Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Facile Synthesis of Chiral Indolines through Asymmetric Hydrogenation of *in situ* Generated Indoles

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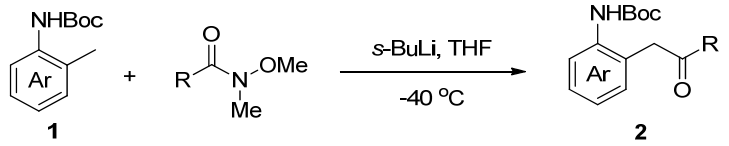
1. General

Commercially available reagents were used without further purification. Solvents were treated prior to use according to the standard methods. ^{1}H NMR and ^{13}C NMR spectra were recorded at room temperature in $CDCl_3$ on 400 MHz instrument with TMS (tetramethylsilane) as internal standard. Enantiomeric excess was determined by HPLC analysis, using chiral column described below in detail. Optical rotations were measured by polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh).

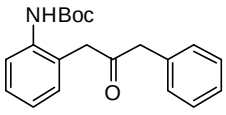
2. General Procedure for the Synthesis of Substrates

The compounds **2** can be prepared from compounds **1** and the corresponding Weinreb amides according to the known literature procedure with minor modification.[1] Coumpounds **1** could be conveniently prepared from di-*tert*-butyl dicarbonate and aryl amines.[2] Weinreb amides could be prepared from the corresponding acyl chloride and *N*,*O*-dimethylhydroxylamine hydrochloride.[3]

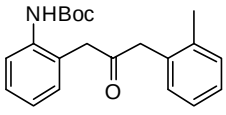


General procedure: Under nitrogen, compounds **1** (3 mmol) in THF (15 mL) was cooled to -40 °C. Then *sec*-butyllithium (*s*-BuLi, 6.90 mL of 1.0 M in hexane, 6.9 mmol) was added slowly. After stirring for one hour, a solution of Weinreb amide (3 mmol) in THF (5 mL) was added. The mixture was stirred at -40 °C over a period of thirty minutes and quenched with water (5 mL) carefully. Then the mixture was extracted with dichloromethane (10 mL×3), the combined organic layer was washed with brine, dried over sodium sulfate and concentrated in *vacuo*. The residue was purified by flash chromatography to give desired compounds **2**.

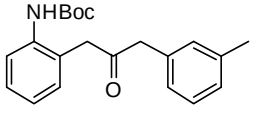
***tert*-Butyl (2-(2-oxo-3-phenylpropyl)phenyl)carbamate (2a):** white solid, mp 98-99 °C, 91% yield, new compound, R_f = 0.42 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 1H), 7.37-7.27 (m, 4H), 7.26-7.21 (m, 1H), 7.18-7.13 (m, 2H), 7.05-6.99 (m, 2H), 3.81 (s, 2H), 3.72 (s, 2H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.5, 153.6, 137.4, 133.3, 130.6, 129.6, 128.9, 128.3, 127.4, 125.4, 124.3, 123.7, 80.3, 49.8, 45.7, 28.4. HRMS Calculated for $C_{20}H_{24}NO_3$ $[M+H]^+$ 326.1751, found: 326.1756.



***tert*-Butyl (2-(2-oxo-3-(*o*-tolyl)propyl)phenyl)carbamate (2b):** white solid, mp 104-105 °C, 72% yield, new compound, R_f = 0.50 (hexanes/ethyl acetate 10/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.46 (brs, 1H), 7.28-7.22 (m, 2H), 7.22-7.14 (m, 3H), 7.12-7.08 (m, 1H), 7.04-6.94 (m, 2H), 3.82 (s, 2H), 3.71 (s, 2H), 2.09 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.7, 153.6, 137.5, 137.1, 132.2, 130.6, 130.6, 130.5, 128.3, 127.7, 126.4, 125.2, 124.3, 123.6, 80.3, 48.0, 45.8, 28.4, 19.4. HRMS Calculated for $C_{21}H_{25}KNO_3$ $[M+K]^+$ 378.1466, found: 378.1471.



***tert*-Butyl (2-(2-oxo-3-(*m*-tolyl)propyl)phenyl)carbamate (2c):** white solid, mp 109-110 °C, 48% yield, new compound, R_f = 0.50 (hexanes/ethyl acetate 10/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 1H), 7.37 (brs, 1H), 7.28-7.20 (m, 2H), 7.10 (d, *J* = 7.5 Hz, 1H), 7.05-7.95 (m, 4H), 3.77 (s, 2H), 3.71 (s, 2H), 2.33 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.7, 153.6, 138.6, 137.4,



133.2, 130.6, 130.3, 128.8, 128.2, 128.1, 126.6, 125.4, 124.3, 123.7, 80.3, 49.7, 45.7, 28.4, 21.4. HRMS Calculated for $C_{21}H_{25}KNO_3$ $[M+H]^+$ 378.1466, found: 378.1468.

