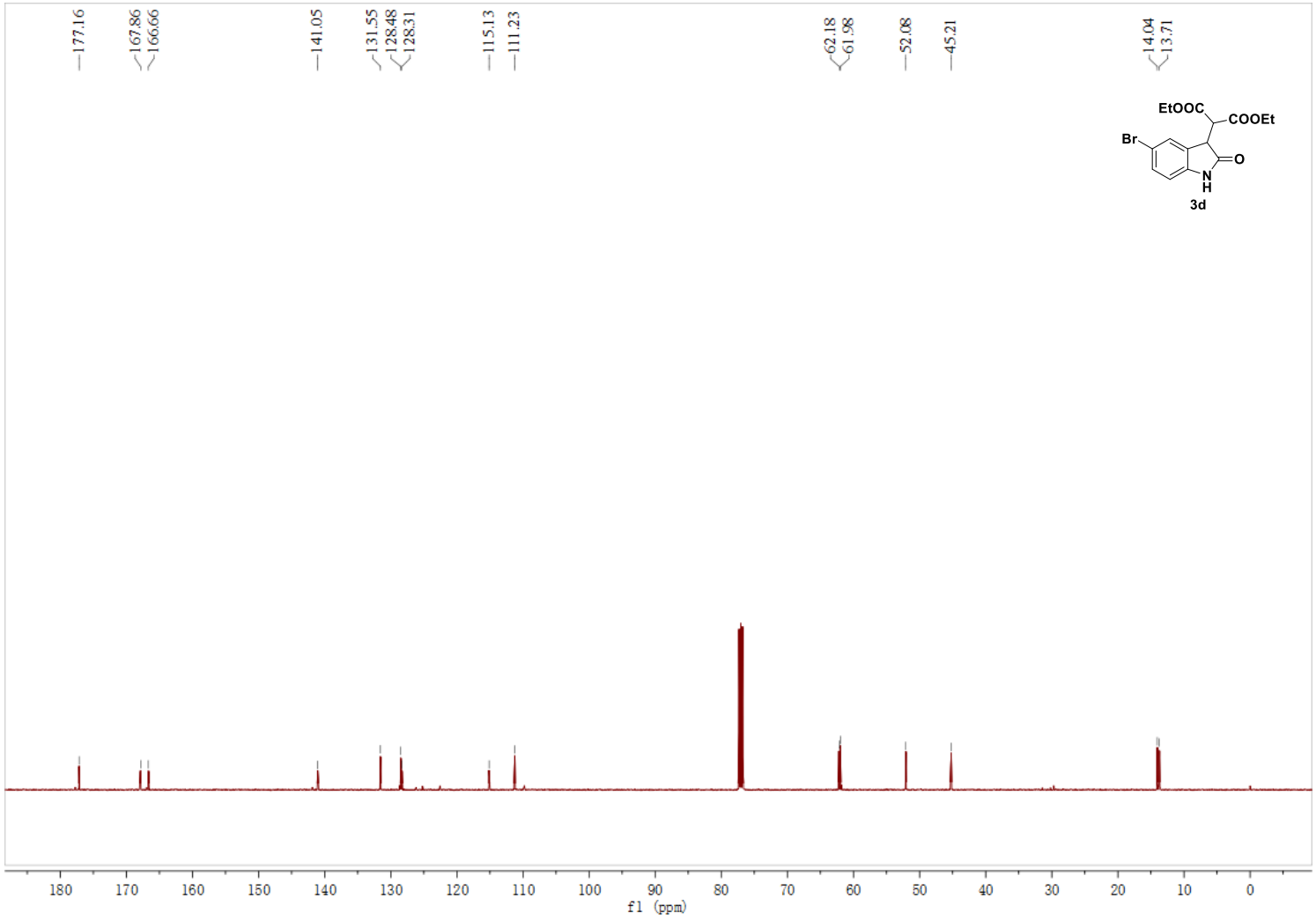

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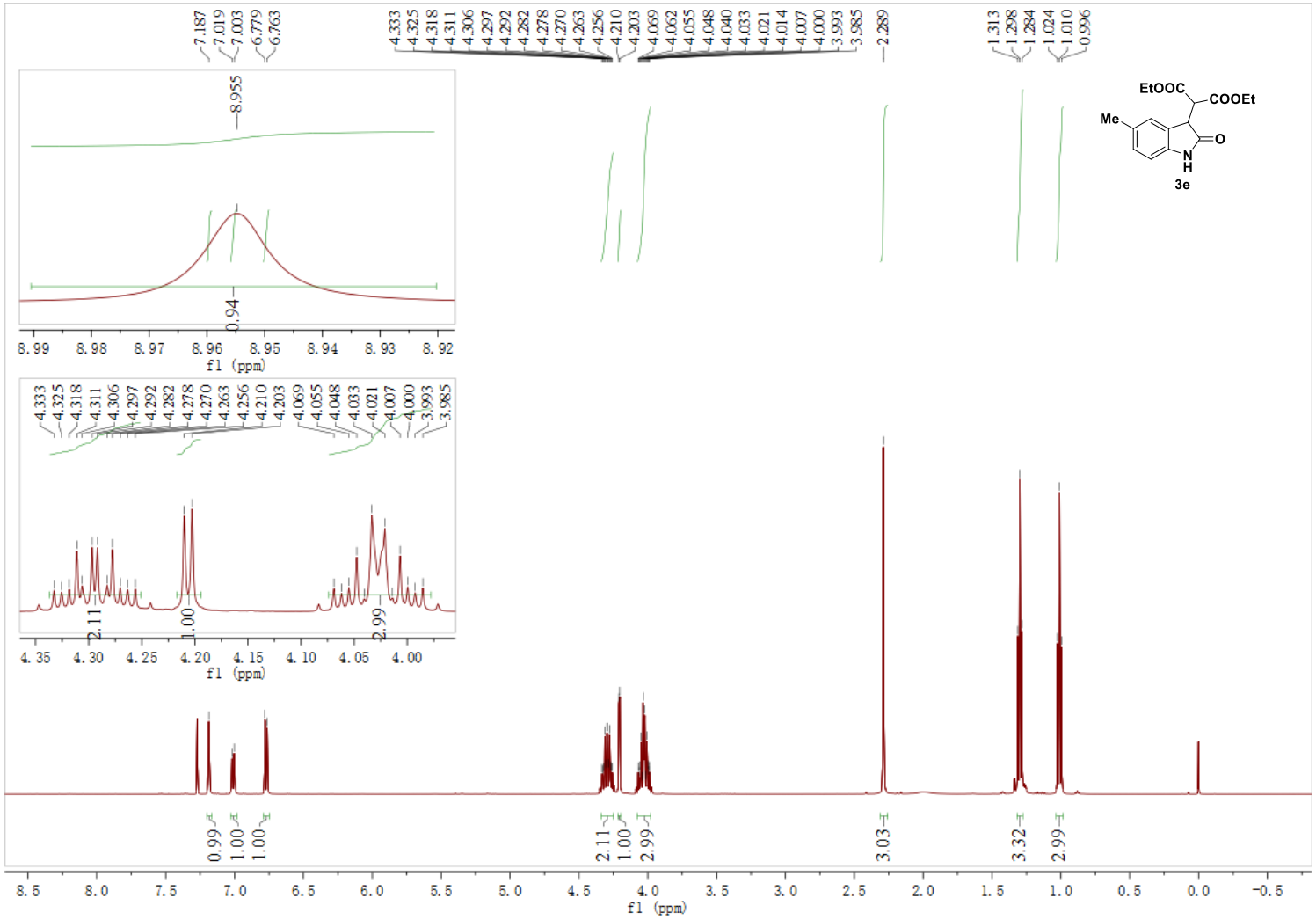

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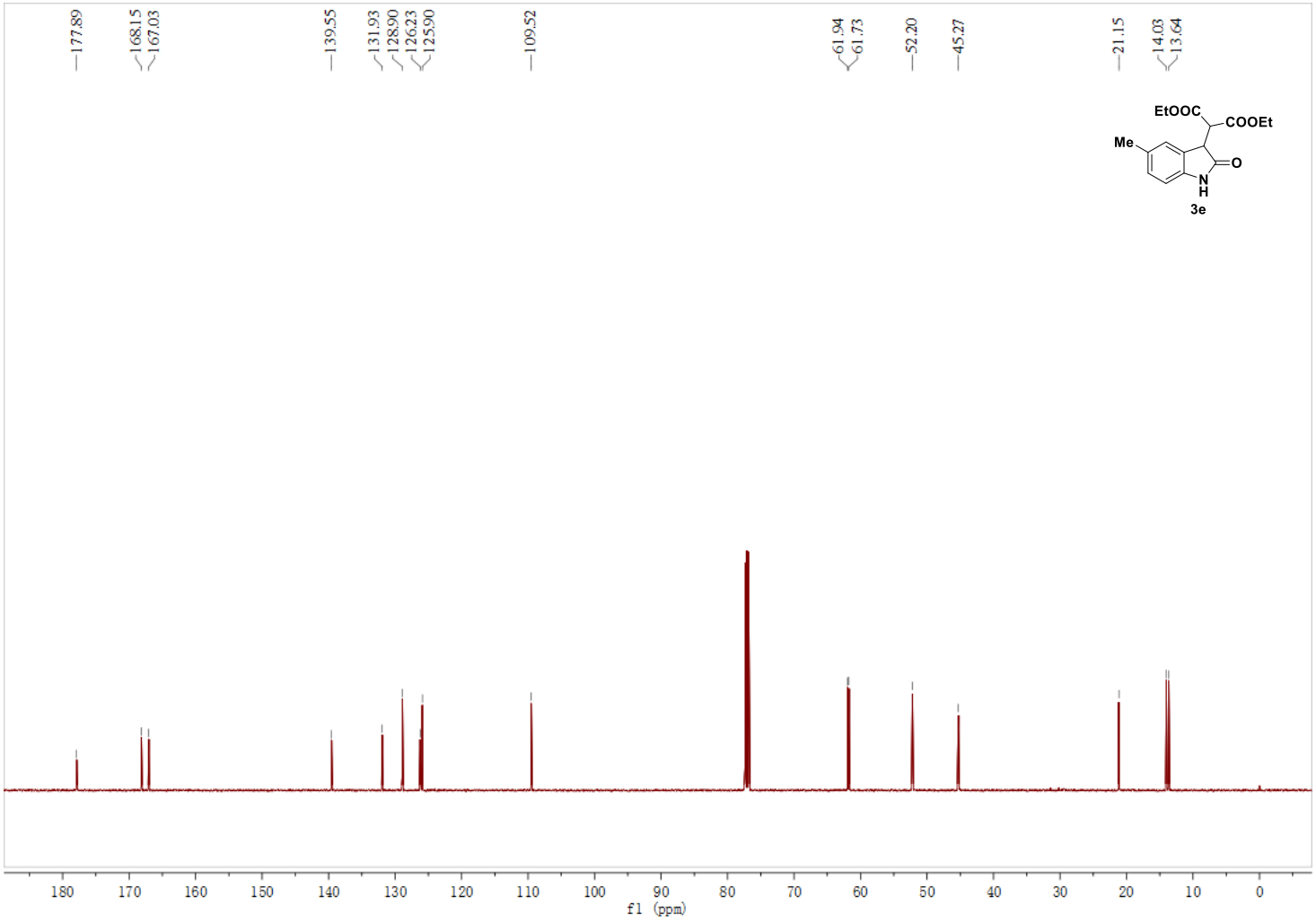

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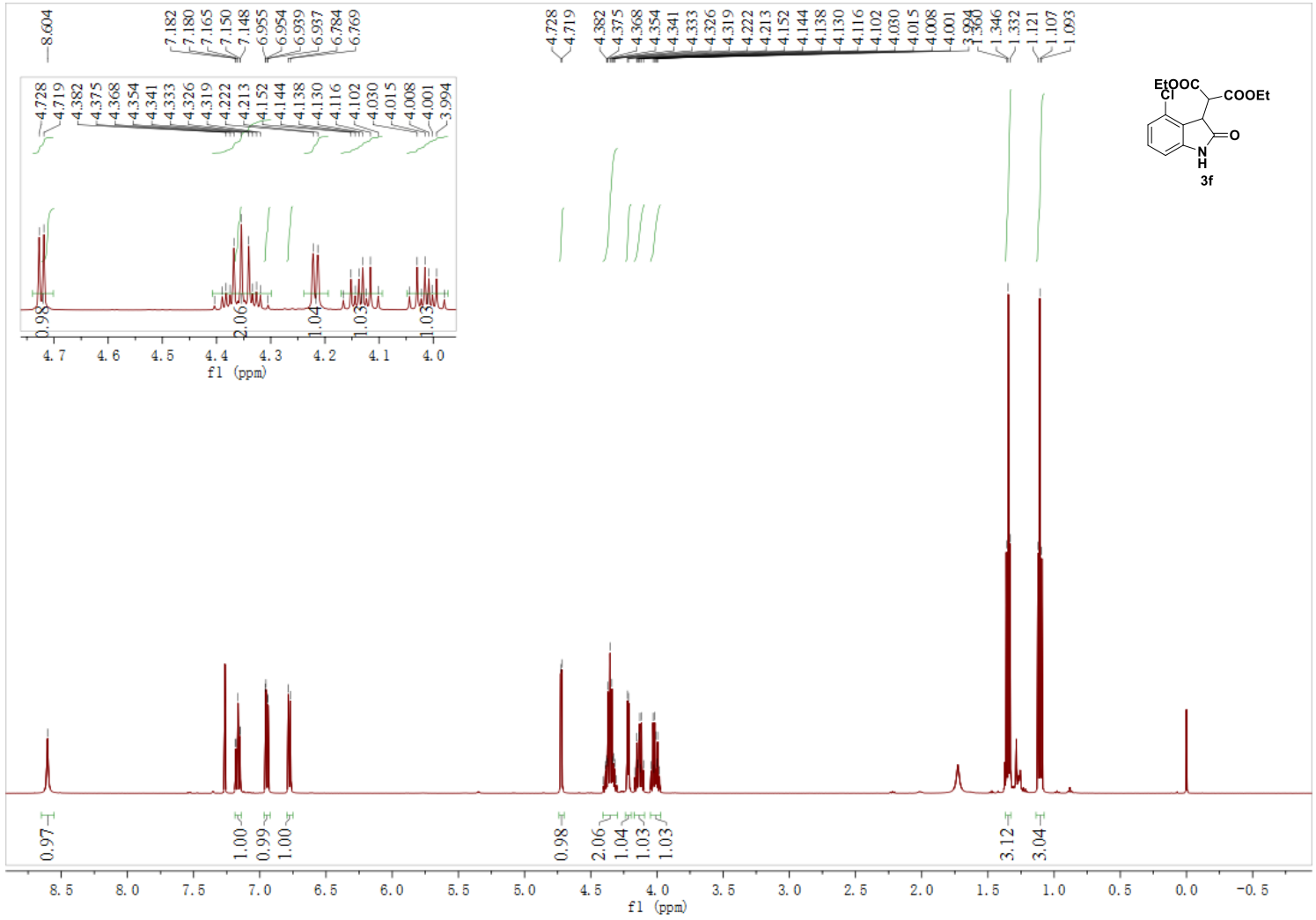

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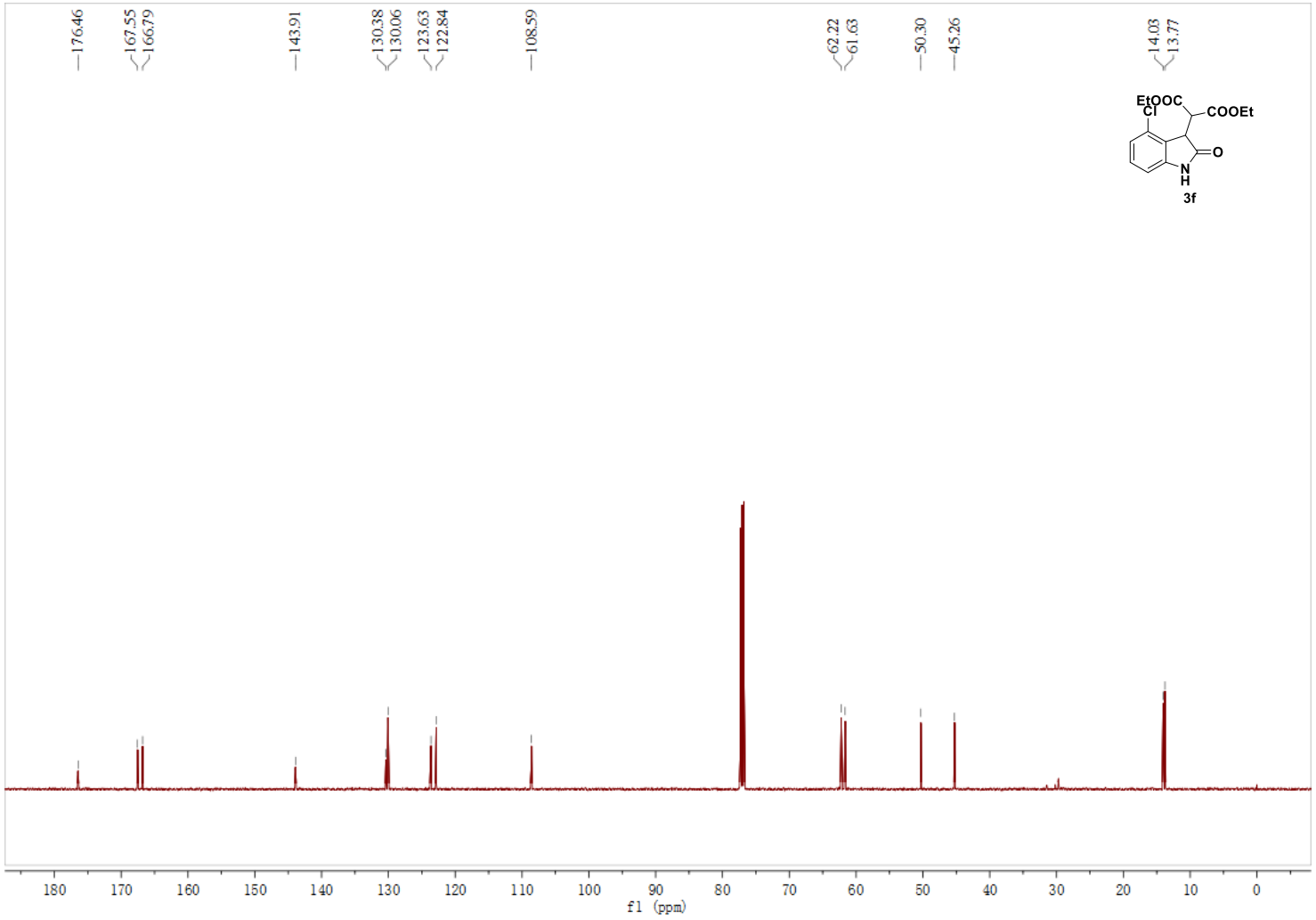

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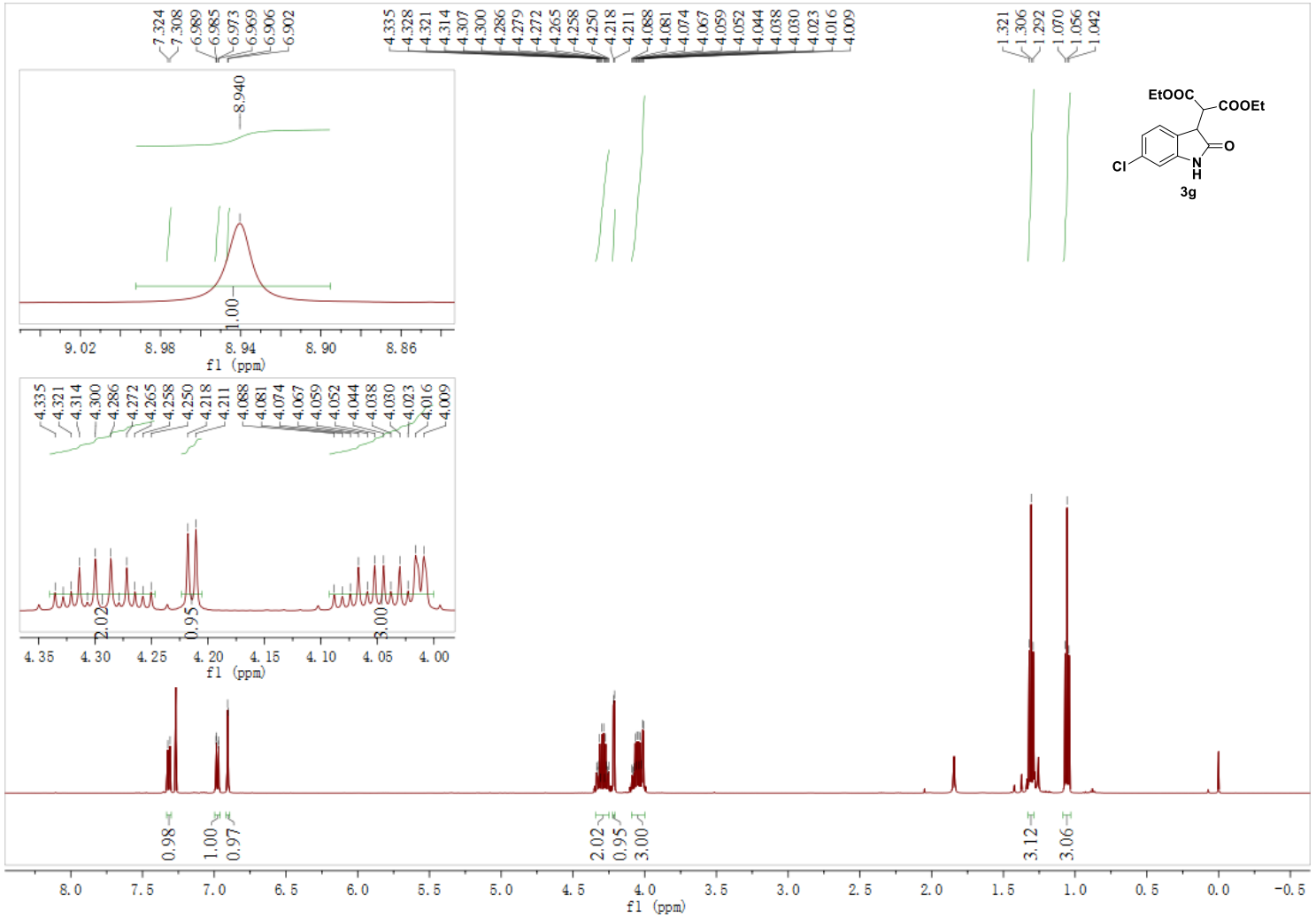

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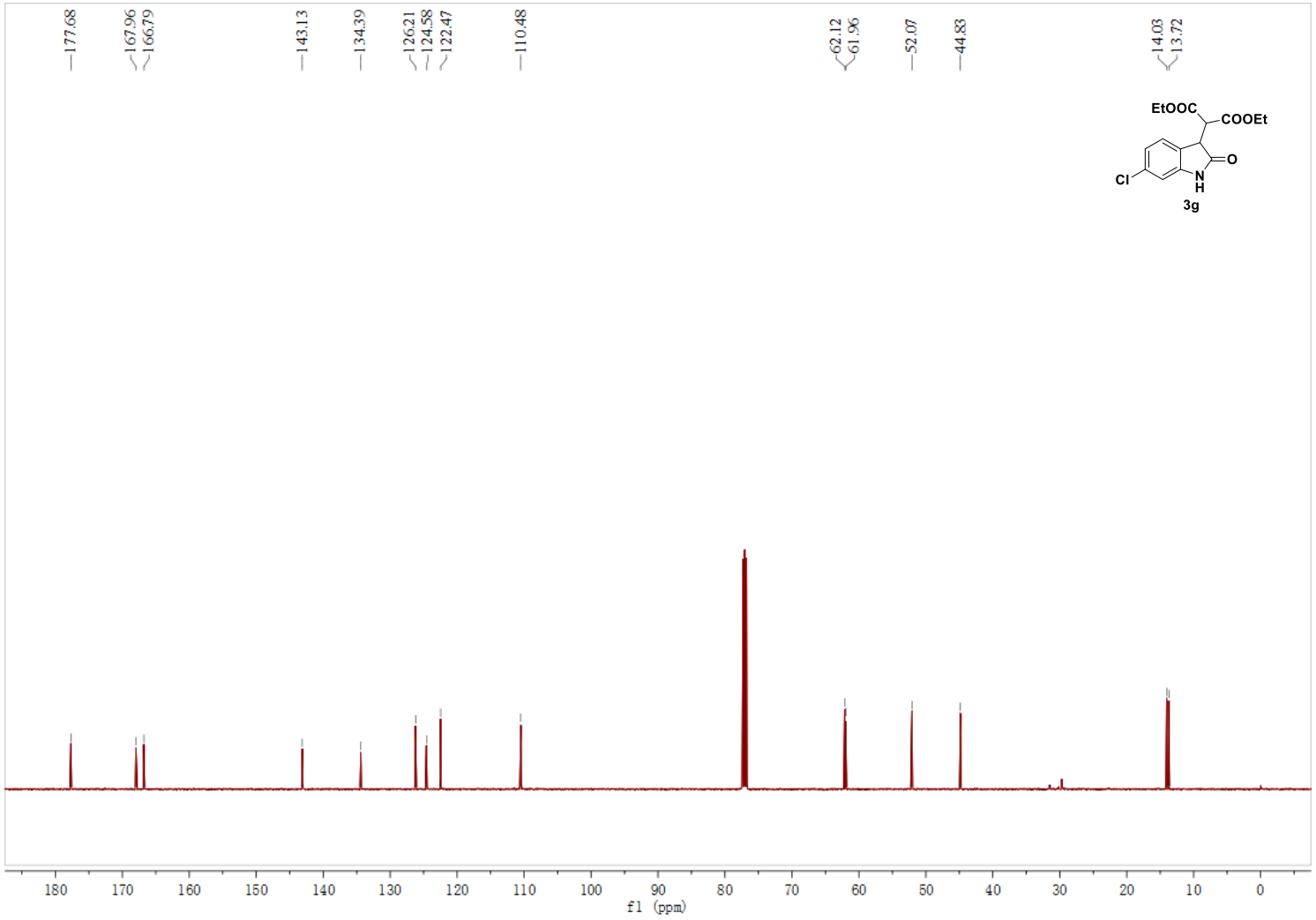

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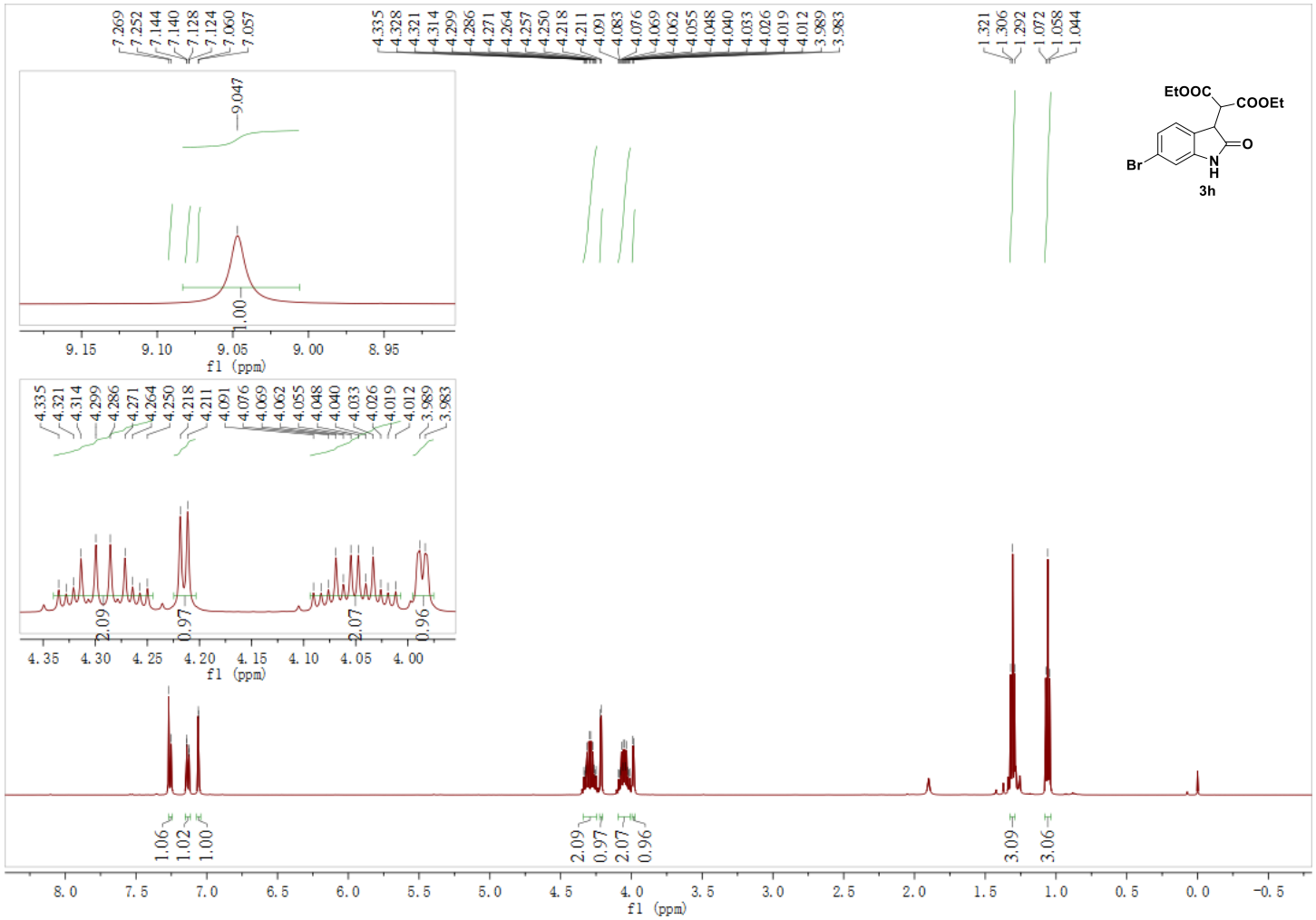

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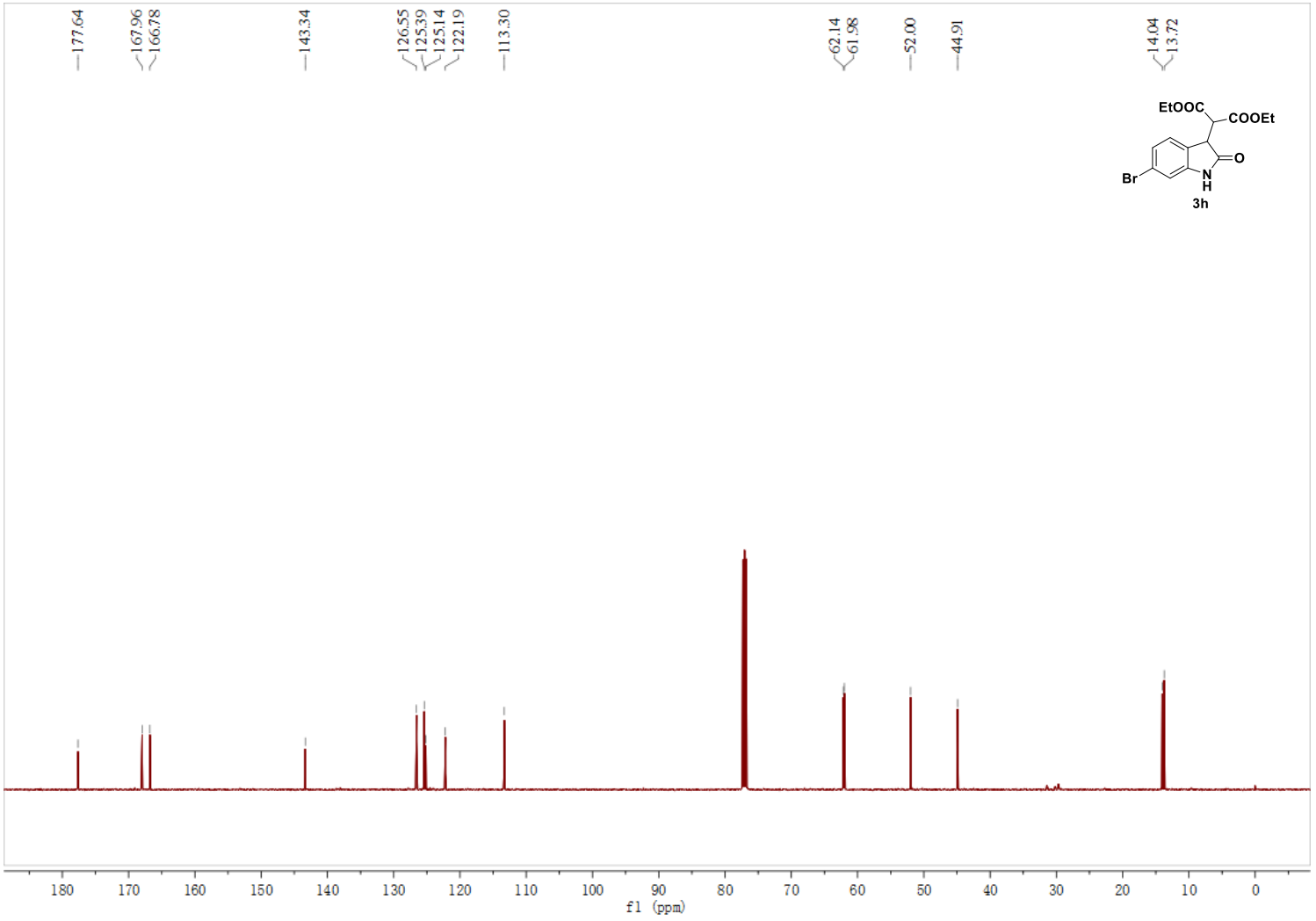

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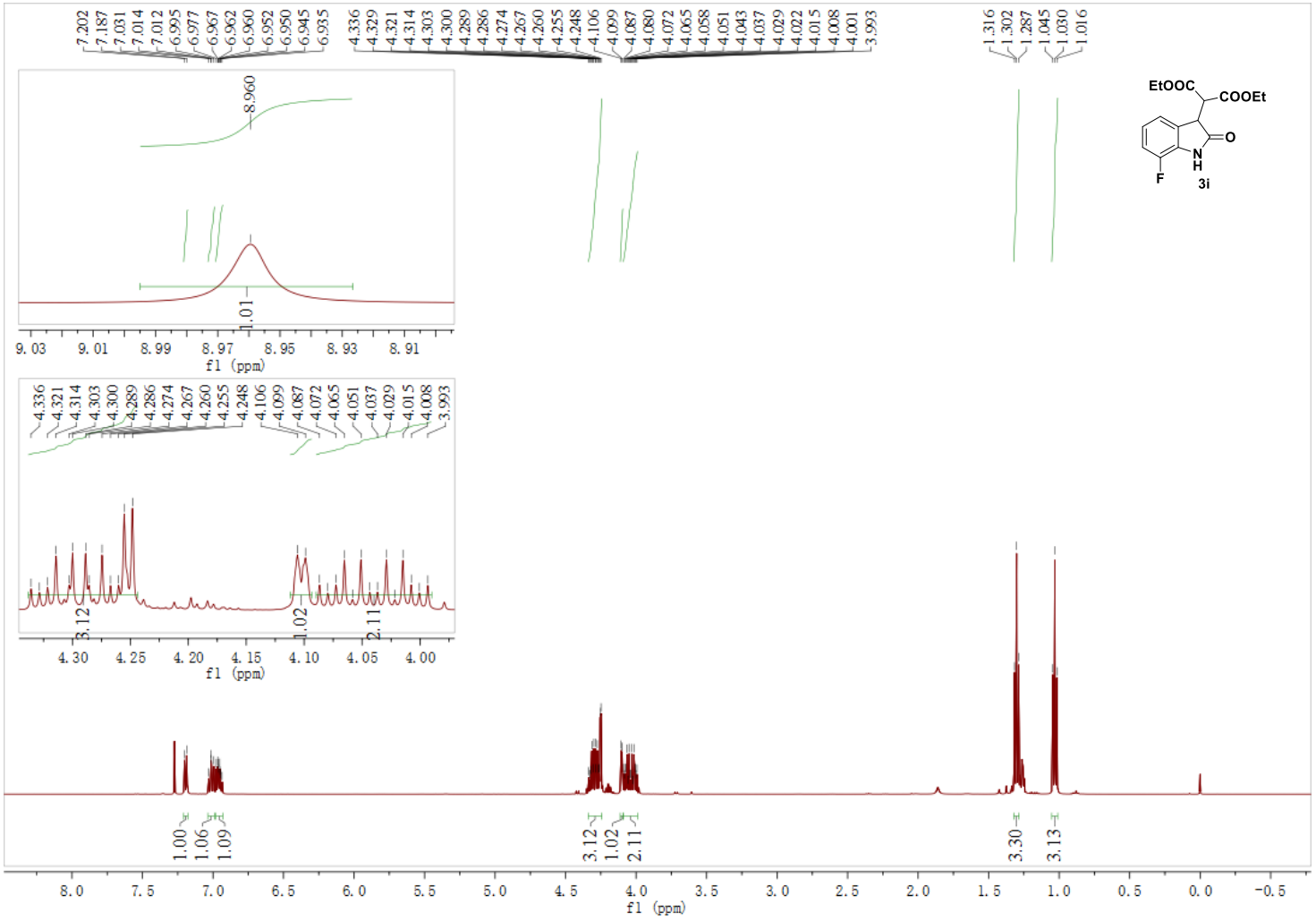

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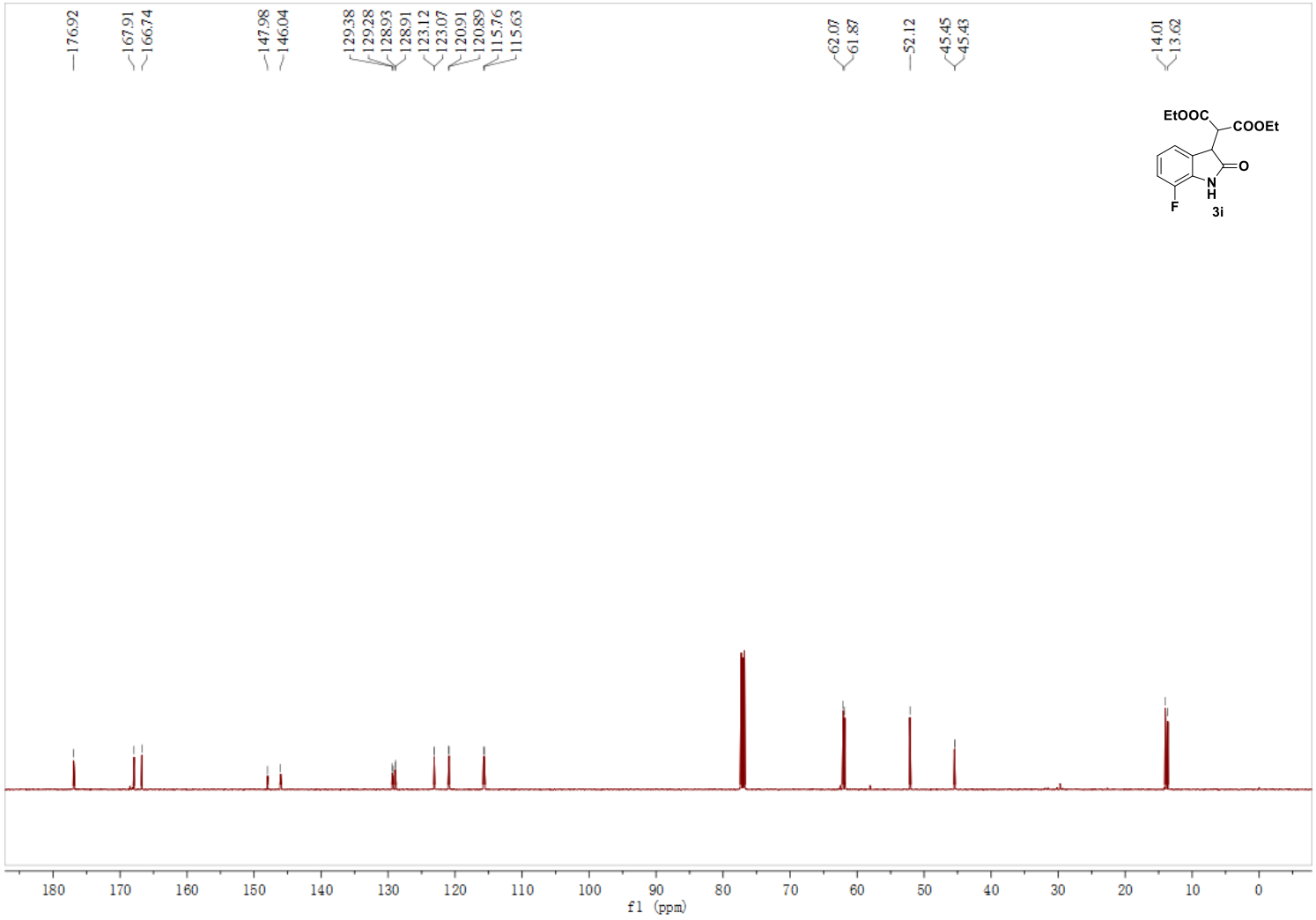

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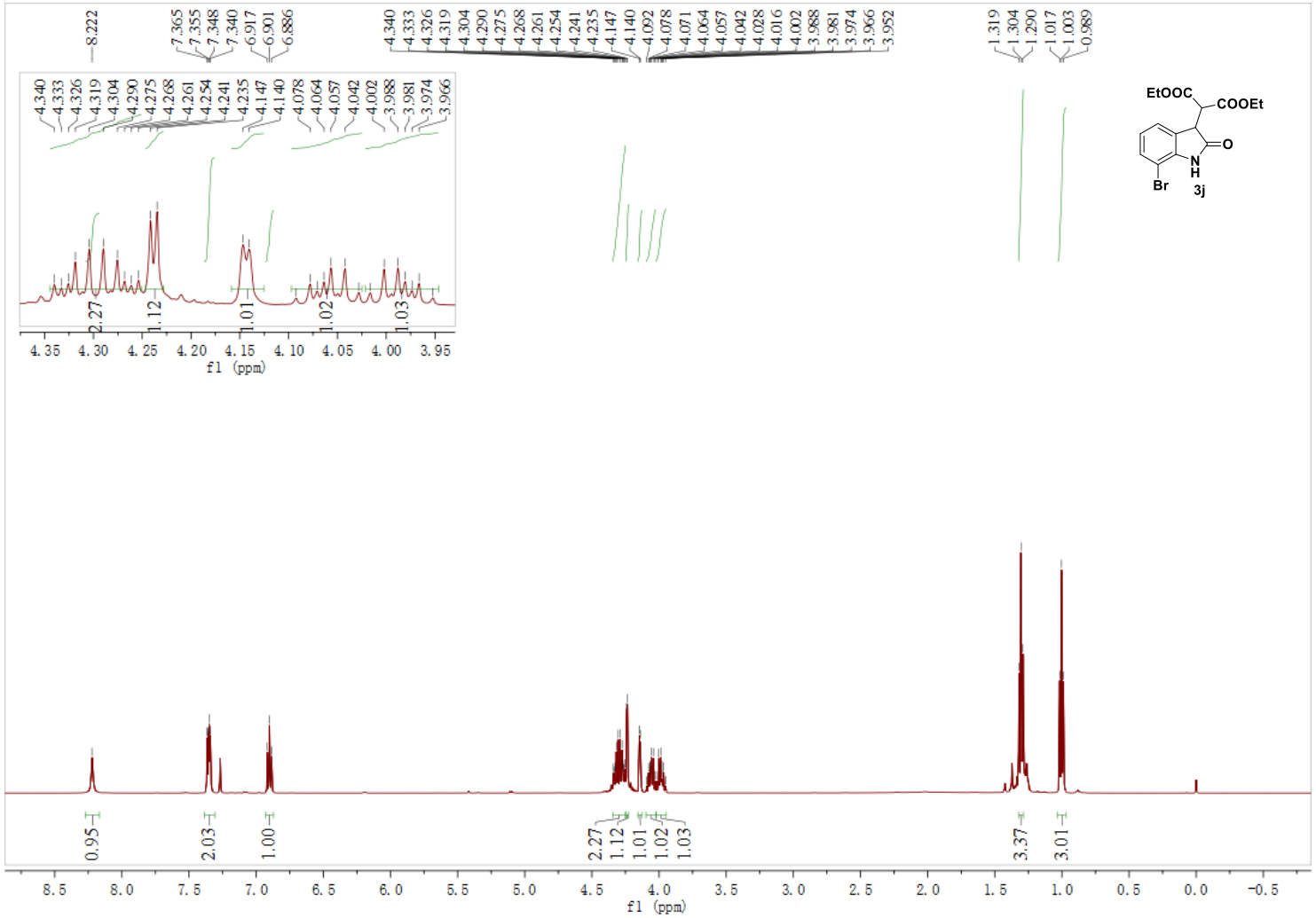

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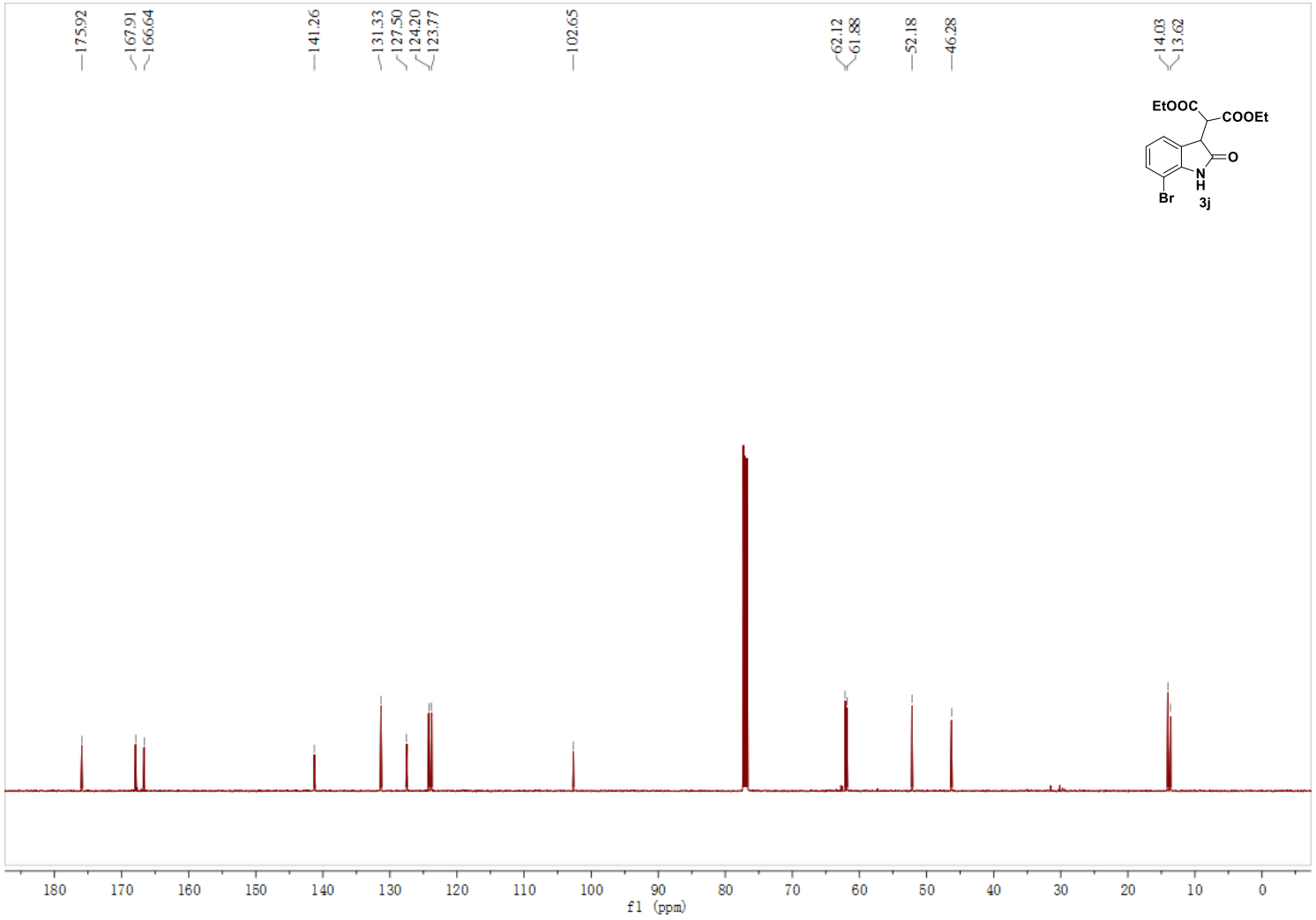

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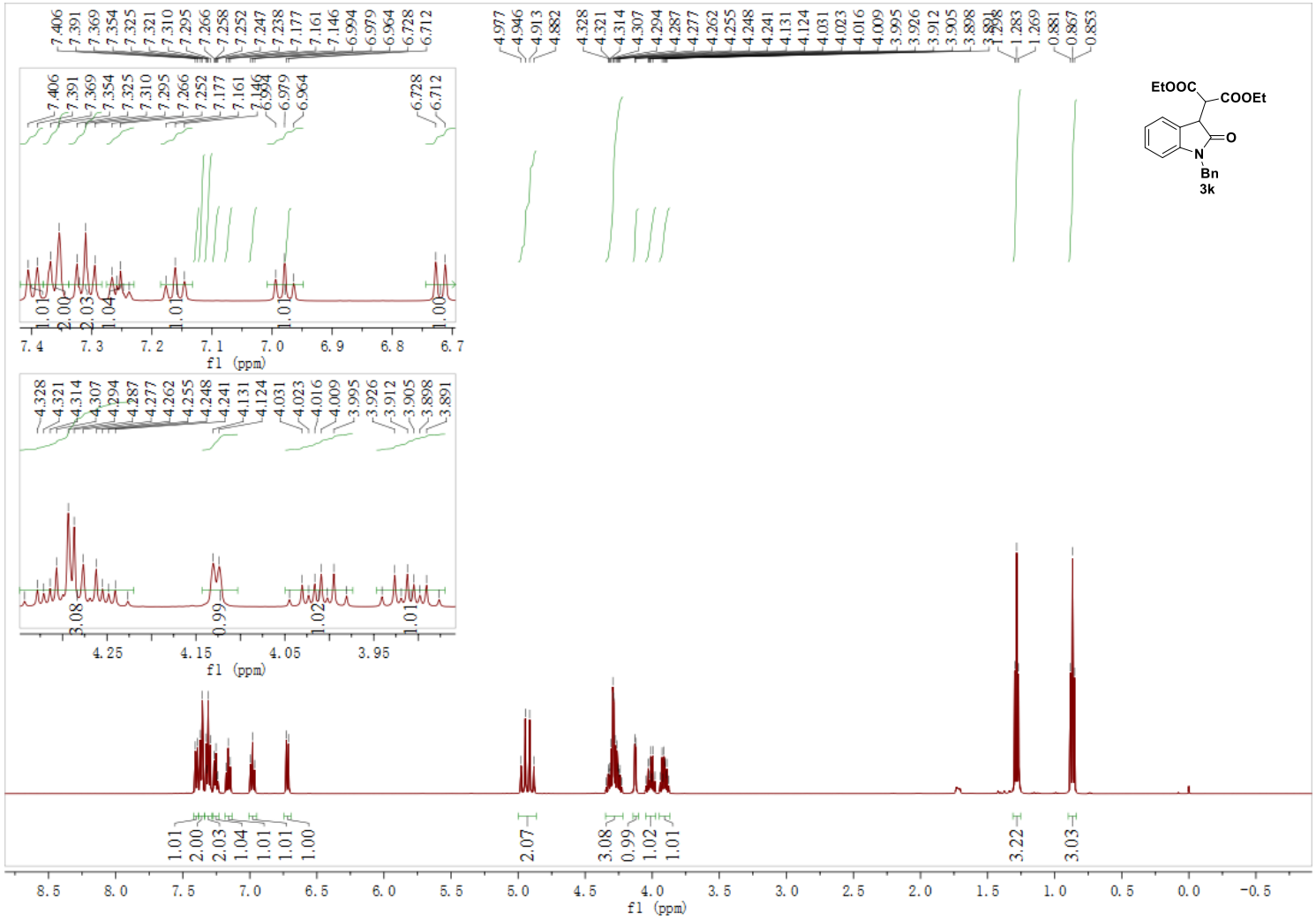

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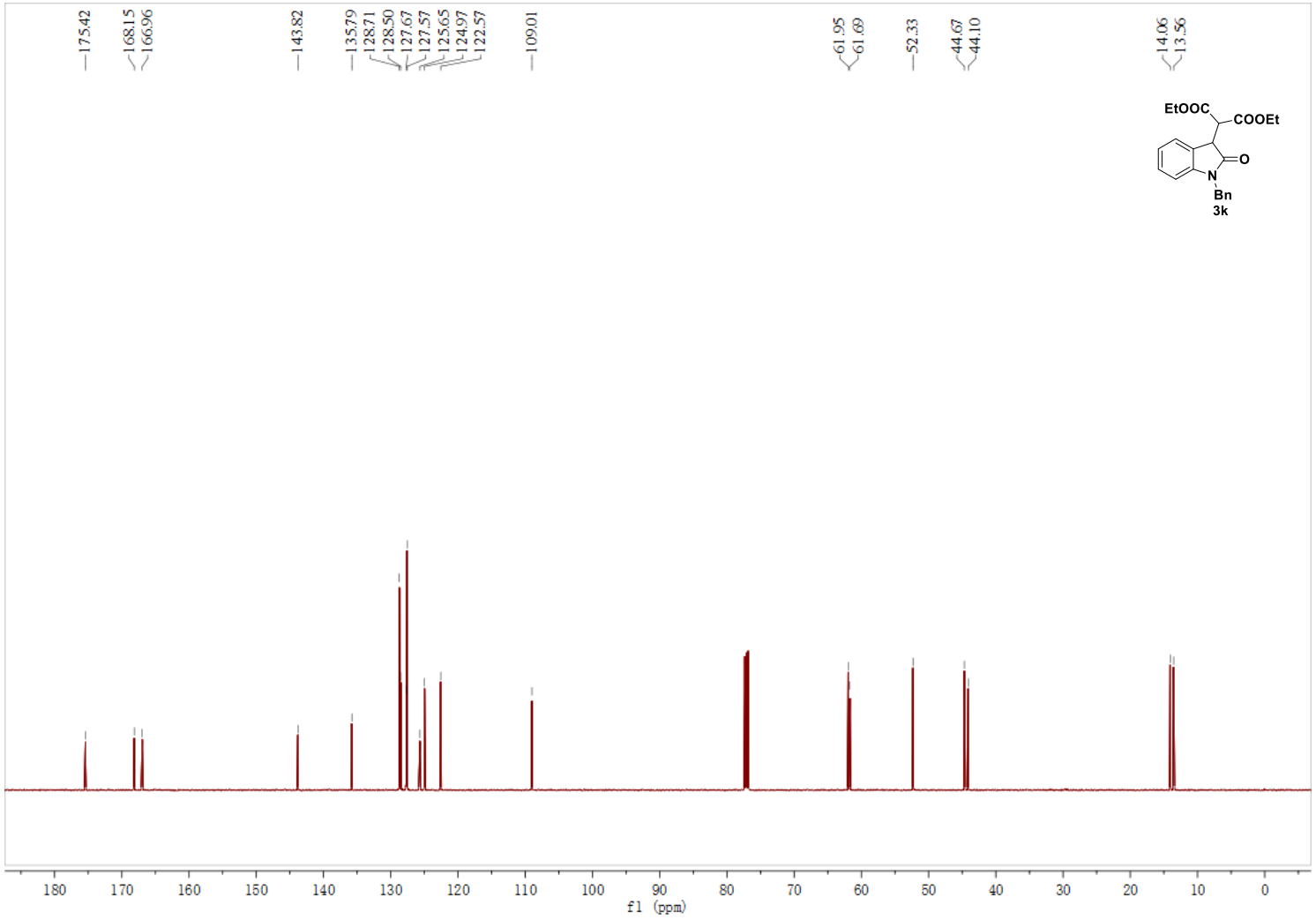

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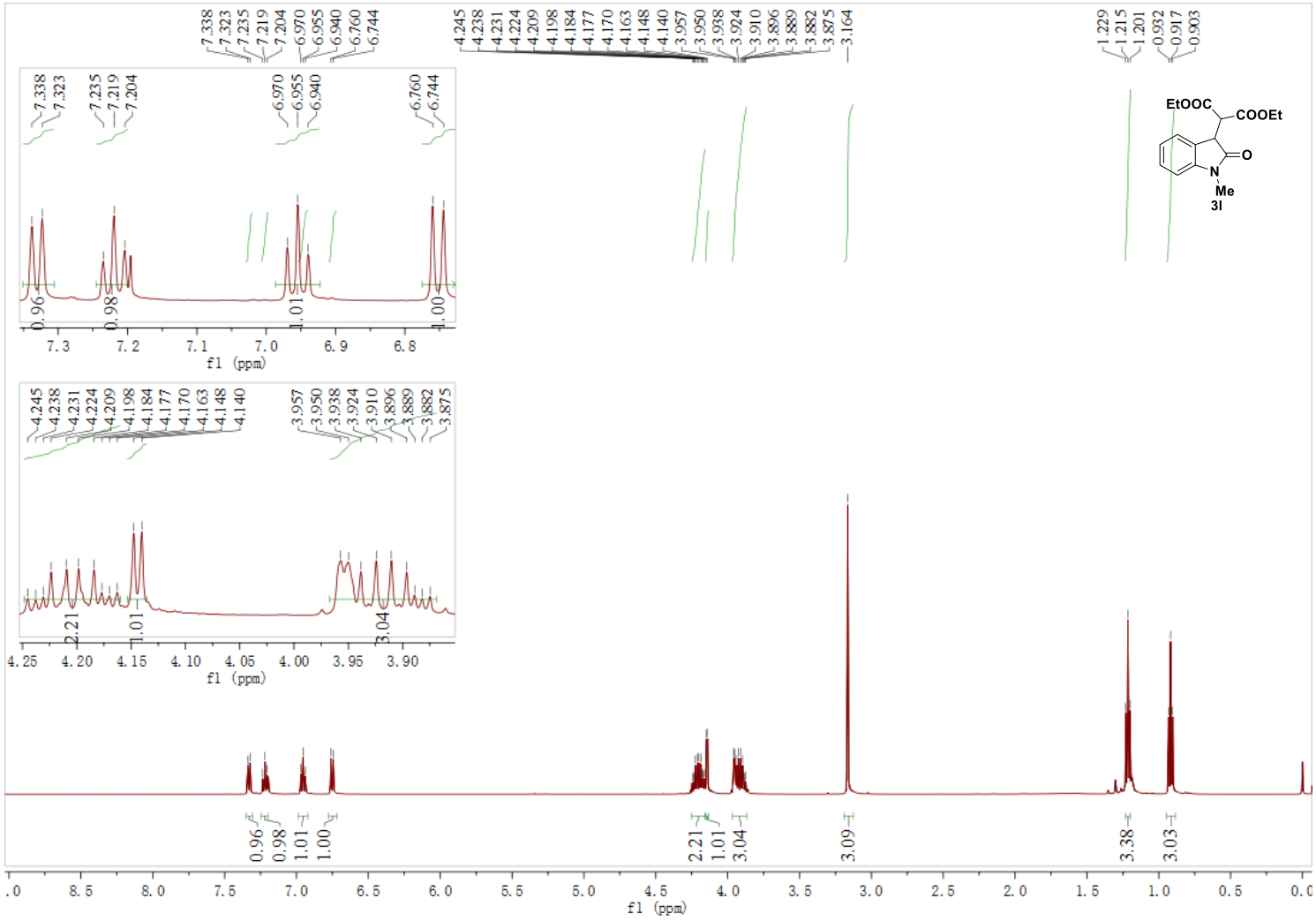

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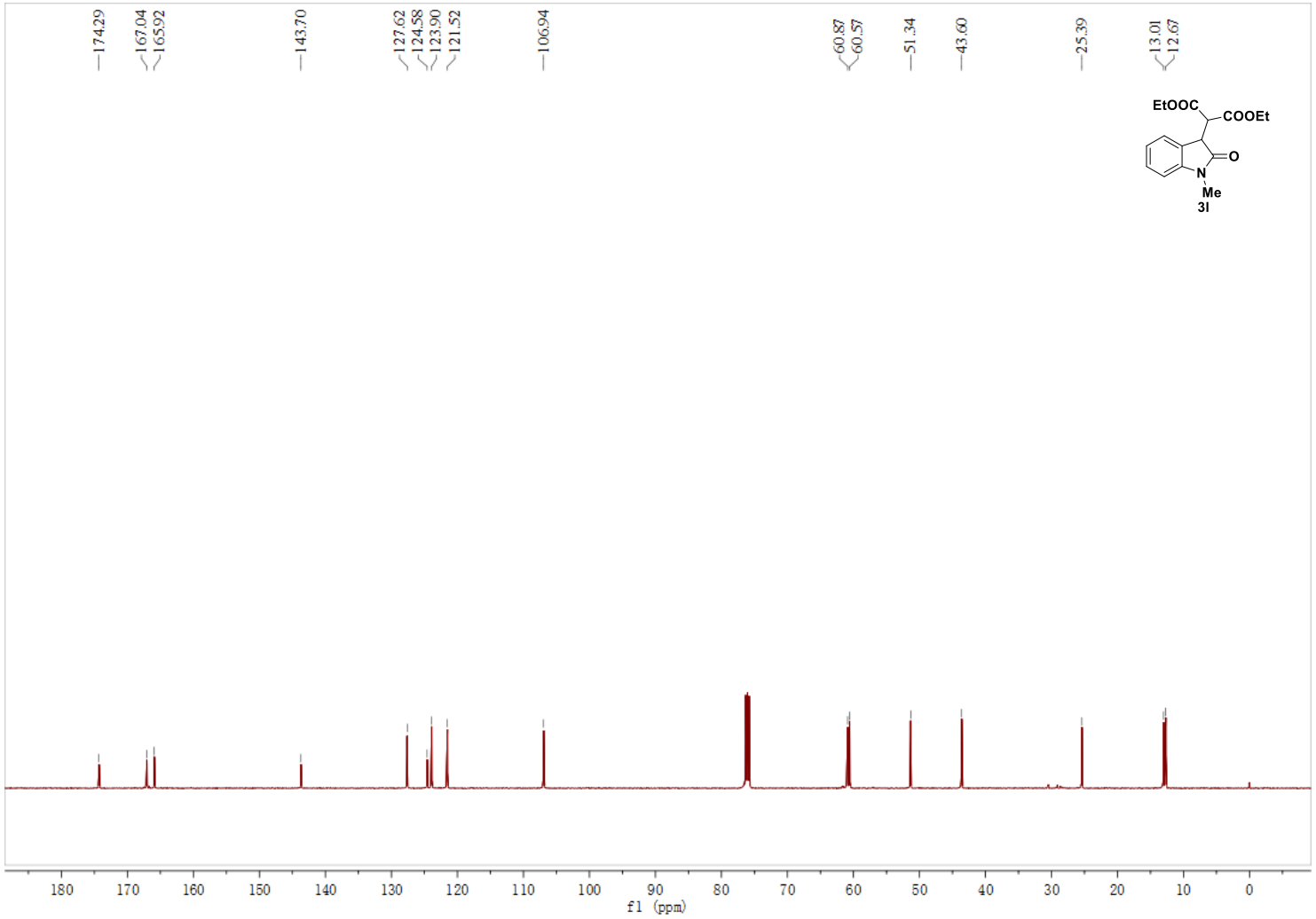

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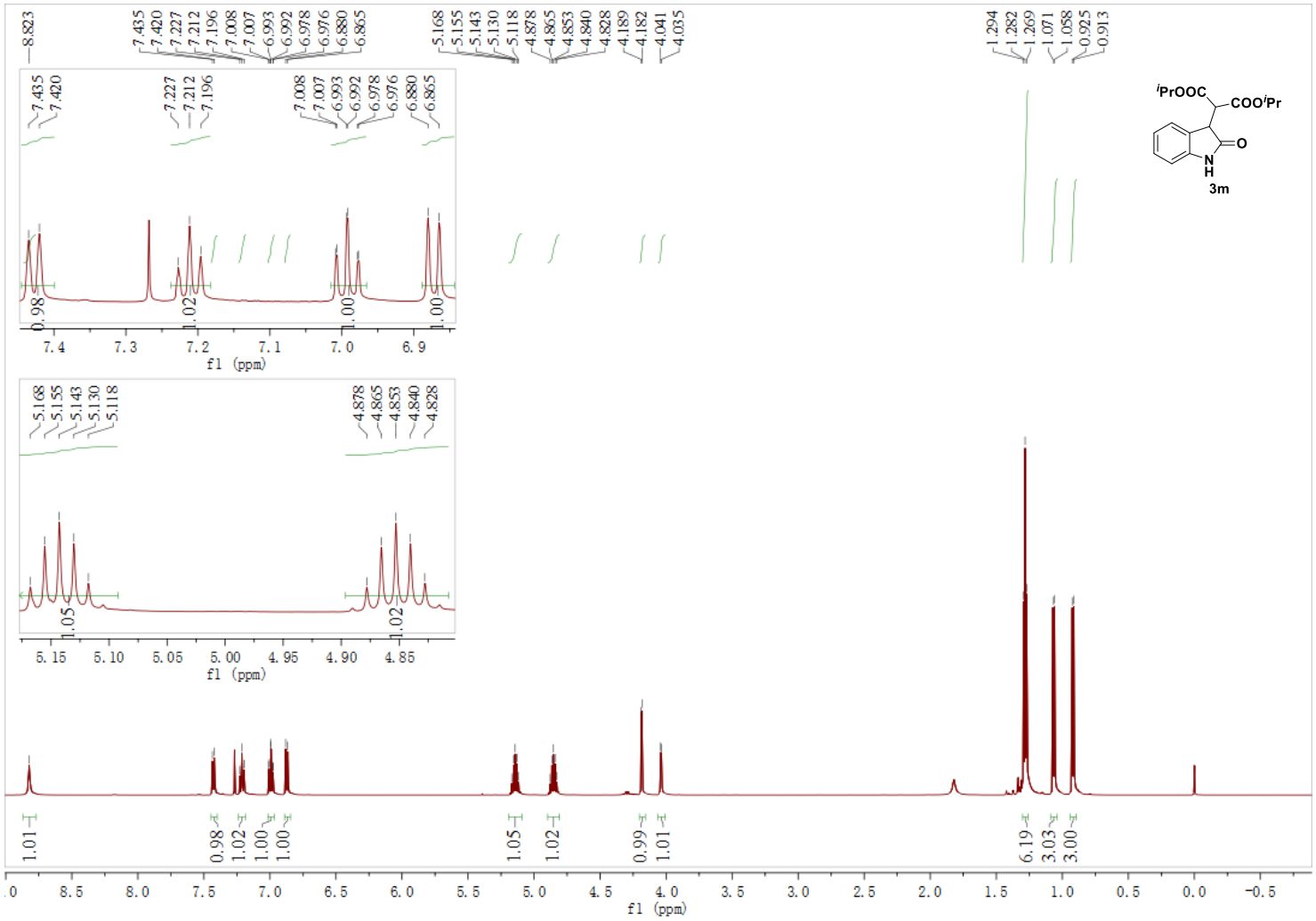

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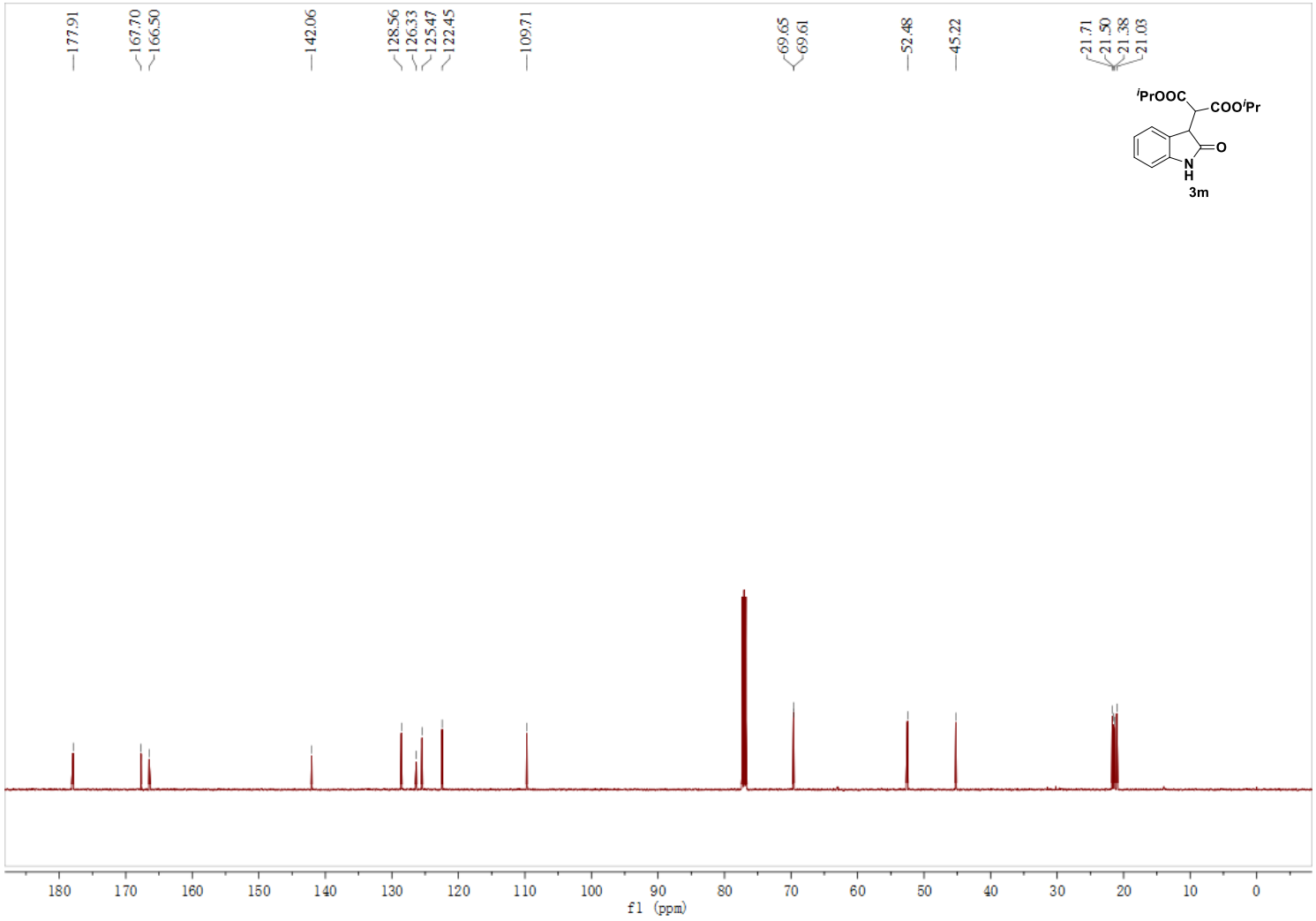

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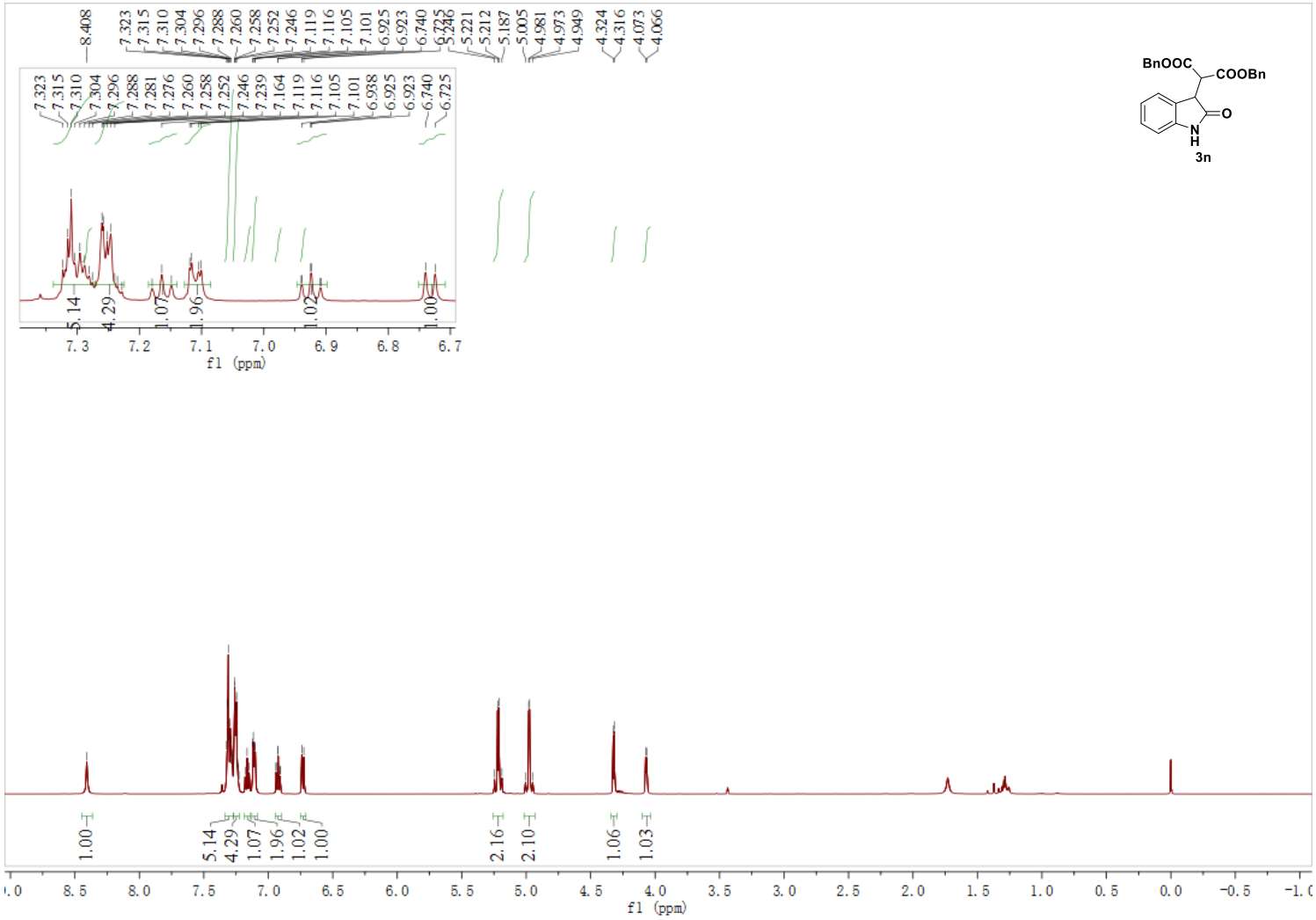

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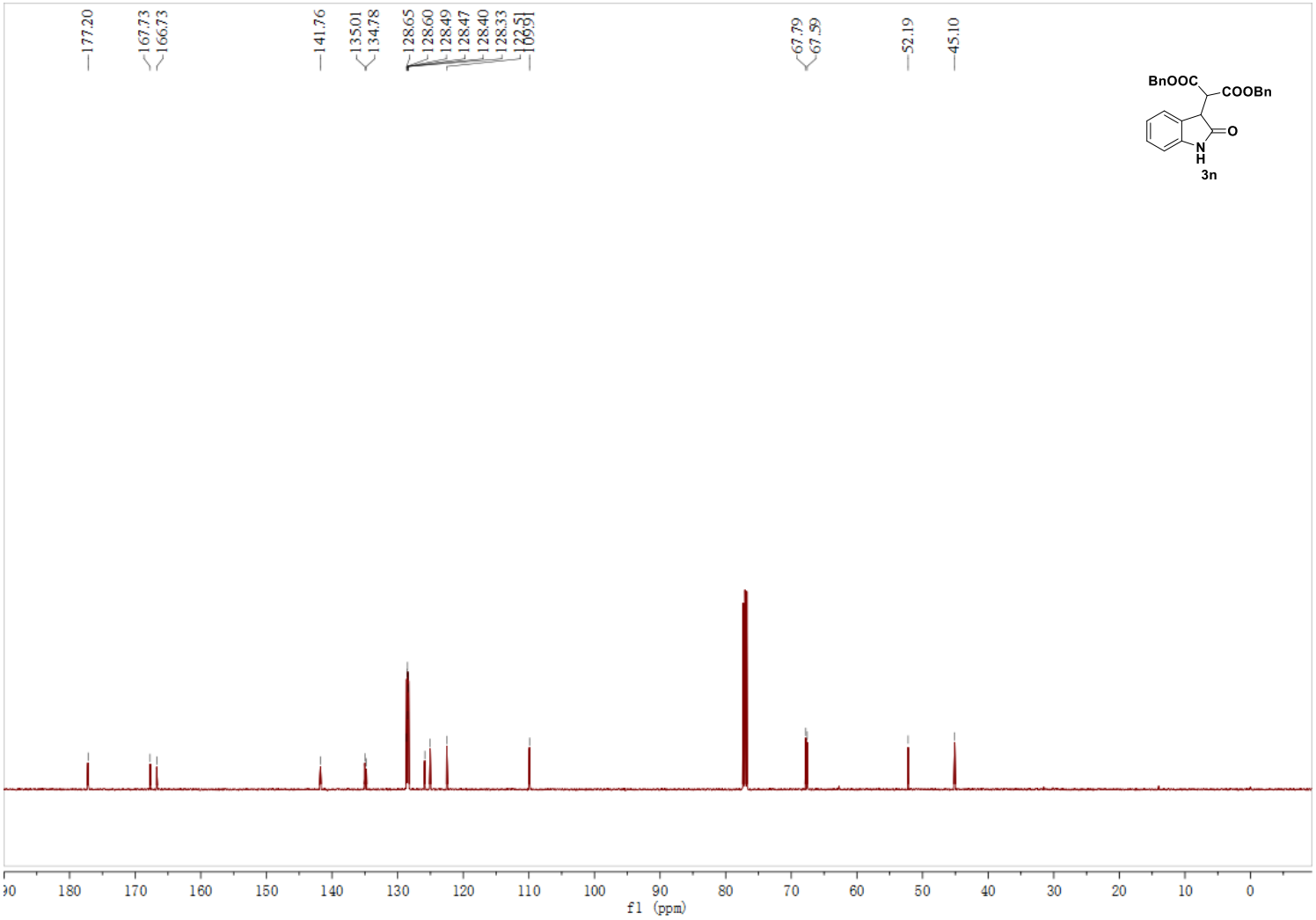

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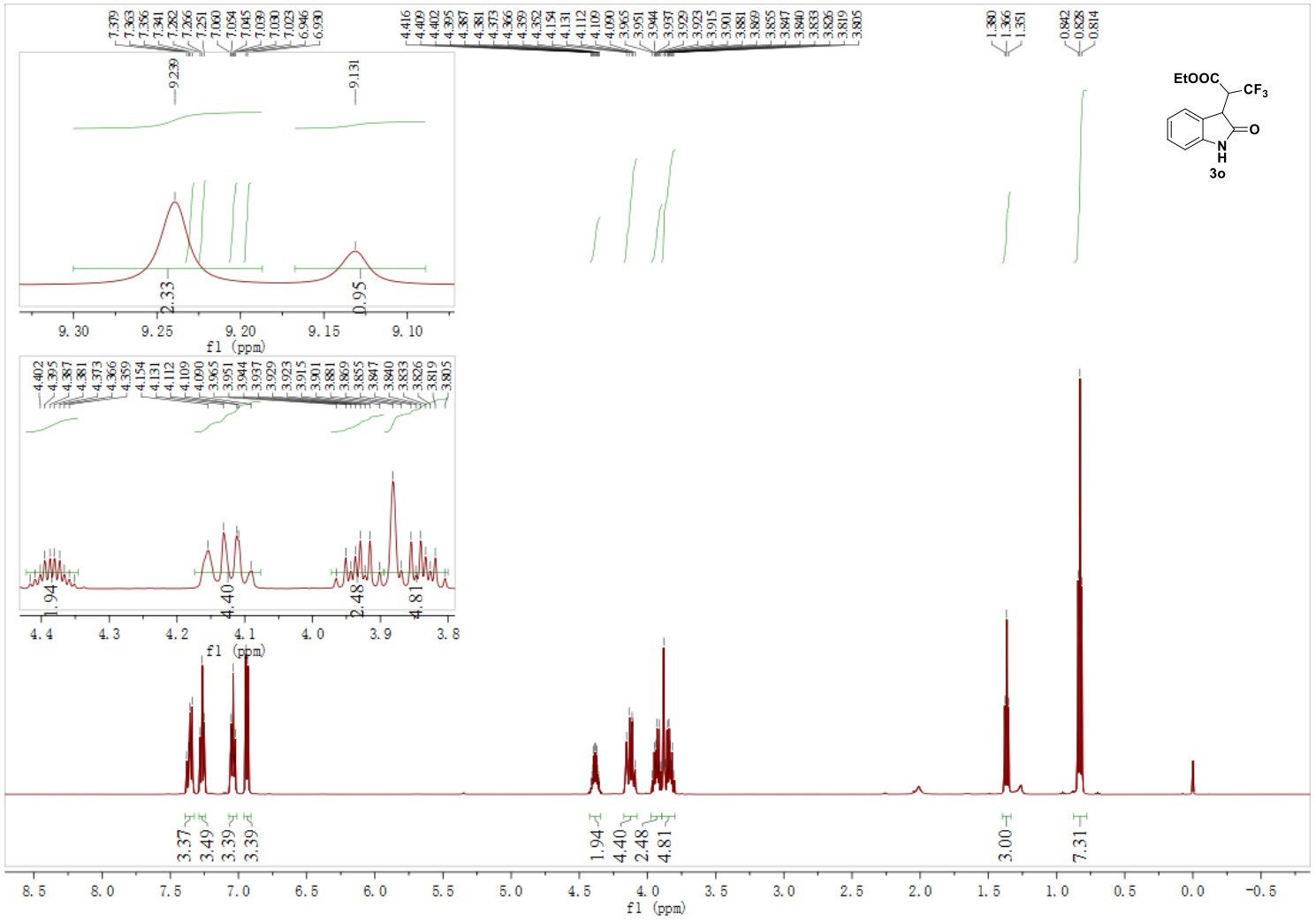

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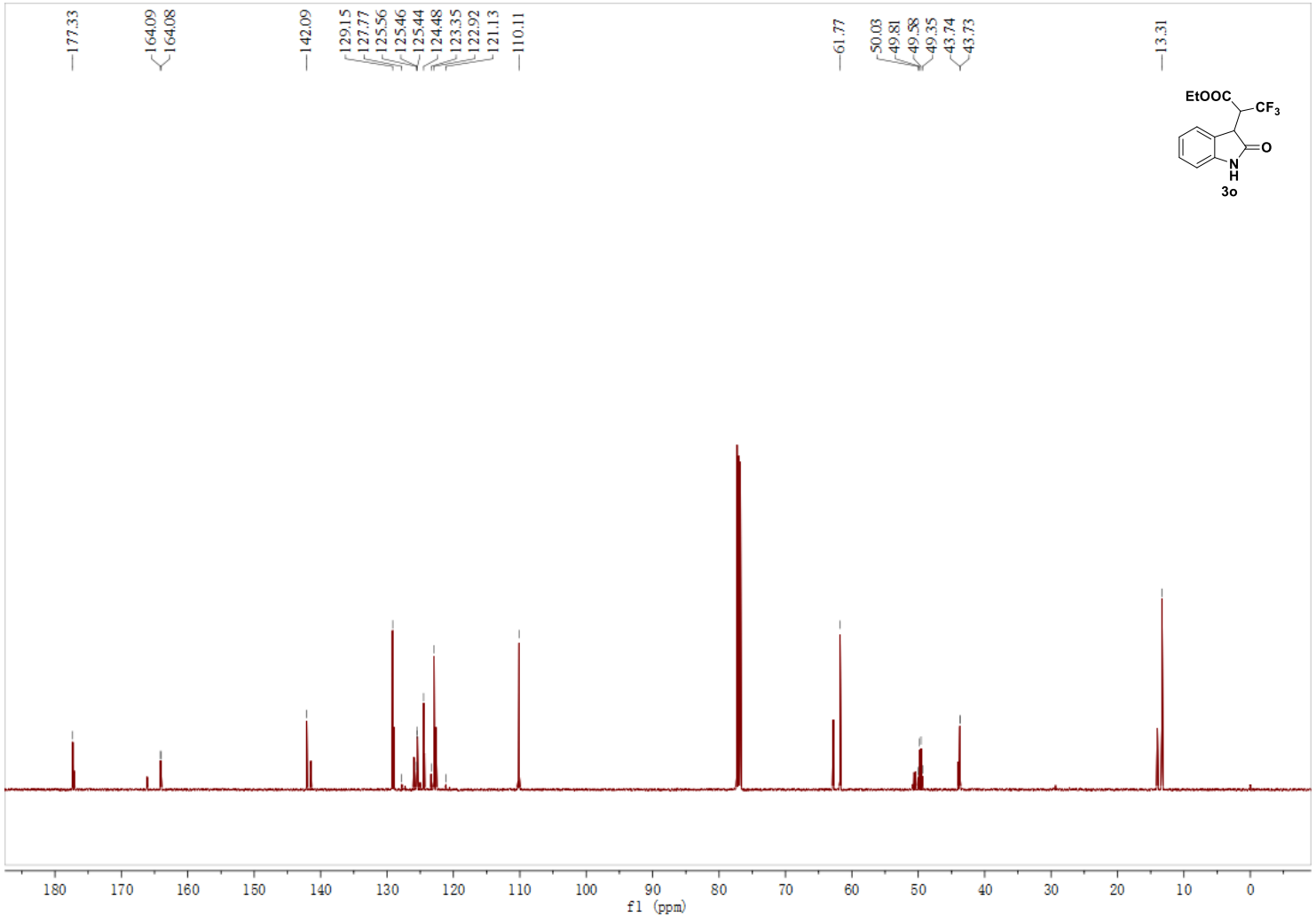

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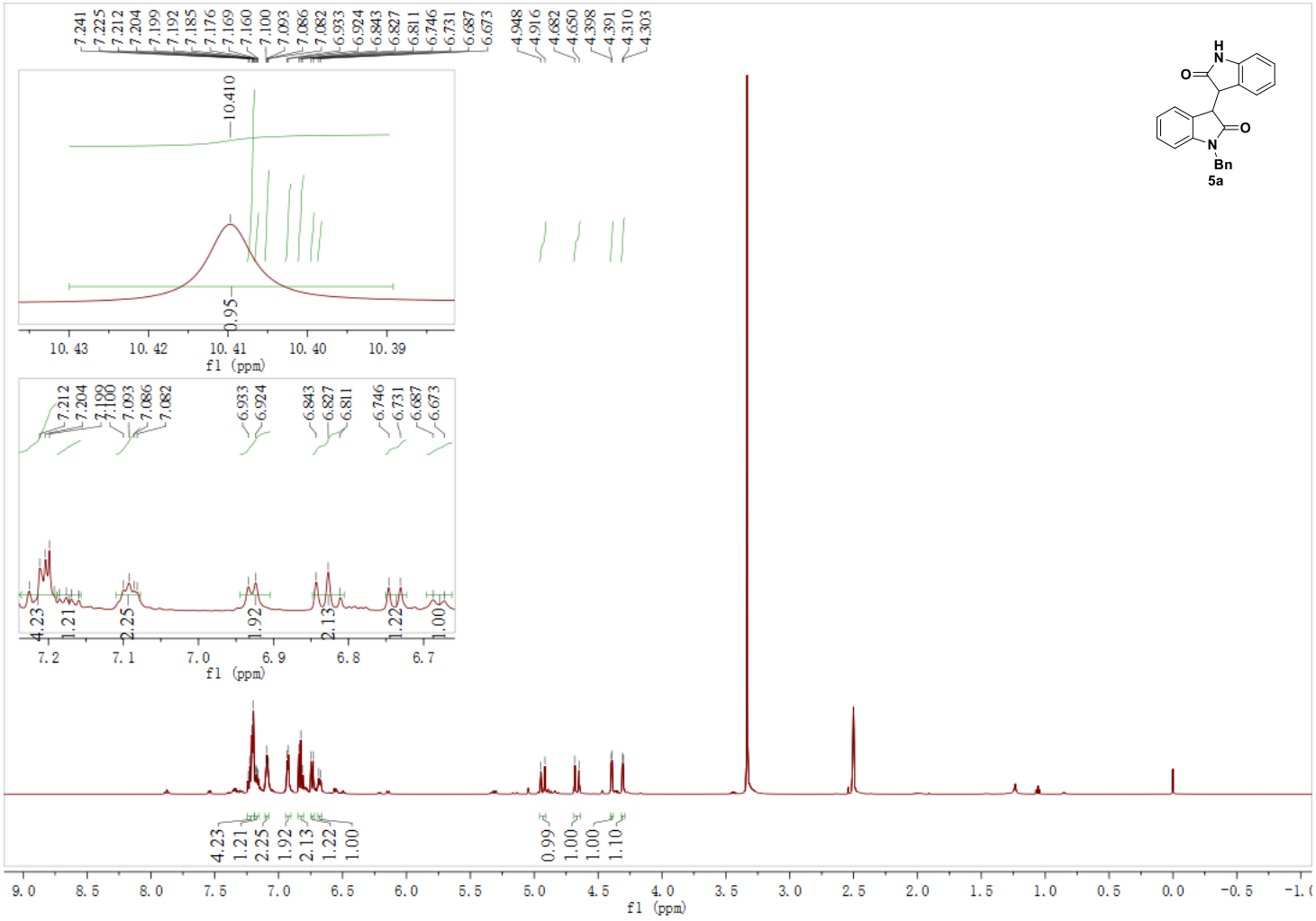

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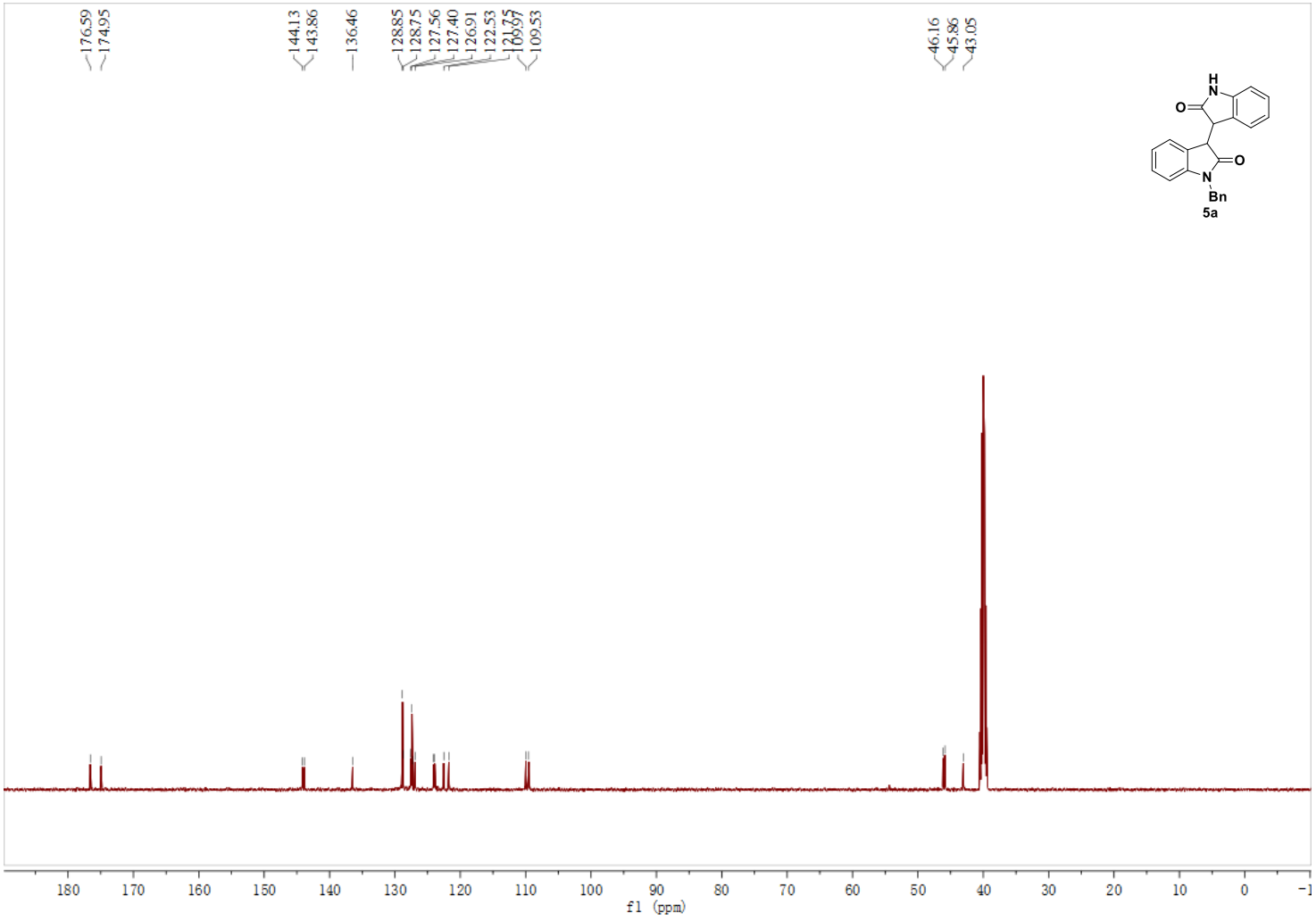

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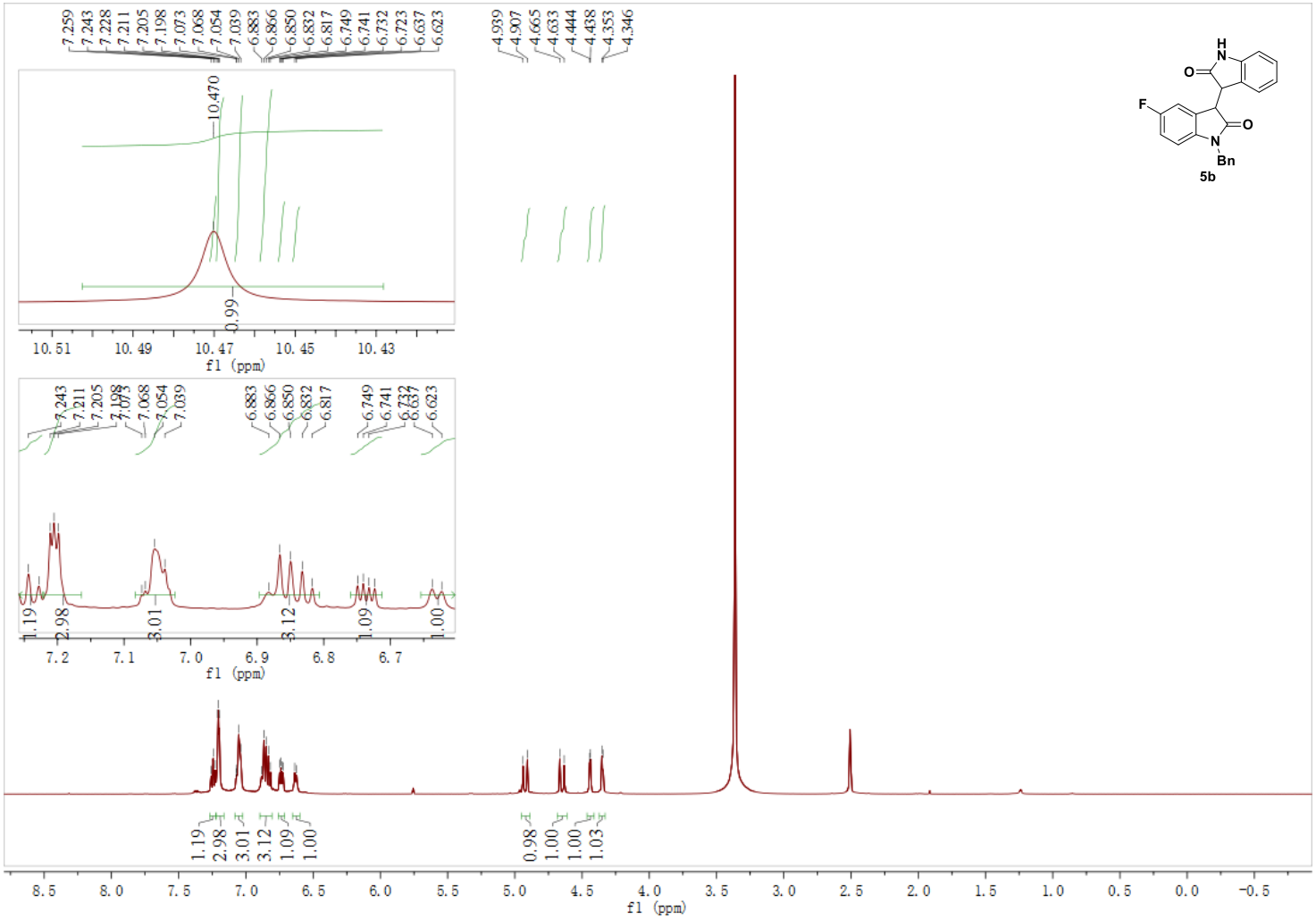

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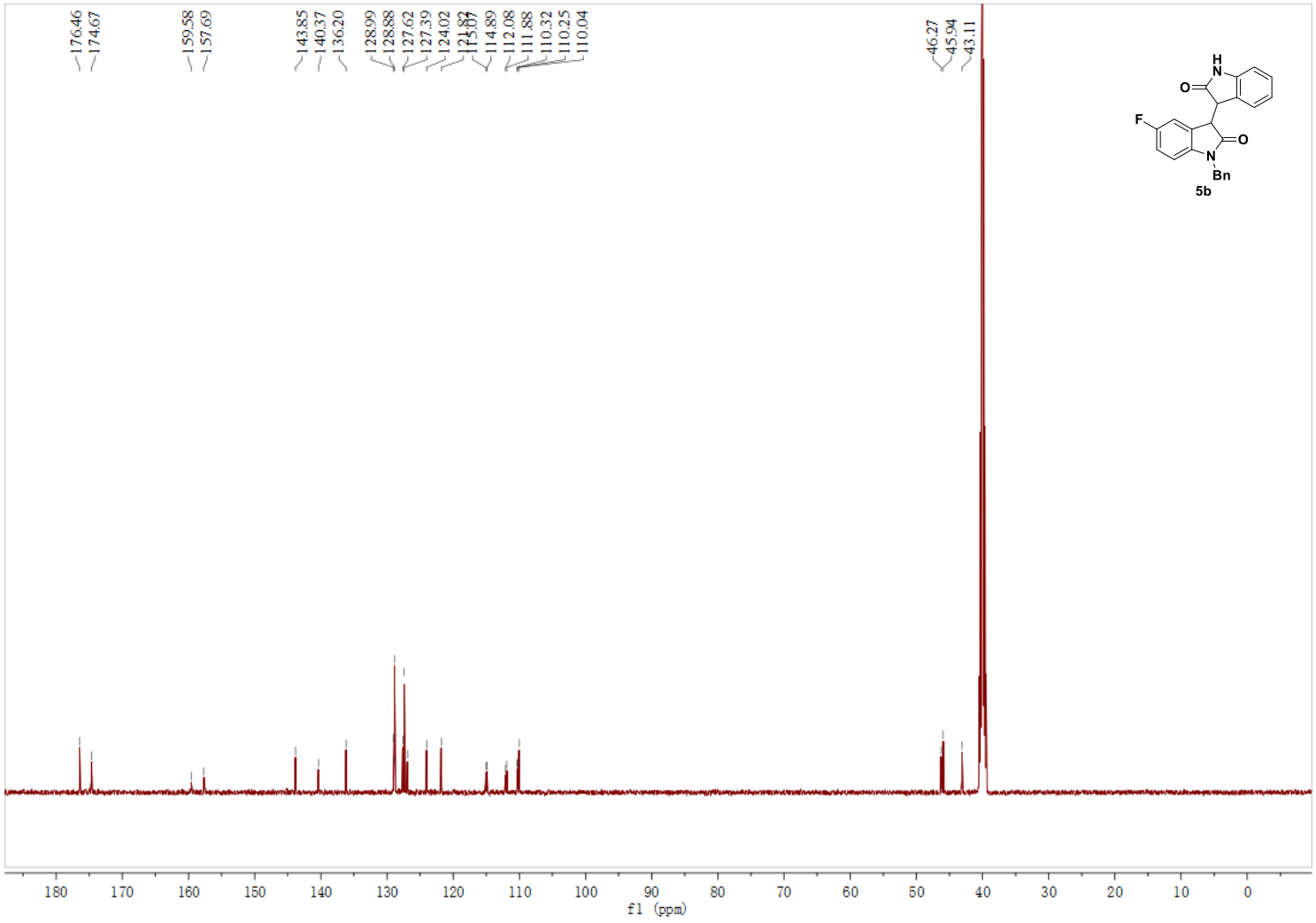

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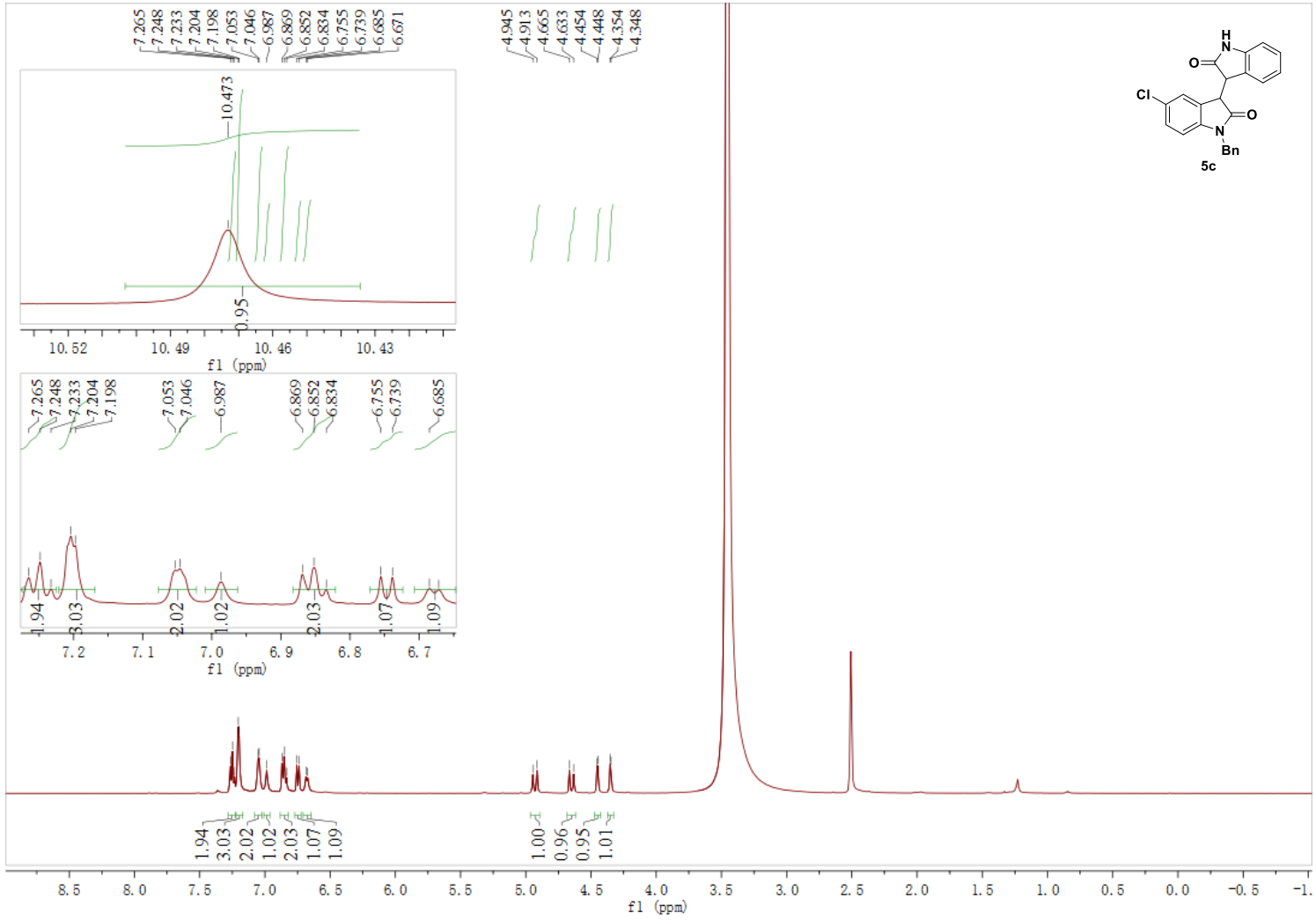

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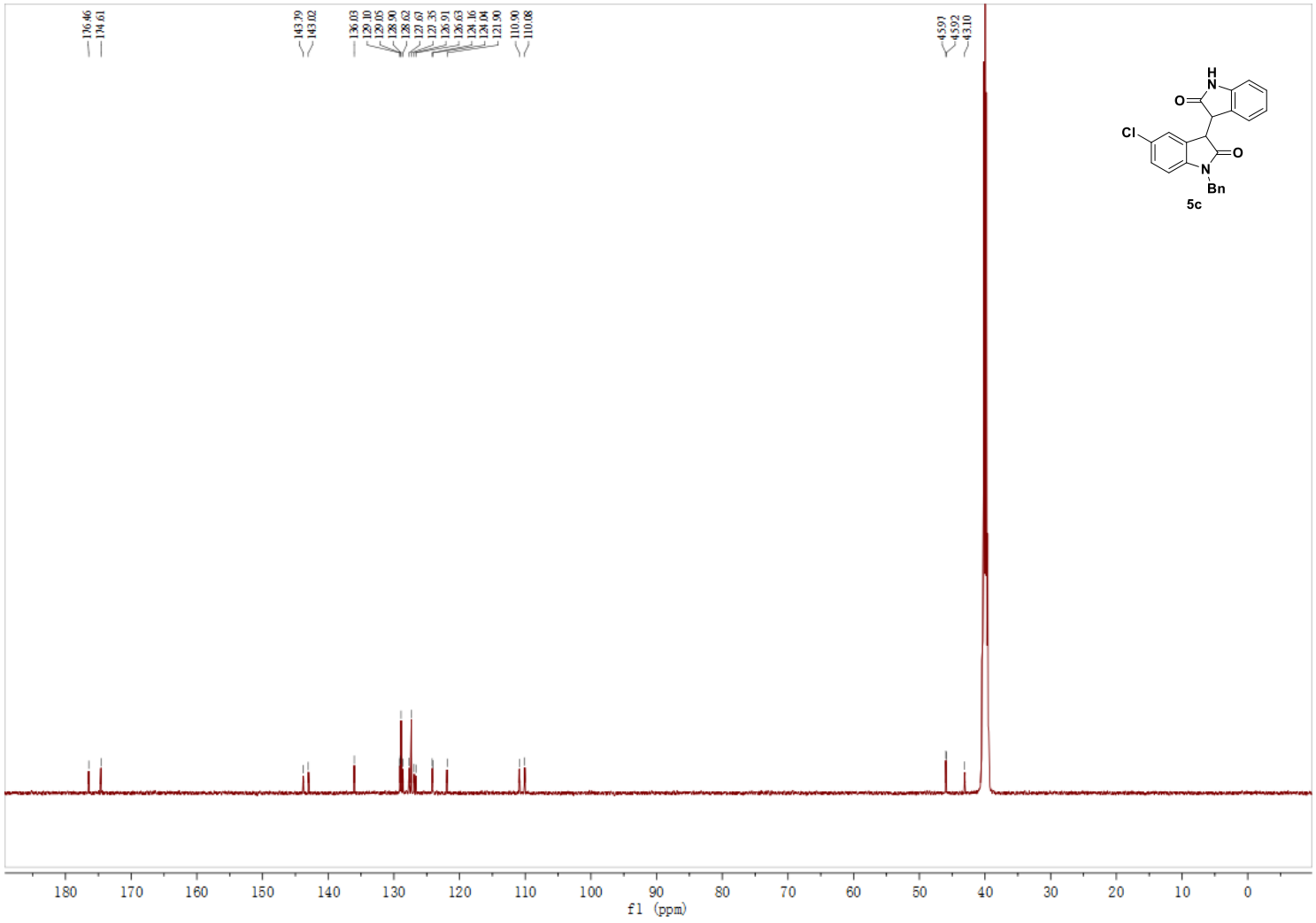

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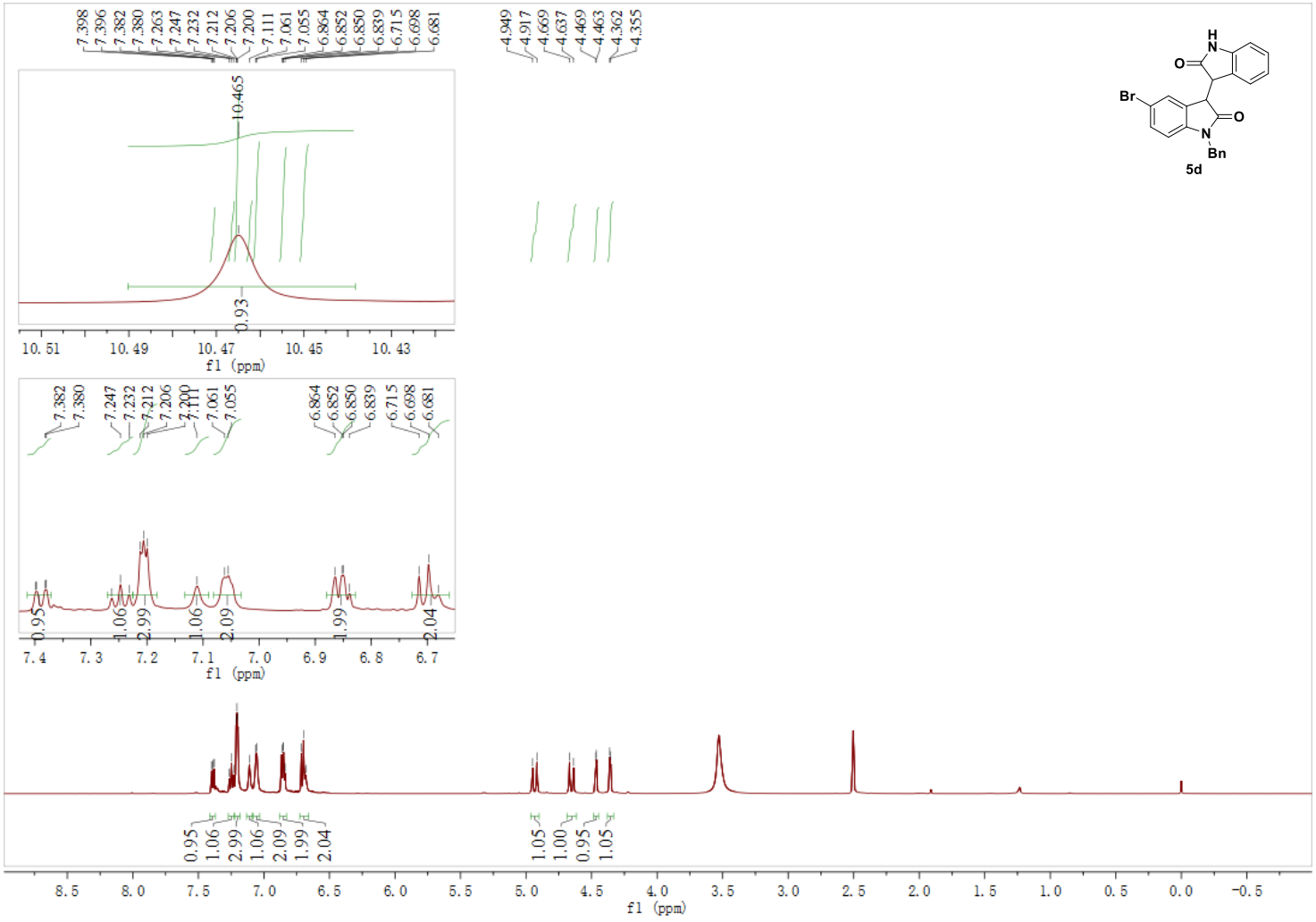

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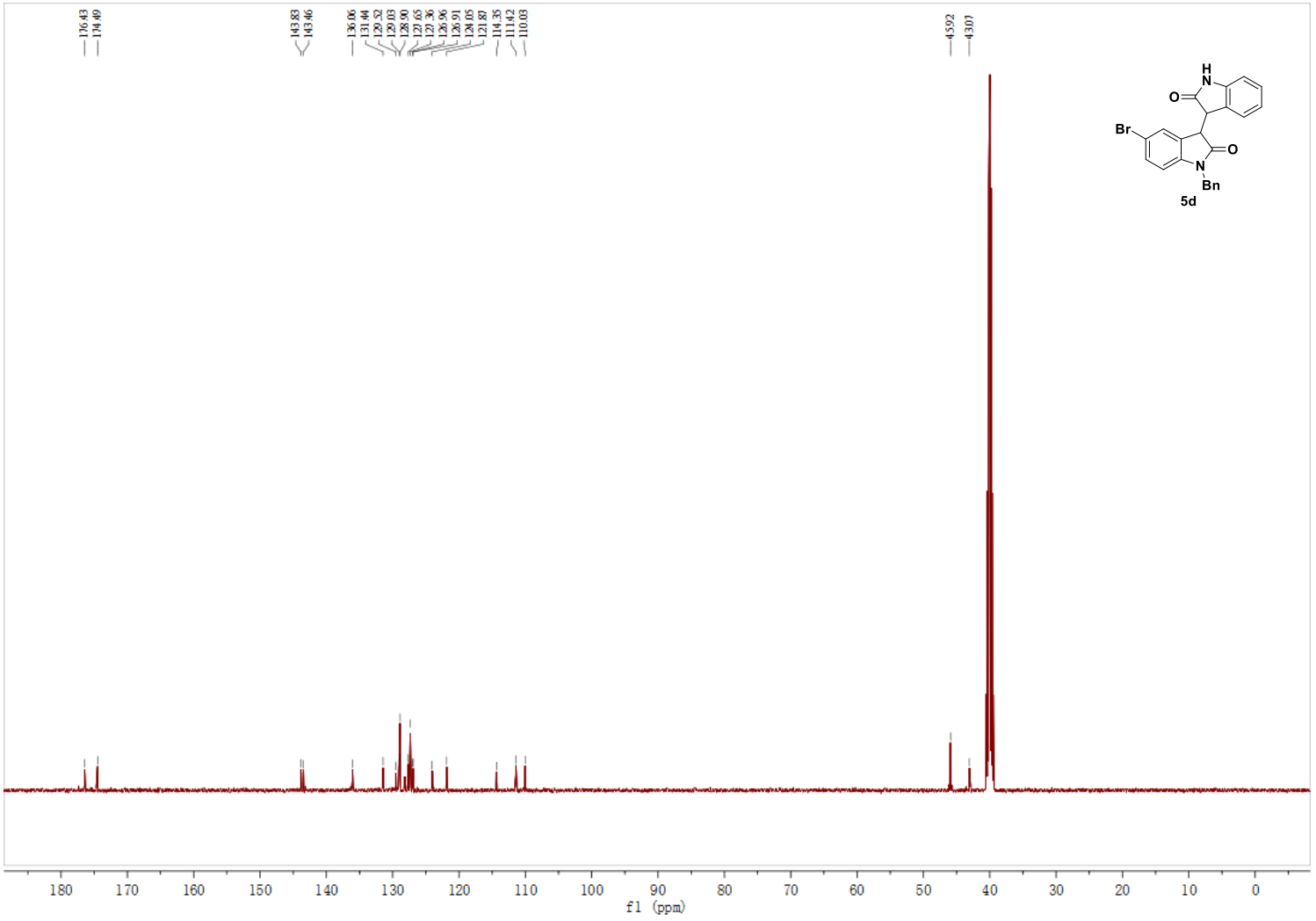

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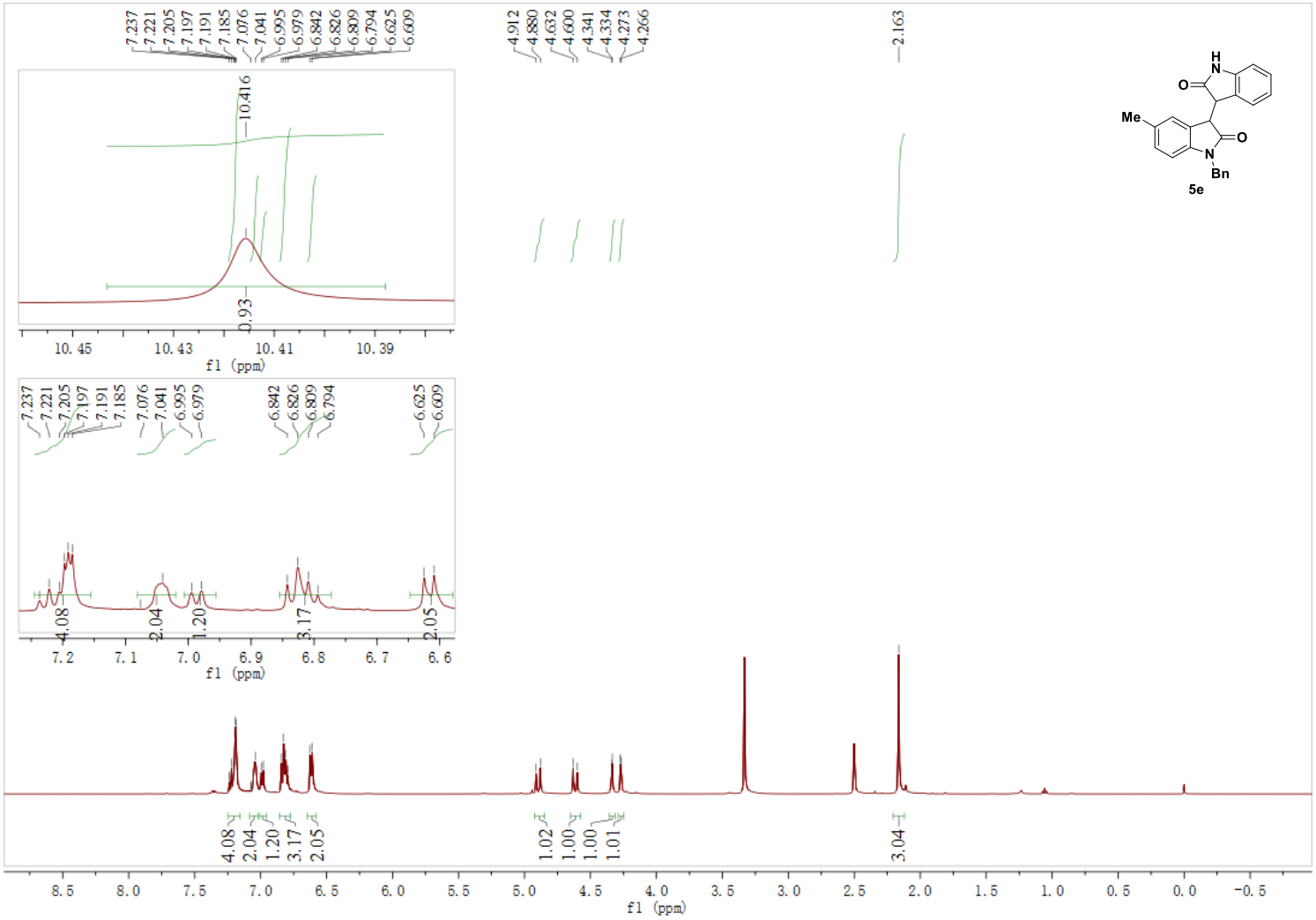
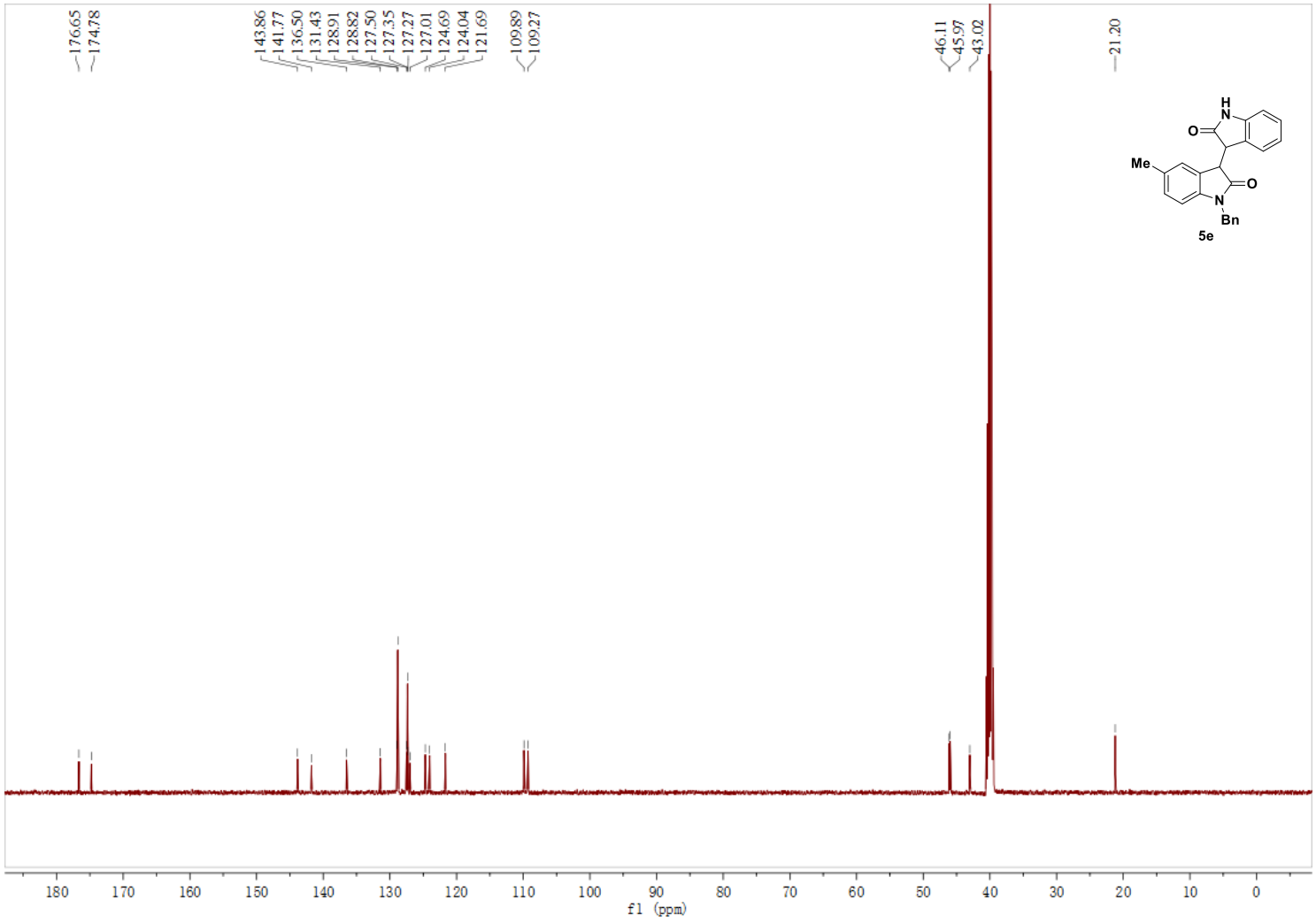
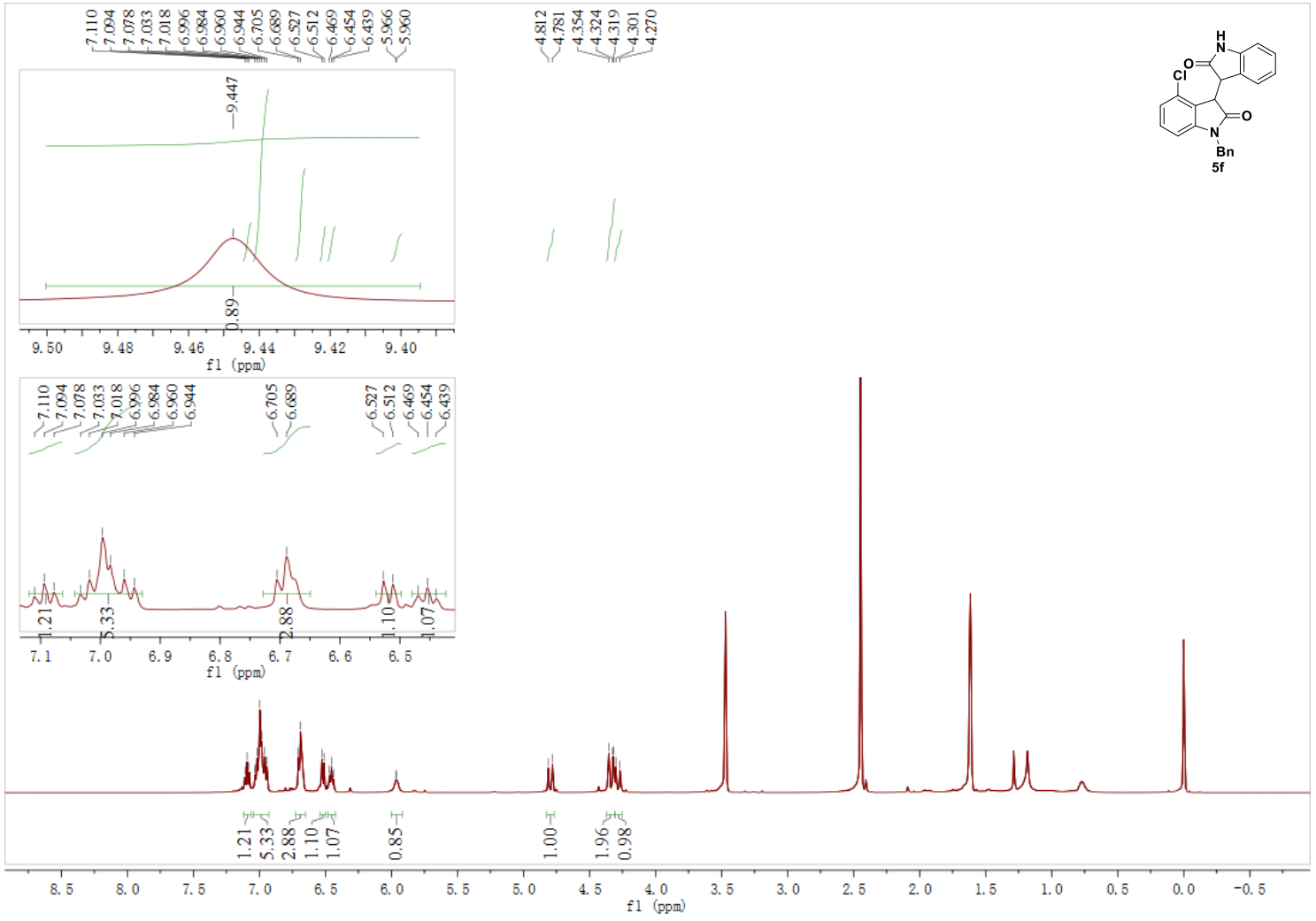


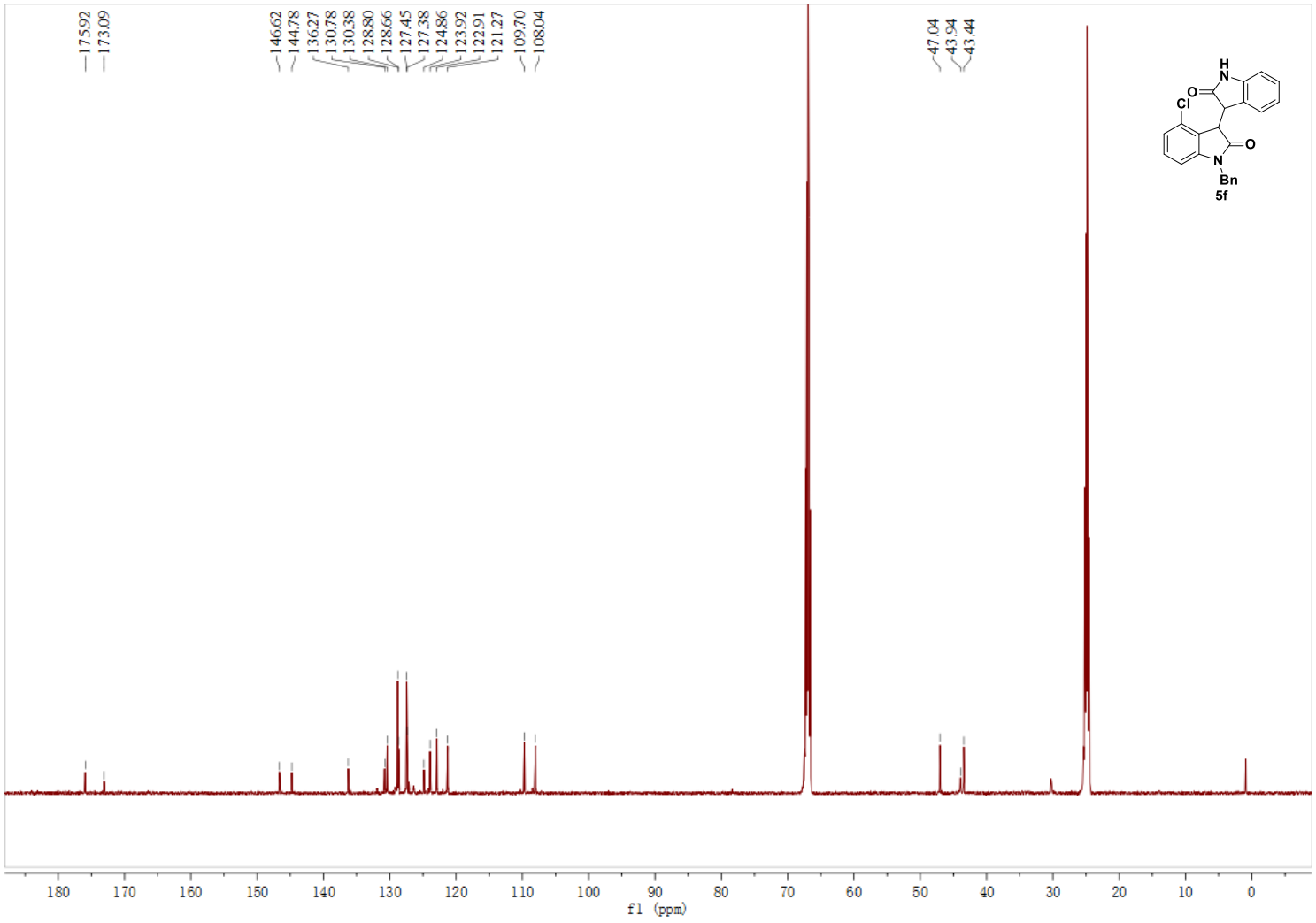
125 MHz, DMSO-d_6, ^{13}C NMR



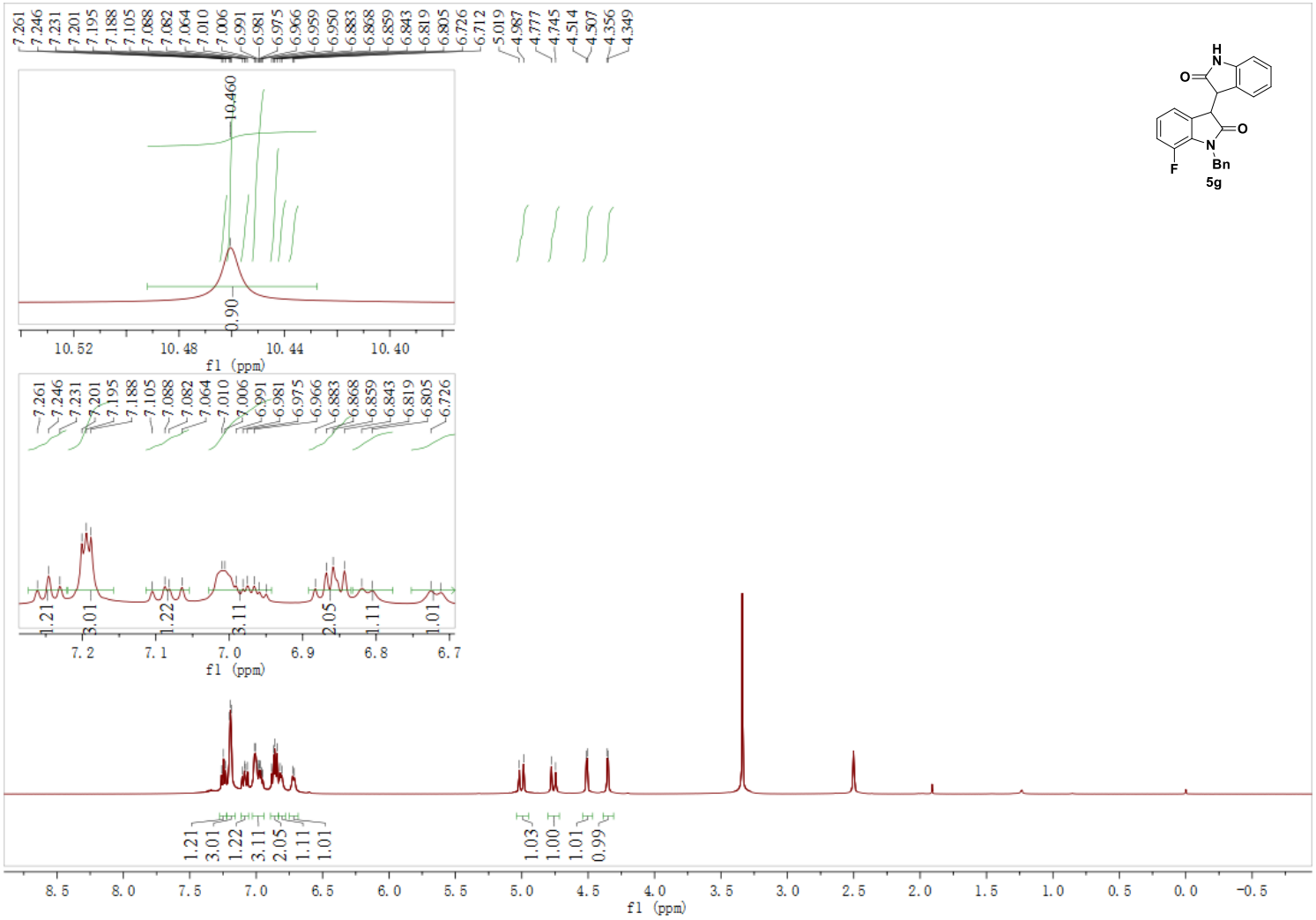
500 MHz, Tetrahydrofuran-d_8, ^{1}H NMR



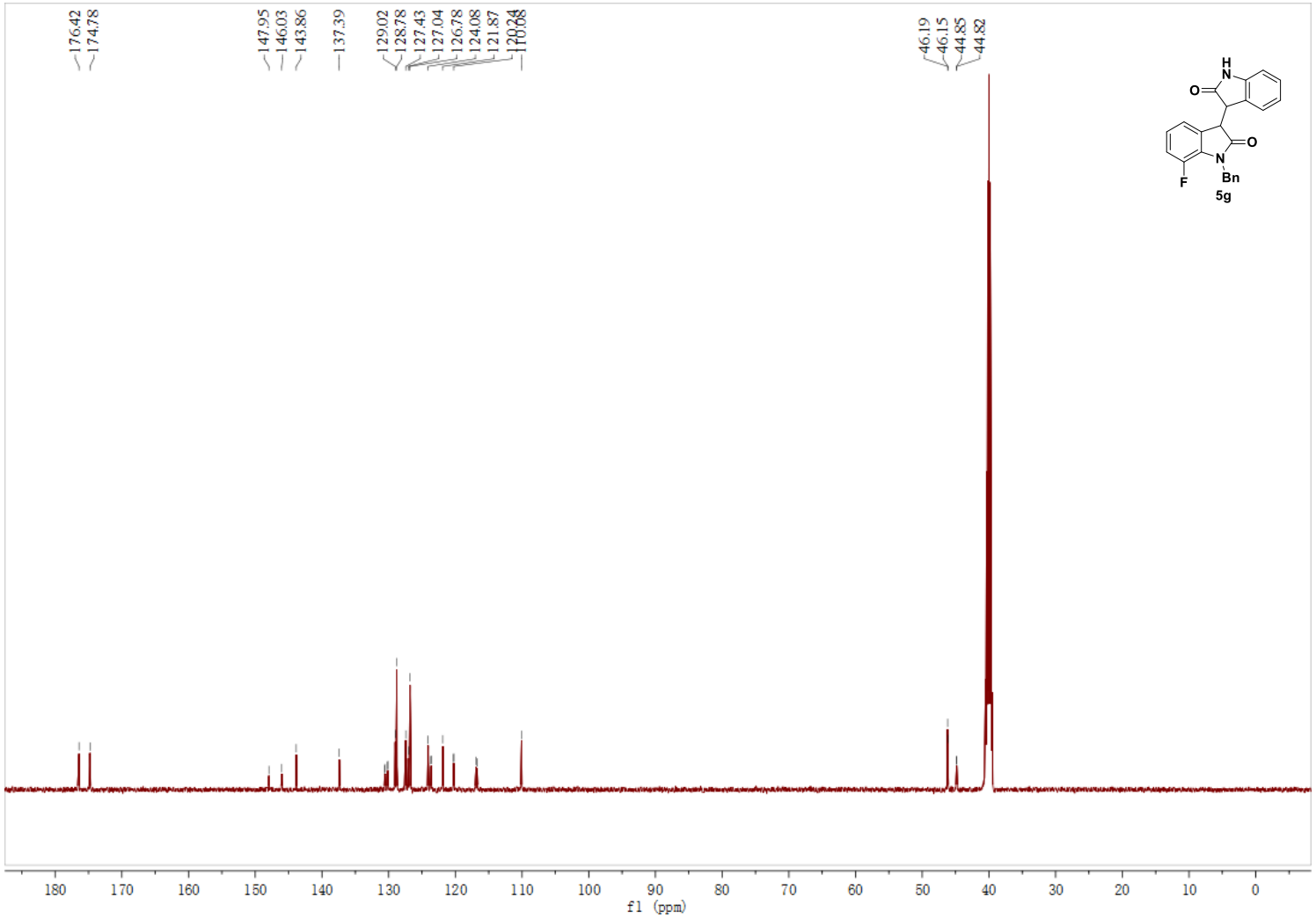
125 MHz, Tetrahydrofuran-d_8, ^{13}C NMR



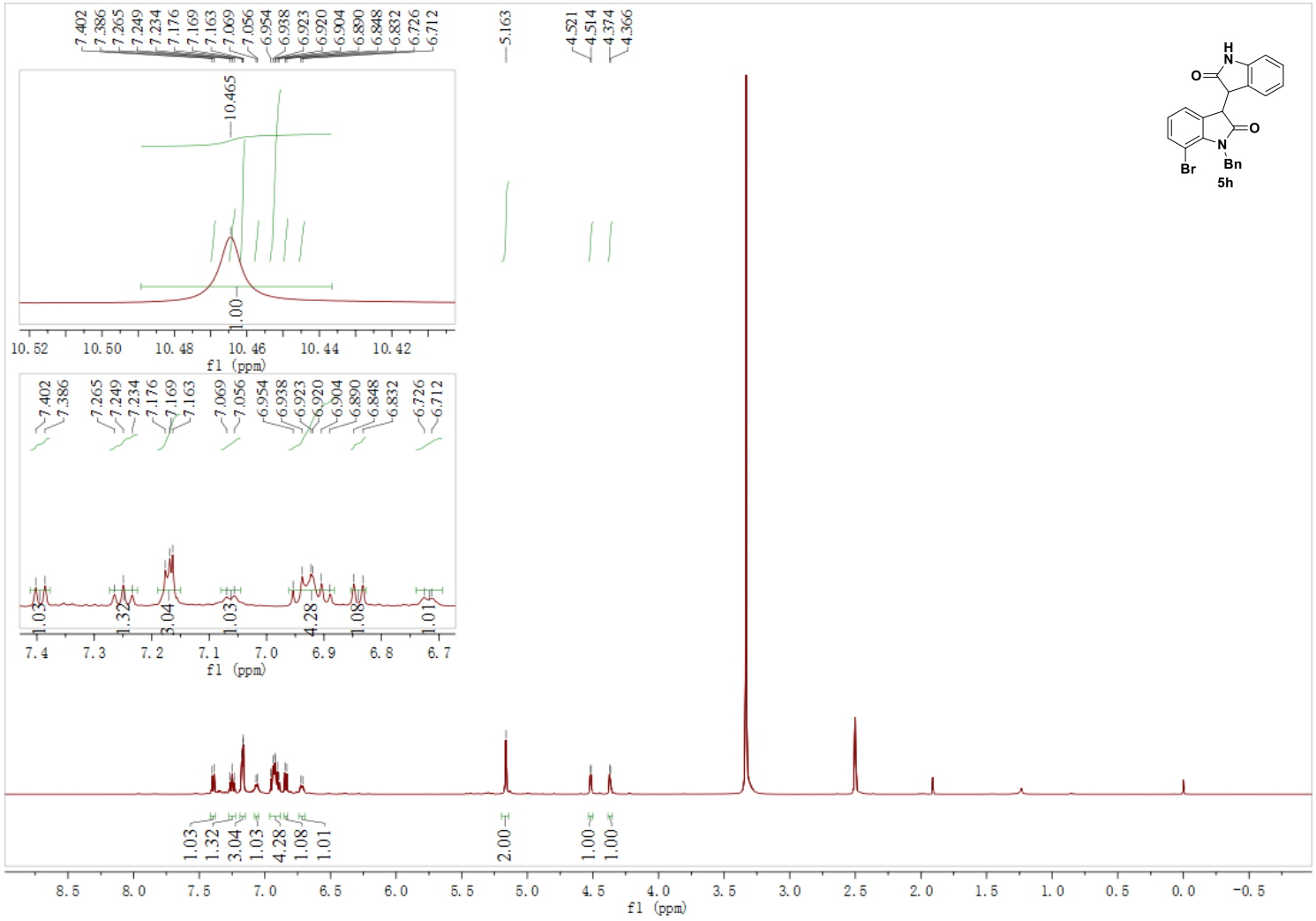
500 MHz, DMSO-d_6, ^{1}H NMR



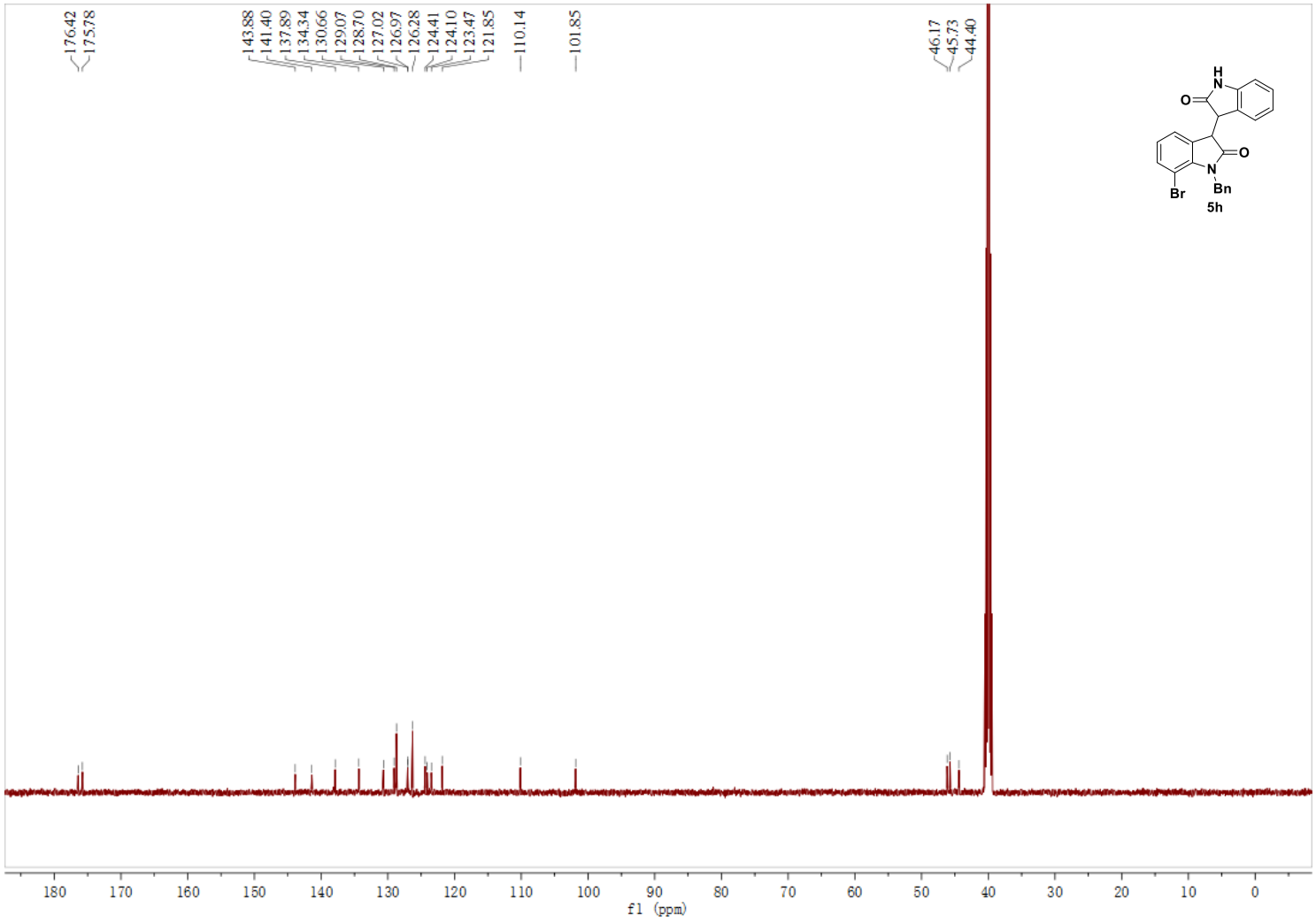
125 MHz, DMSO-d_6, ^{13}C NMR



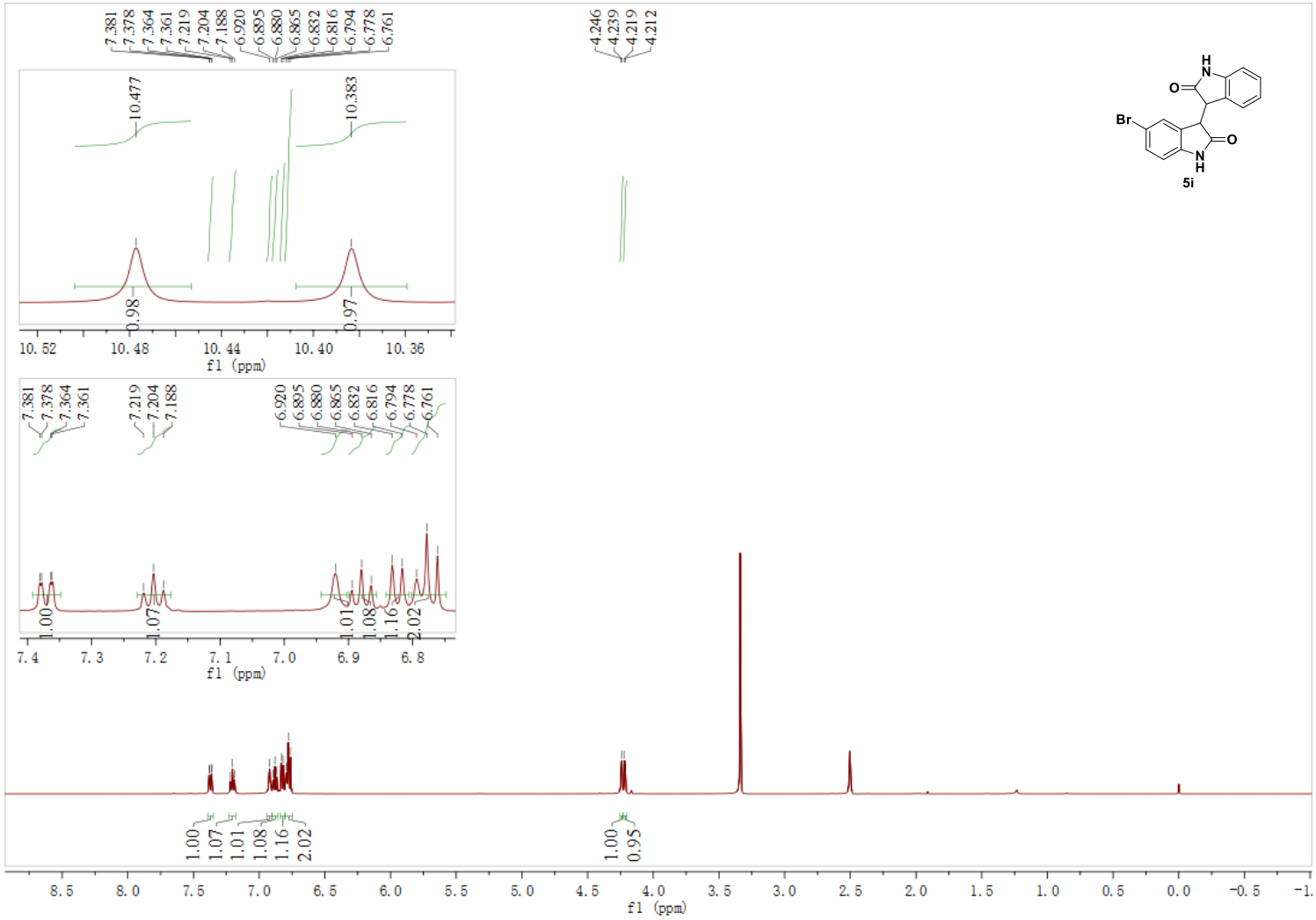
500 MHz, DMSO-d_6, ^{1}H NMR



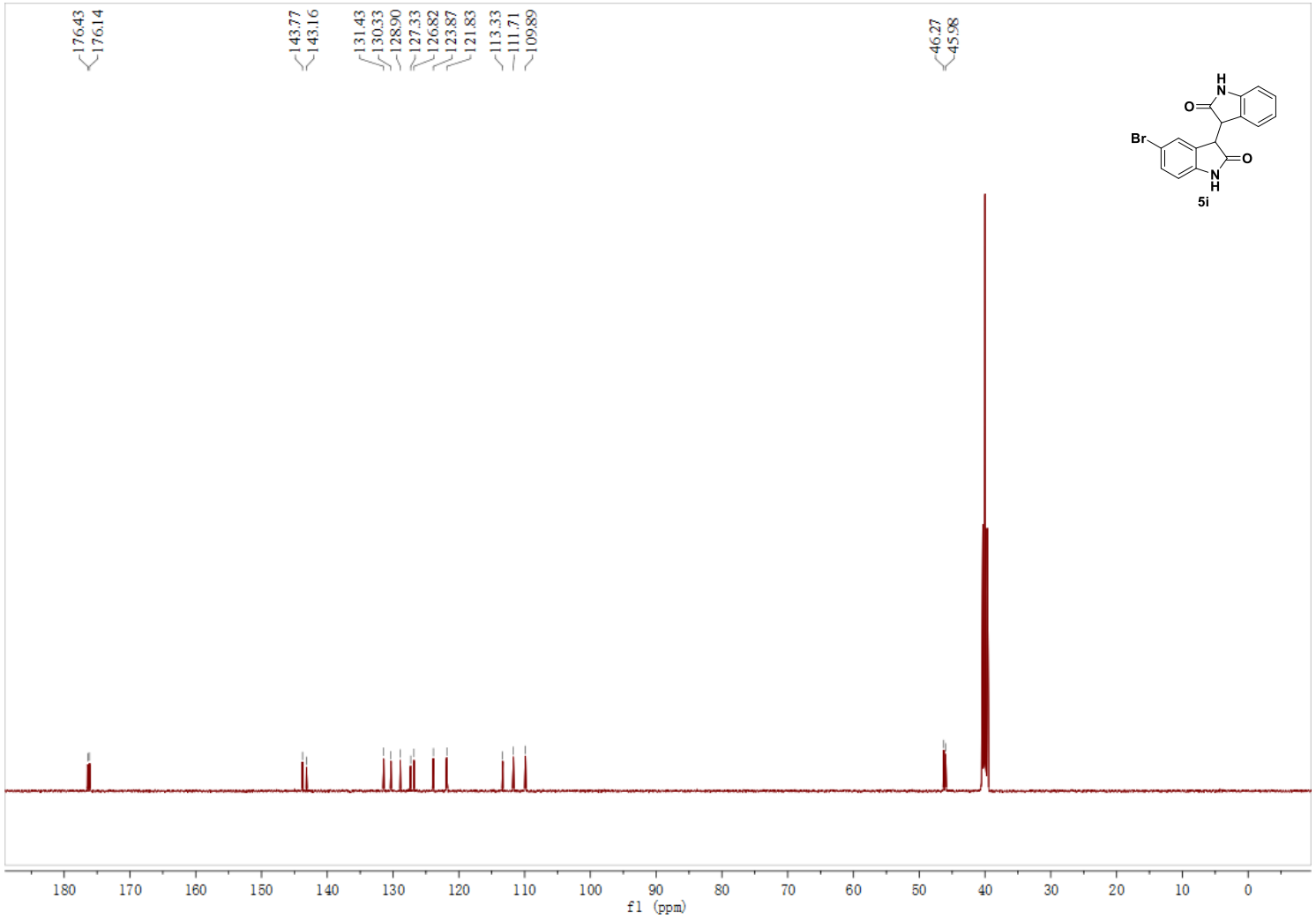
125 MHz, DMSO-d_6, ^{13}C NMR



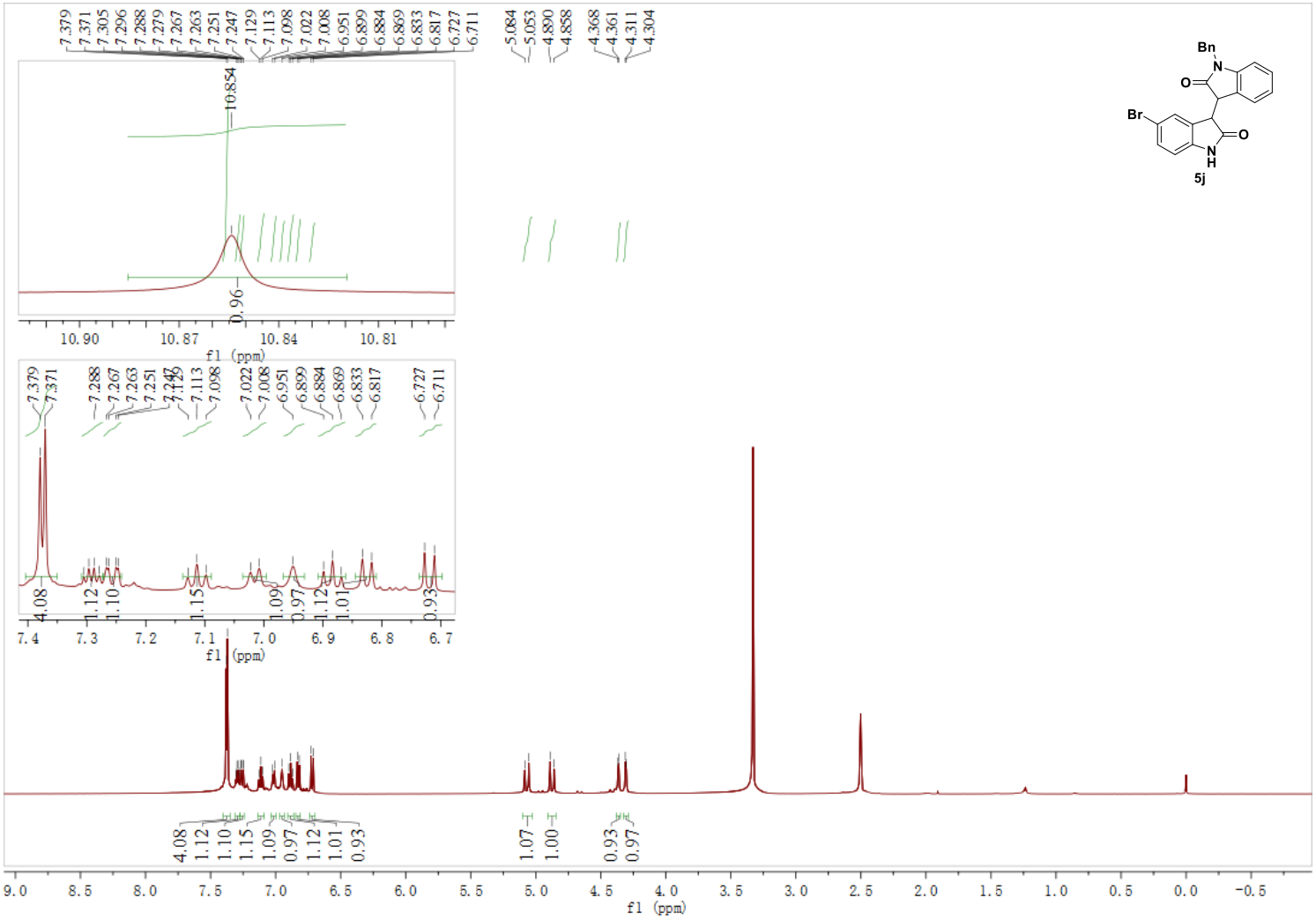
500 MHz, DMSO-d_6, ^{1}H NMR



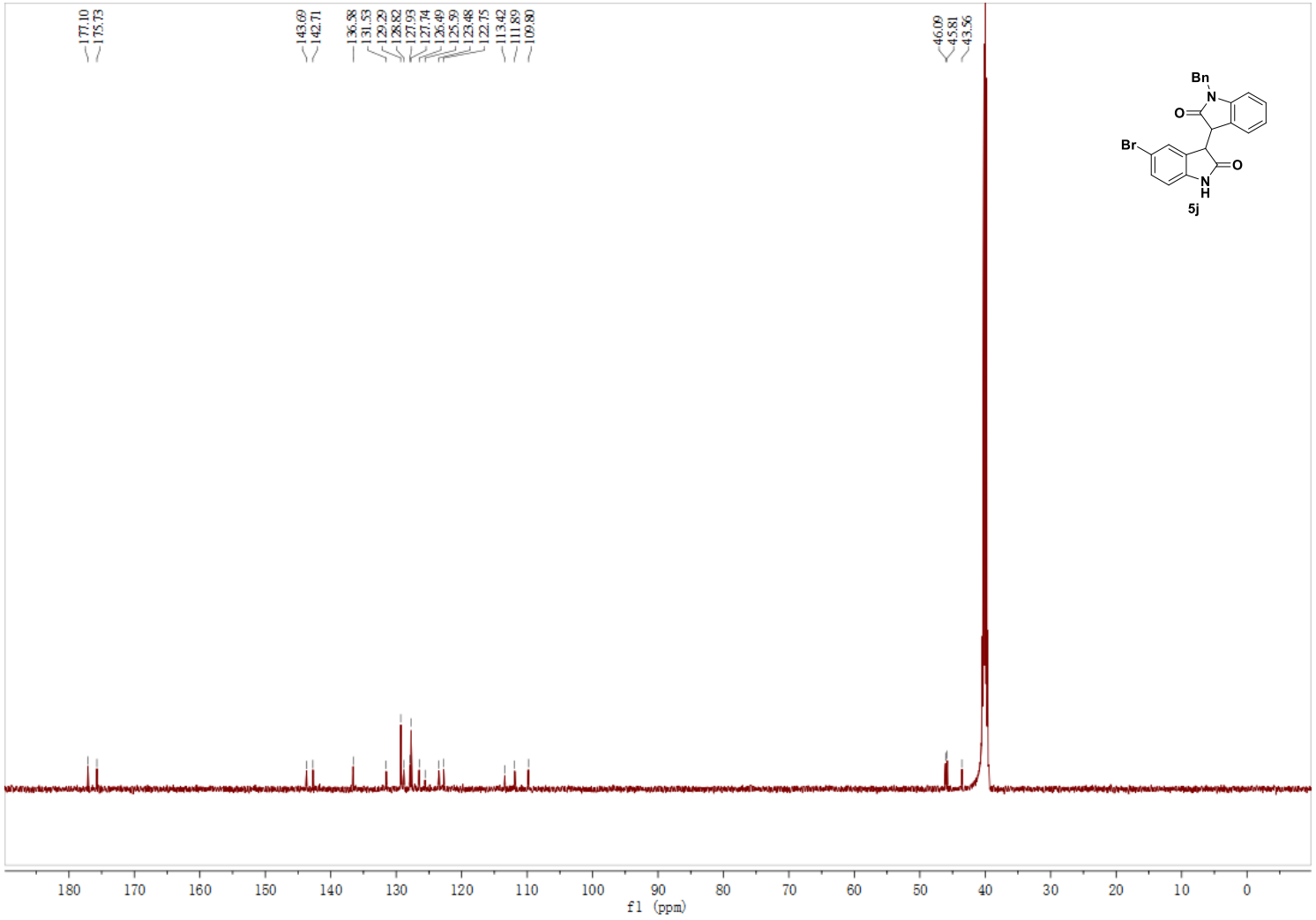
125 MHz, DMSO-d_6, ^{13}C NMR



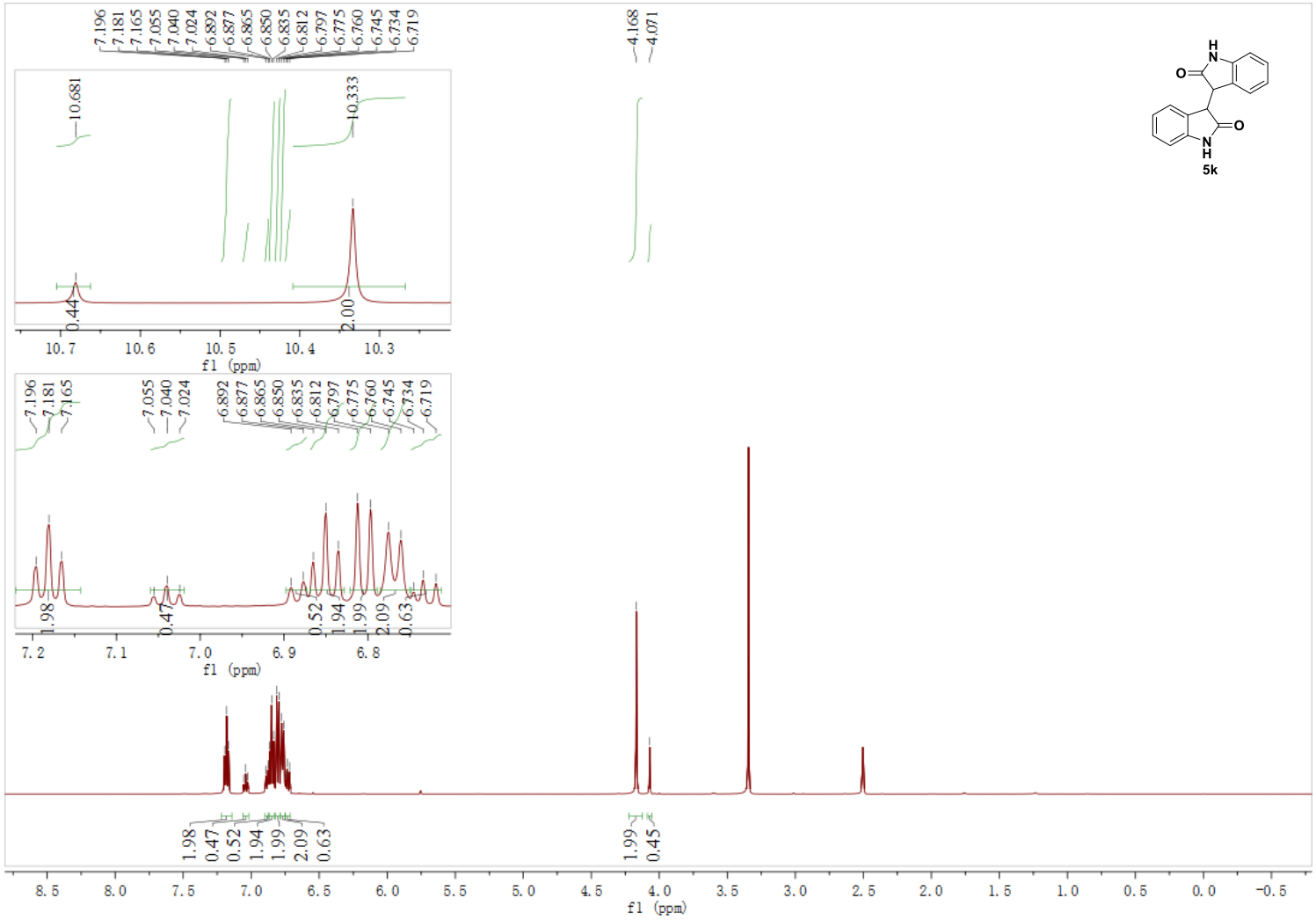
500 MHz, DMSO-d_6, ^{1}H NMR



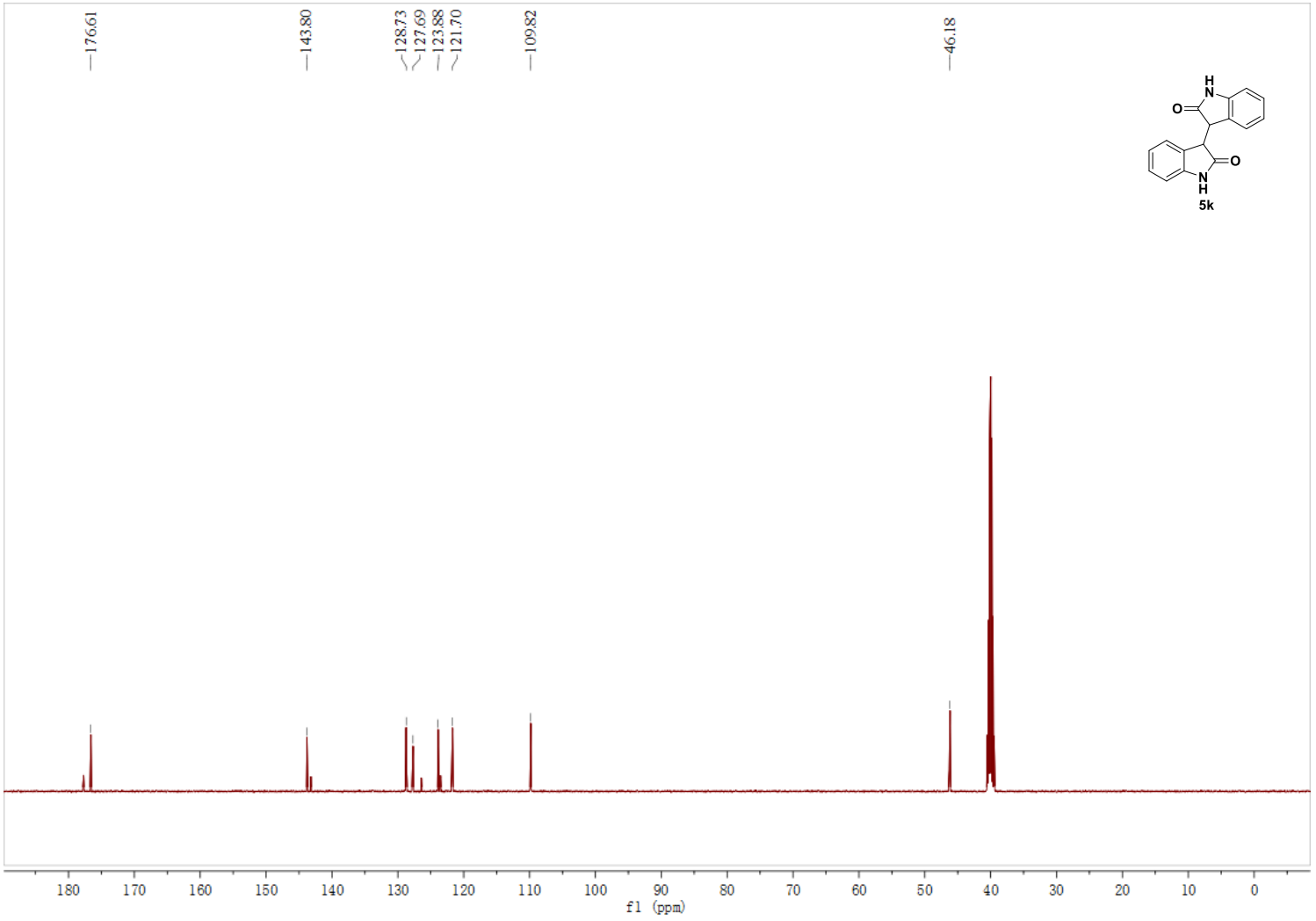
125 MHz, DMSO-d_6, ^{13}C NMR



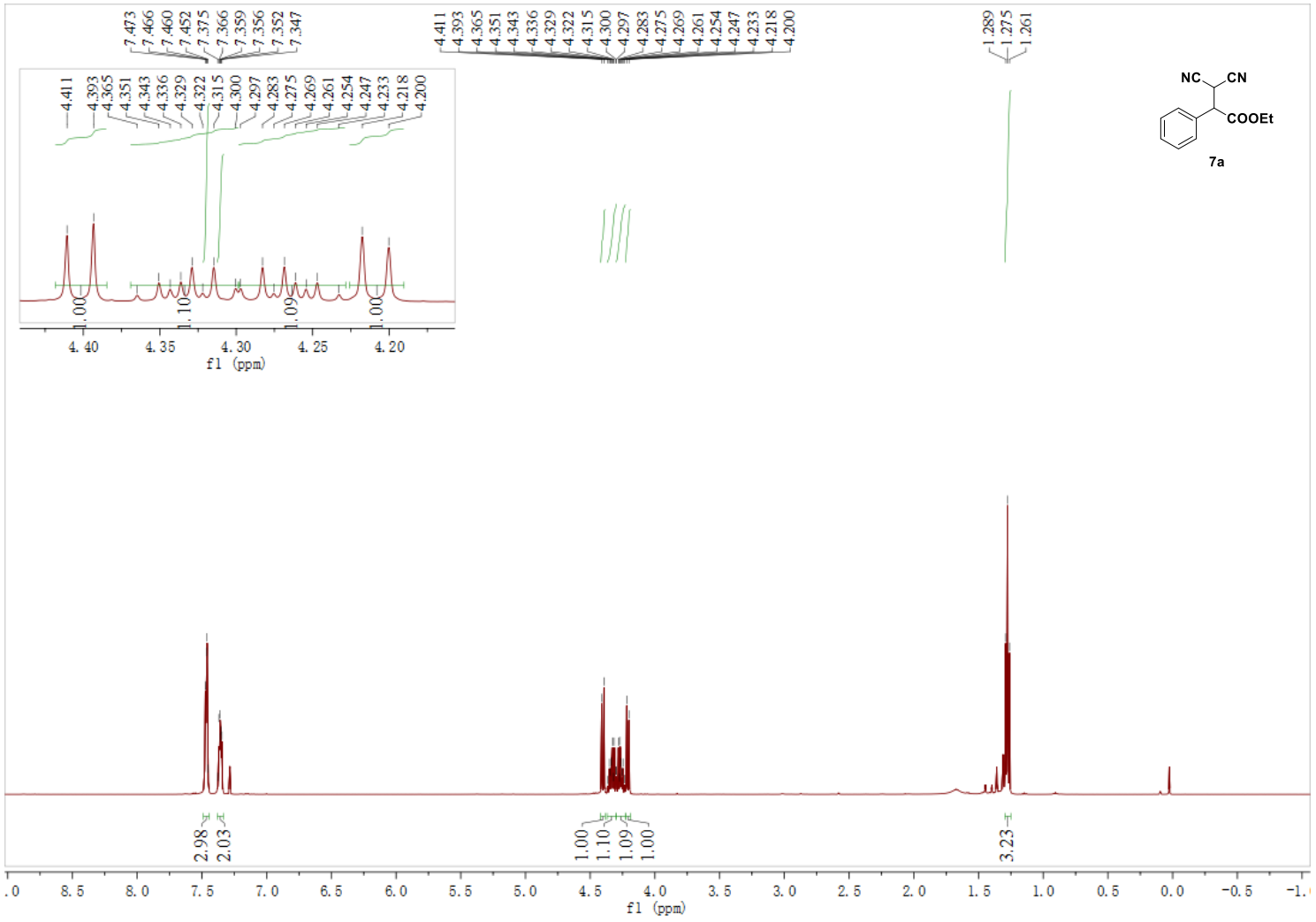
500 MHz, DMSO-d_6, ^{1}H NMR



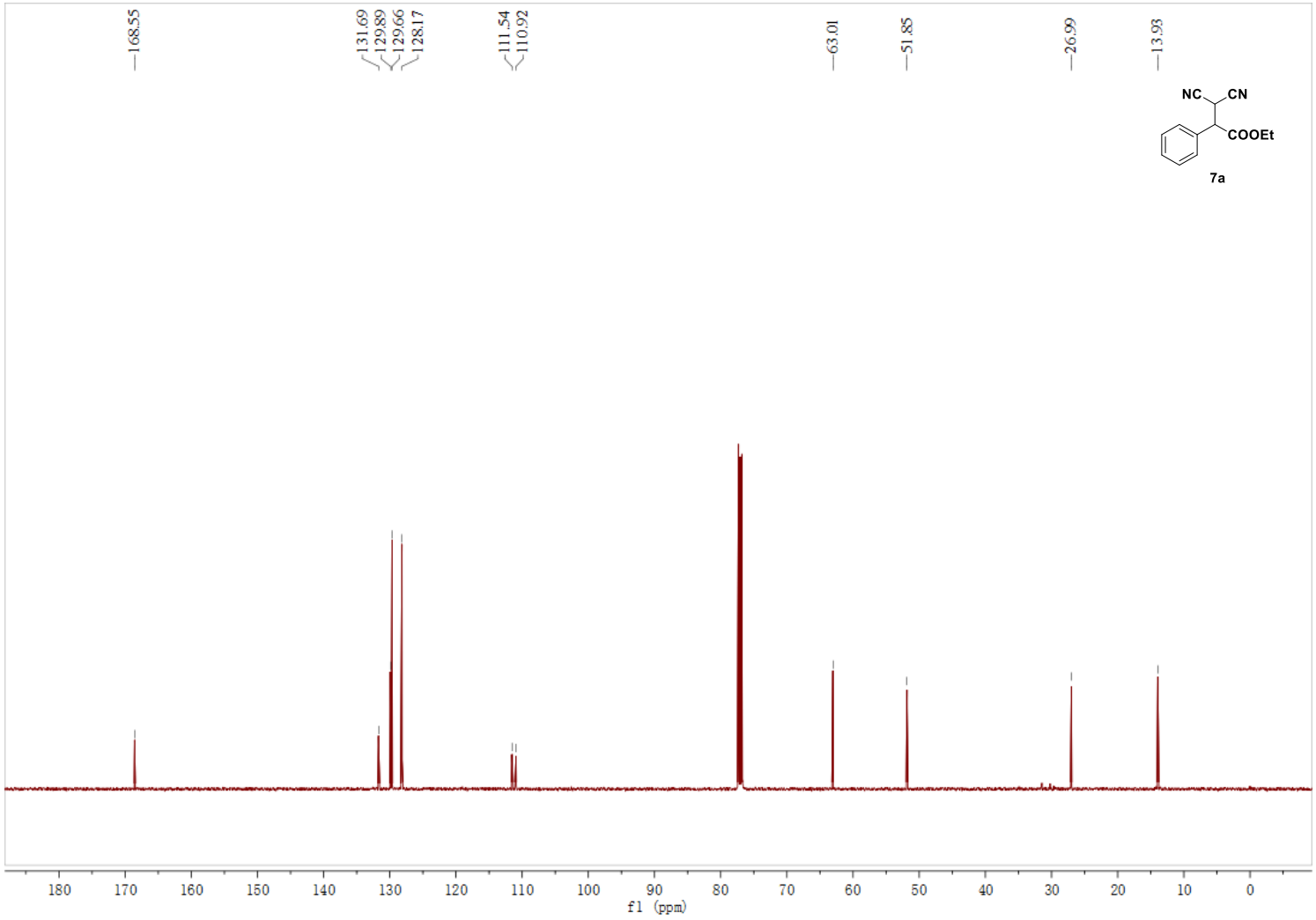
125 MHz, DMSO-d_6, ^{13}C NMR



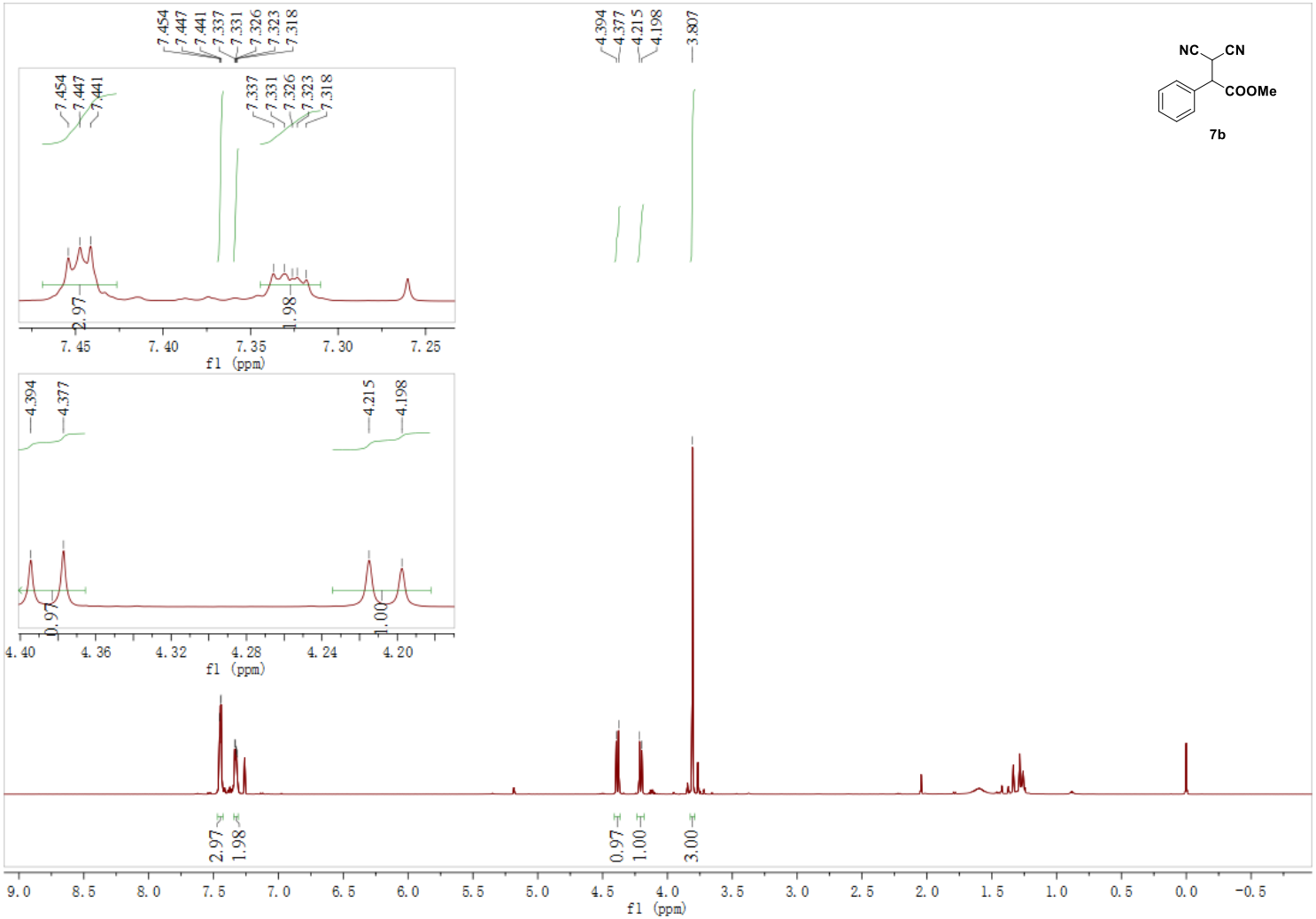
500 MHz, $CDCl_3$, ^1H NMR



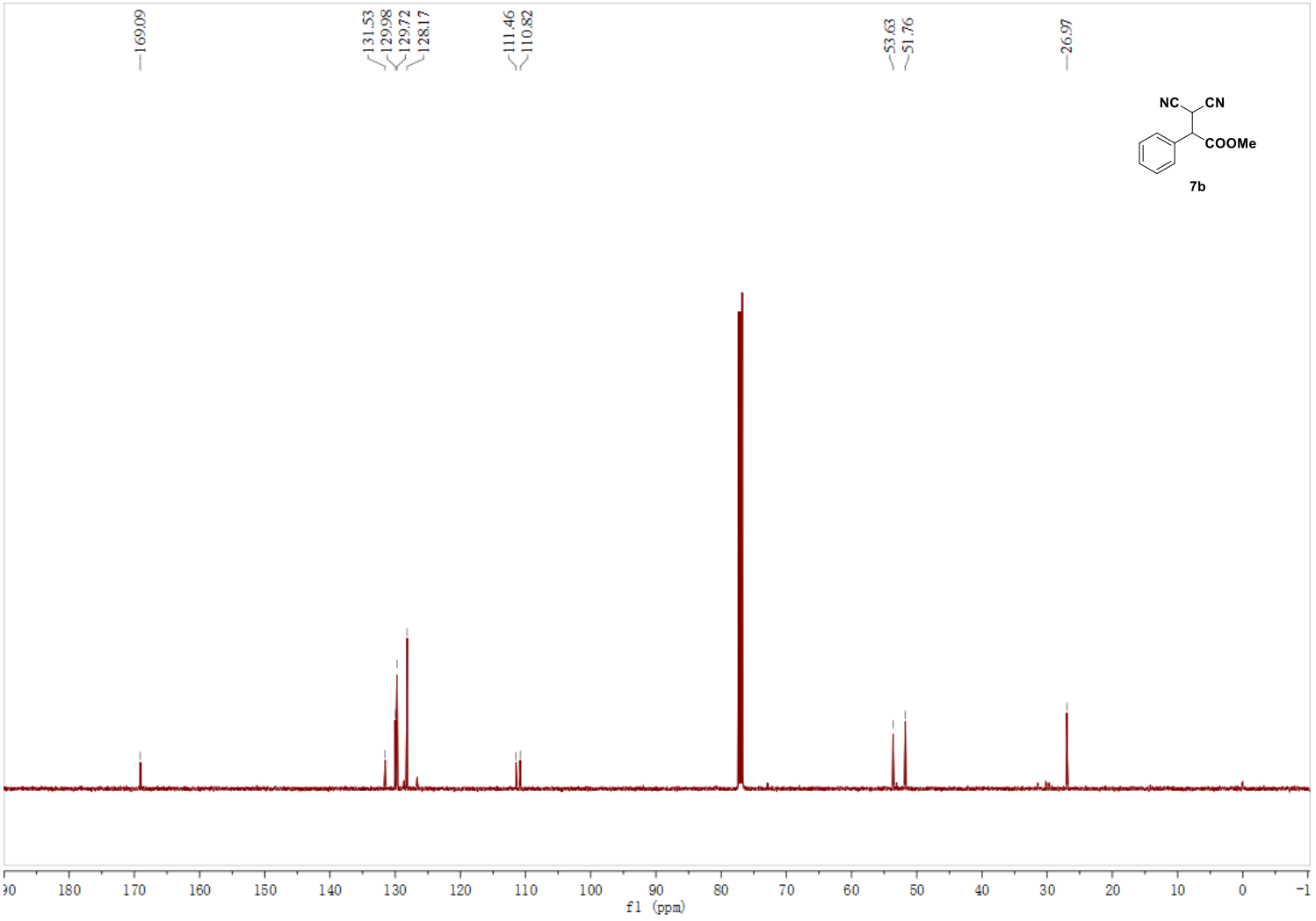
125 MHz, $CDCl_3$, ^{13}C NMR



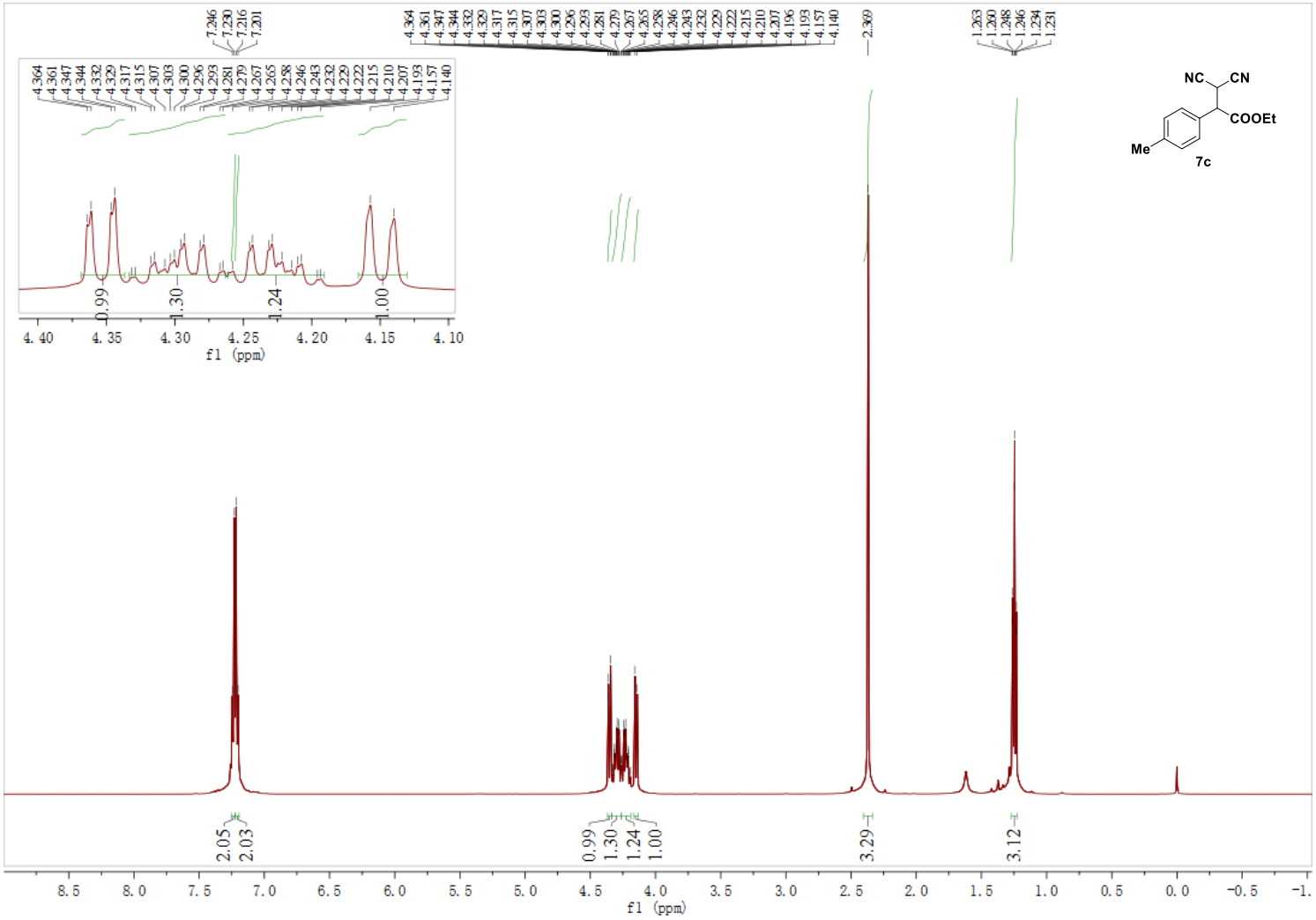
500 MHz, $CDCl_3$, ^1H NMR



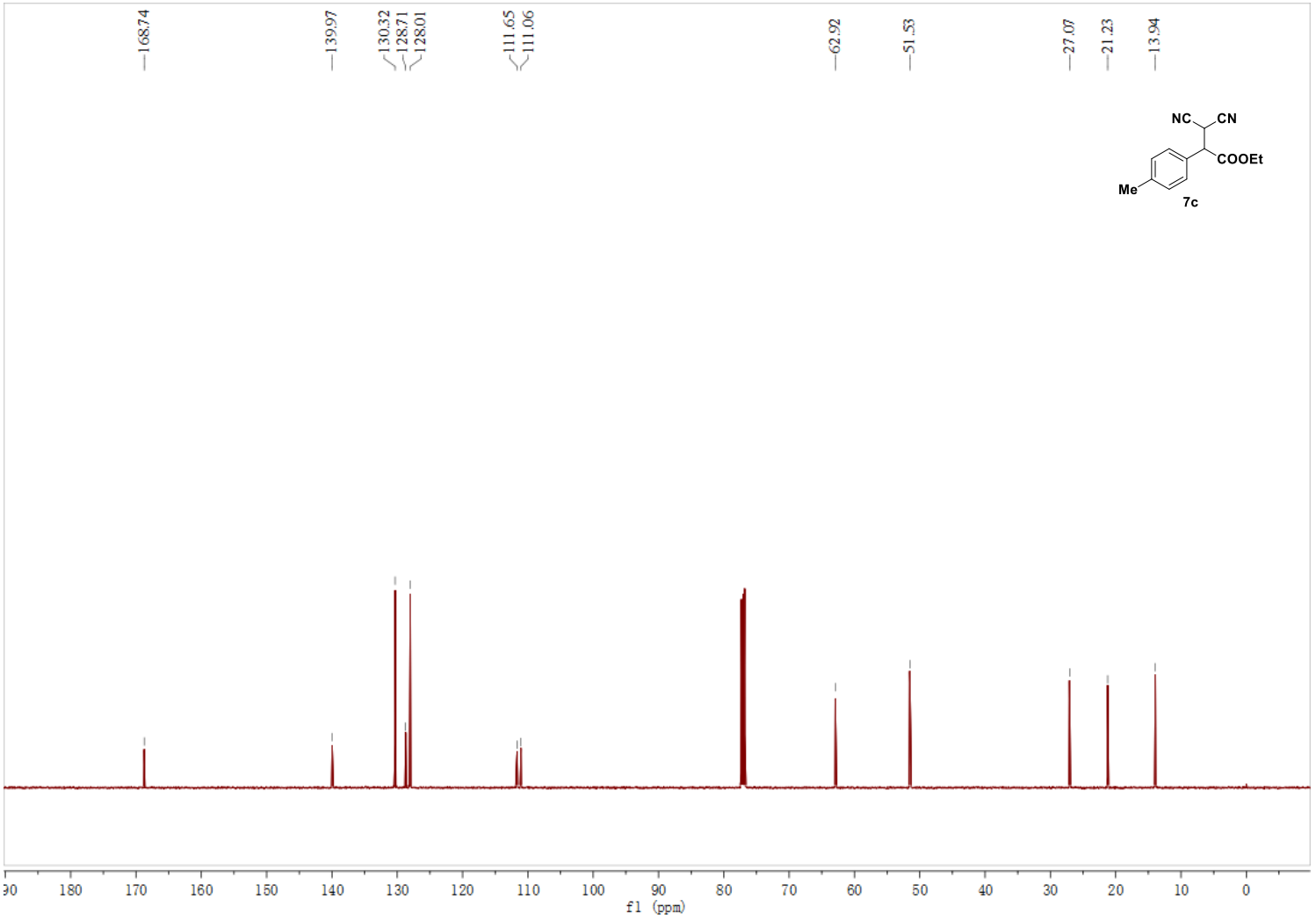
125 MHz, $CDCl_3$, ^{13}C NMR



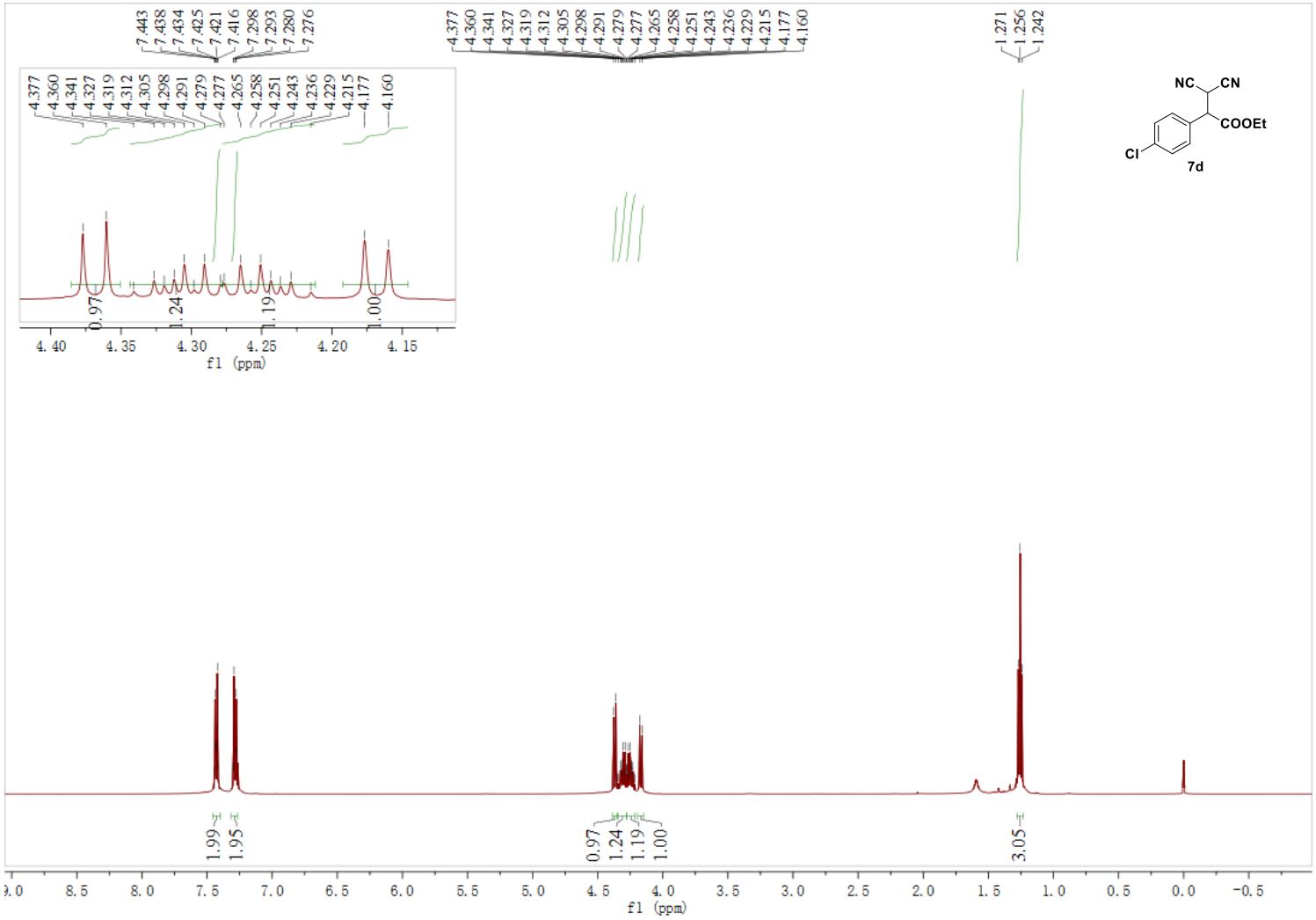
500 MHz, $CDCl_3$, ^1H NMR



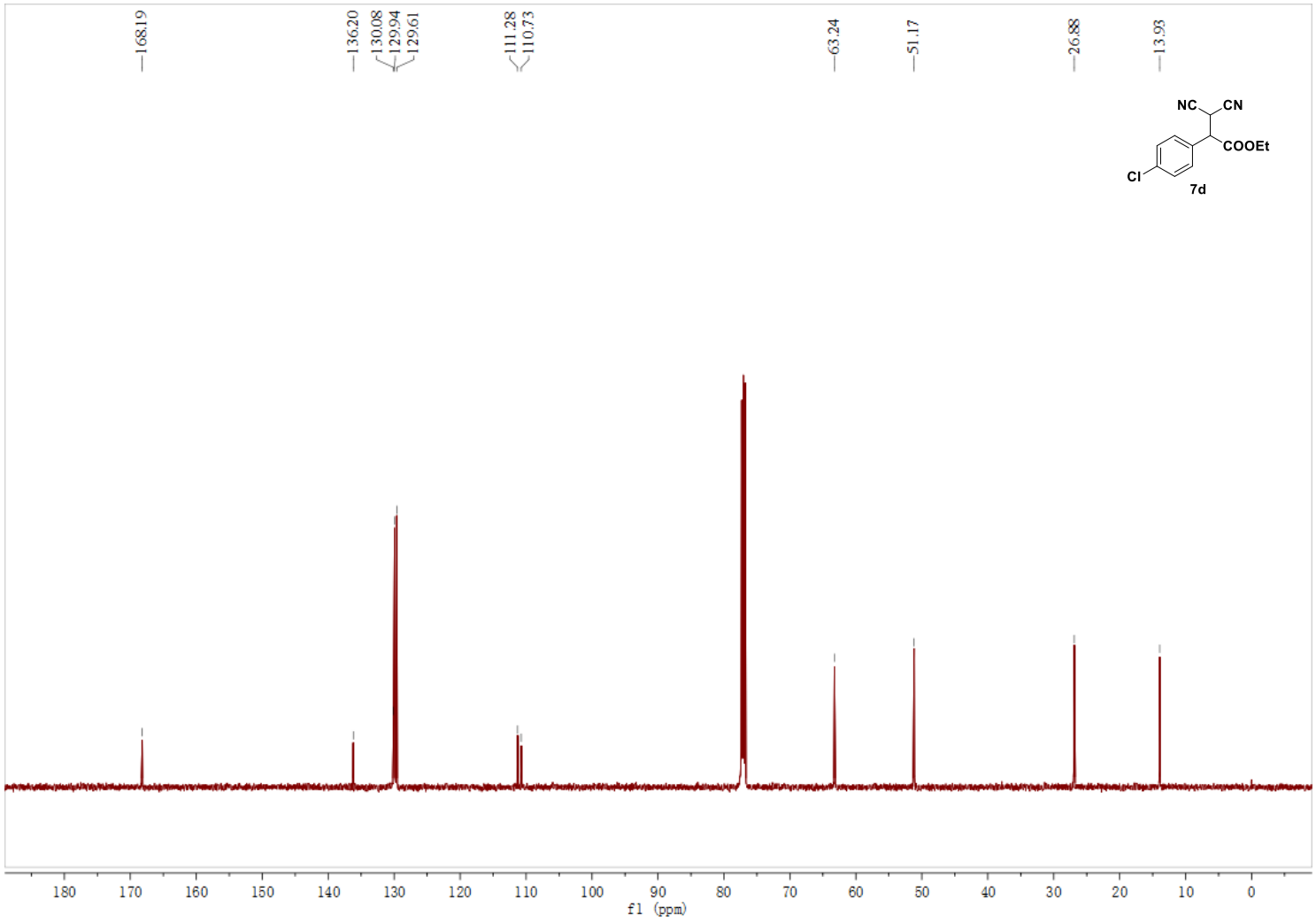
125 MHz, $CDCl_3$, ^{13}C NMR



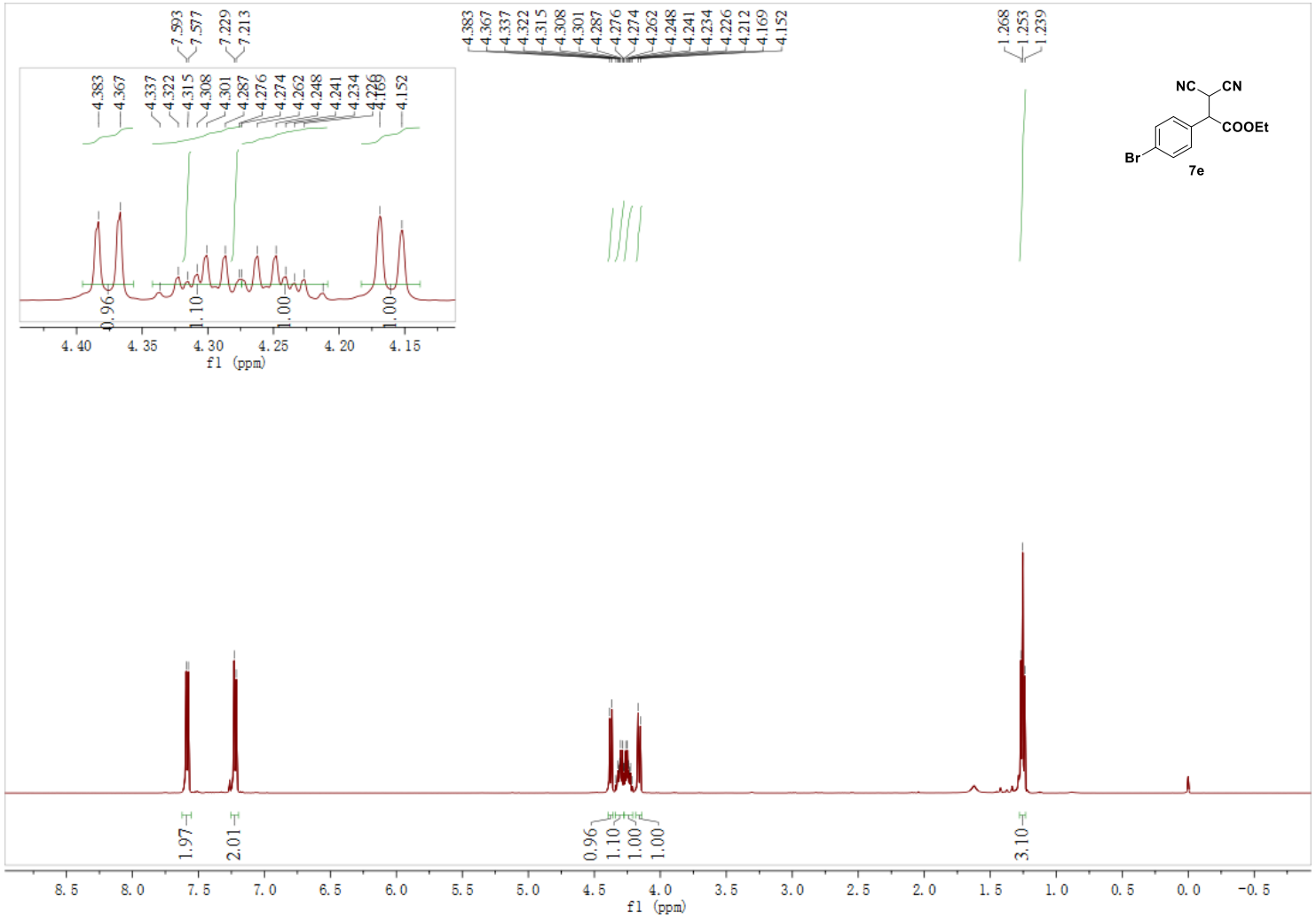
500 MHz, $CDCl_3$, ^1H NMR



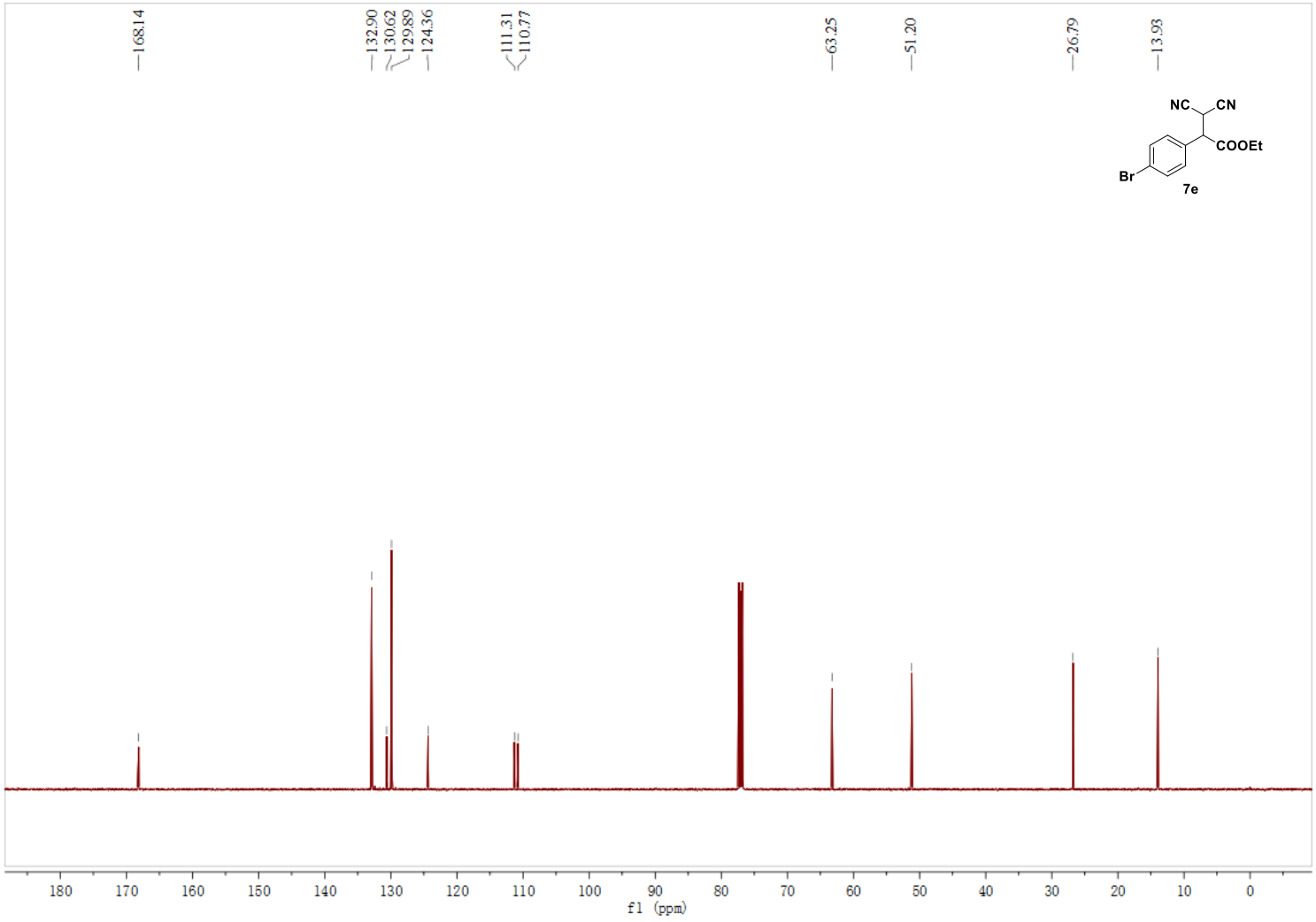
125 MHz, $CDCl_3$, ^{13}C NMR



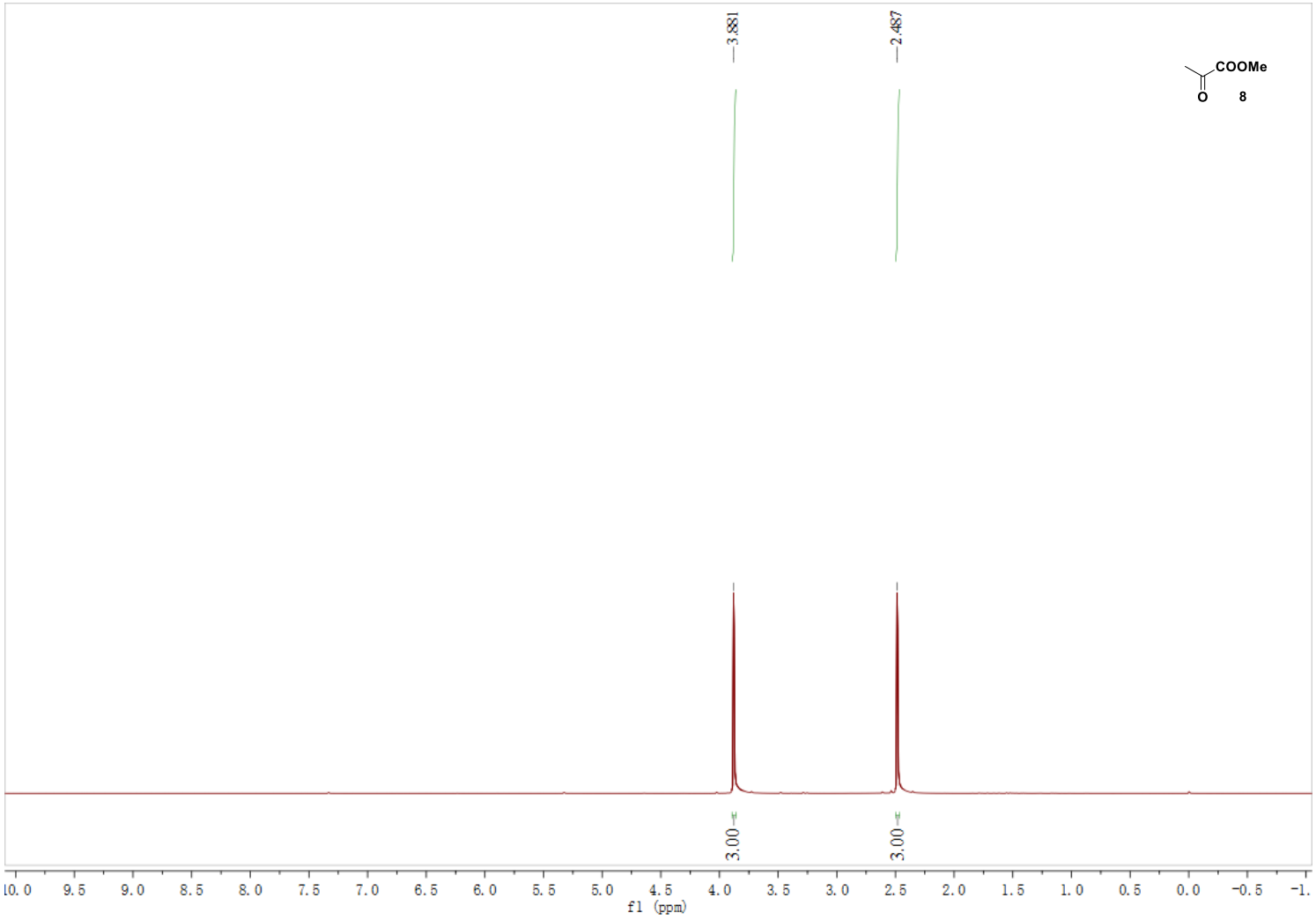
500 MHz, $CDCl_3$, ^1H NMR



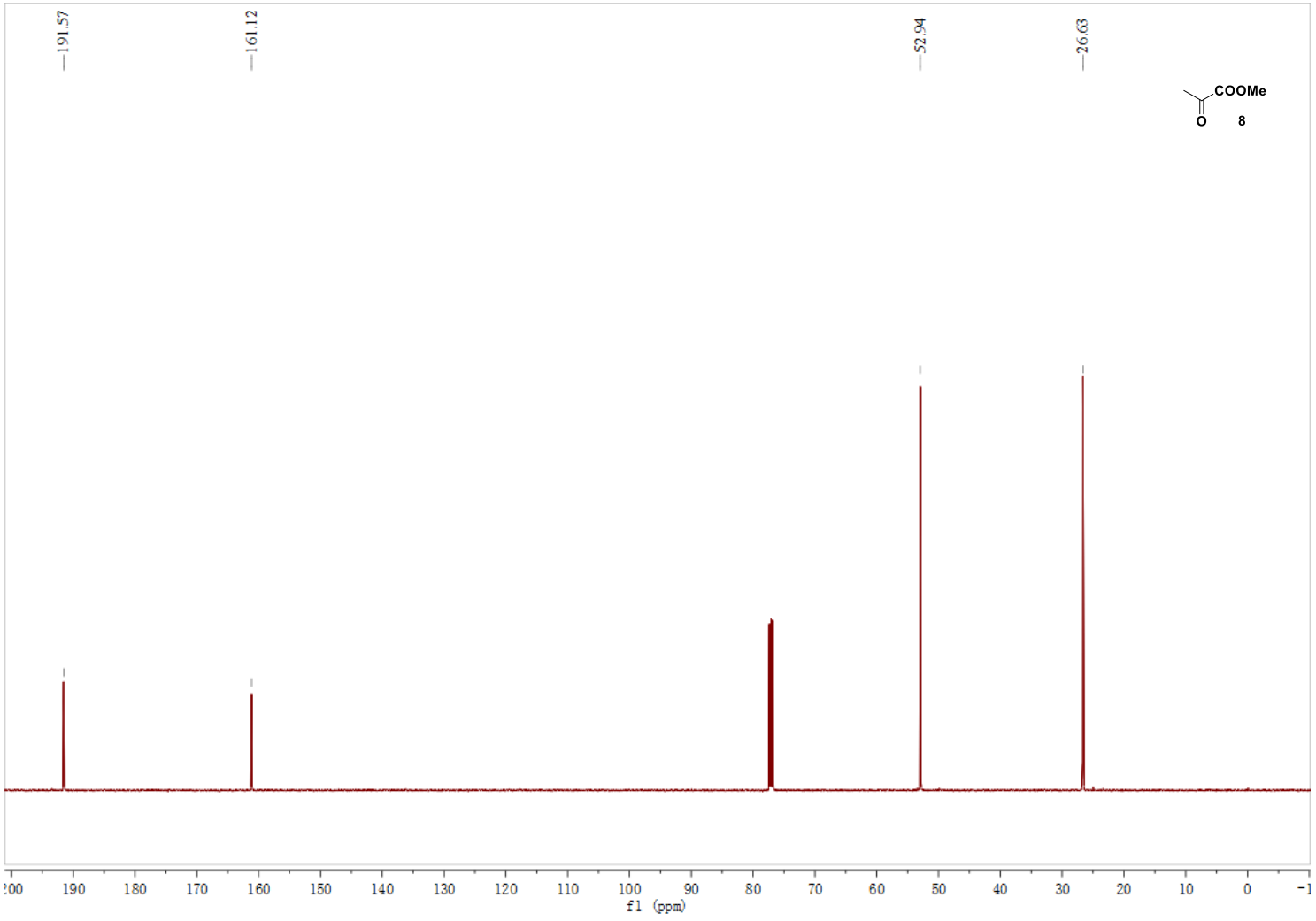
125 MHz, $CDCl_3$, ^{13}C NMR



500 MHz, $CDCl_3$, ^1H NMR



125 MHz, $CDCl_3$, ^{13}C NMR



500 MHz, $CDCl_3$, ^1H NMR