***tert*-Butyl (2-(2-oxo-3-(*p*-tolyl)propyl)phenyl)carbamate (2d):** white solid, mp 91-92 °C, 33% yield, new compound, R_f = 0.50 (hexanes/ethyl acetate 10/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 1H), 7.38 (brs, 1H), 7.28-7.22 (m, 1H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.08-6.98 (m, 4H), 3.76 (s, 2H), 3.70 (s, 2H), 2.33 (s, 3H), 1.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.8, 153.6, 137.4, 137.0, 130.6, 130.2, 129.6, 129.4, 128.2, 125.5, 124.3, 123.7, 80.3, 49.4, 45.6, 28.4, 21.1. HRMS Calculated for $C_{21}H_{25}KNO_3$ $[M+K]^+$ 378.1466, found: 378.1463.

***tert*-Butyl (2-(2-oxopropyl)phenyl)carbamate (2e):** white solid, mp 54-55 °C, 50% yield, new compound, R_f = 0.33 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.34-7.22 (m, 2H), 7.15 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.4 Hz, 1H), 3.71 (s, 2H), 2.25 (s, 3H), 1.52 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.9, 153.6, 137.3, 130.6, 128.3, 125.4, 124.4, 123.6, 80.3, 47.7, 29.9, 28.4. HRMS Calculated for $C_{14}H_{20}NO_3$ $[M+H]^+$ 250.1438, found: 250.1442.

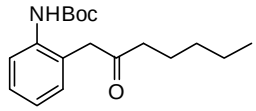
***tert*-Butyl (2-(2-oxobutyl)phenyl)carbamate (2f):** pale yellow solid, mp 44-45 °C, 48% yield, new compound, R_f = 0.40 (hexanes/ethyl acetate 20/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 1H), 7.58 (brs, 1H), 7.29-7.23 (m, 1H), 7.14 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.07-7.00 (m, 1H), 3.69 (s, 2H), 2.59 (q, *J* = 7.2 Hz, 2H), 1.52 (s, 9H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.8, 153.6, 137.4, 130.5, 128.2, 125.6, 124.2, 123.5, 80.2, 46.5, 36.0, 28.4, 7.5. HRMS Calculated for $C_{15}H_{21}NNaO_3$ $[M+Na]^+$ 286.1414, found: 286.1412.

***tert*-Butyl (2-(2-oxopentyl)phenyl)carbamate (2g):** pale red solid, mp 67-68 °C, 35% yield, new compound, R_f = 0.50 (hexanes/ethyl acetate 20/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 1H), 7.54 (brs, 1H), 7.29-7.23 (m, 1H), 7.17-7.12 (m, 1H), 7.04 (t, *J* = 7.4 Hz, 1H), 3.68 (s, 2H), 2.54 (t, *J* = 7.2 Hz, 2H), 1.63-1.57 (m, 2H), 1.52 (s, 9H), 0.88 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.3, 153.6, 137.4, 130.5, 128.2, 125.5, 124.2, 123.5, 80.2, 46.9, 44.6, 28.4, 17.0, 13.5. HRMS Calculated for $C_{16}H_{23}KNO_3$ $[M+K]^+$ 316.1310, found: 316.1309.

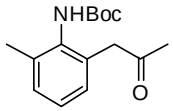
***tert*-Butyl (2-(3-methyl-2-oxobutyl)phenyl)carbamate (2h):** white solid, mp 73-74 °C, 58% yield, new compound, R_f = 0.55 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.65 (brs, 1H), 7.28-7.22 (m, 1H), 7.13 (d, *J* = 6.5 Hz, 1H), 7.06-7.01 (m, 1H), 3.75 (s, 2H), 2.86-2.74 (m, 1H), 1.52 (s, 9H), 1.14 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 214.0, 153.6, 137.5, 130.5, 128.1, 126.0, 124.2, 123.7, 80.2, 44.6, 41.0, 28.4, 18.0. HRMS Calculated for $C_{16}H_{24}NO_3$ $[M+H]^+$ 278.1751, found: 278.1755.

***tert*-Butyl (2-(2-oxohexyl)phenyl)carbamate (2i):** white solid, mp 51-52 °C, 50% yield, new compound, R_f = 0.60 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.0 Hz, 1H), 7.55 (brs, 1H), 7.30-7.21 (m, 1H), 7.16-7.11 (m, 1H), 7.07-7.01 (m, 1H), 3.68 (s, 2H), 2.56 (t, *J* = 7.3 Hz, 2H), 1.63-1.48 (m, 2H; s, 9H), 1.33-1.22 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.5, 153.6, 137.4, 130.5, 128.2, 125.4, 124.2, 123.4, 80.2, 46.9, 42.5, 28.4, 25.6, 22.2, 13.8. HRMS Calculated for $C_{17}H_{26}NO_3$ $[M+H]^+$ 292.1907, found: 292.1909.

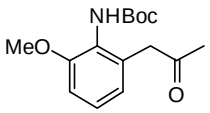
***tert*-Butyl (2-(2-oxoheptyl)phenyl)carbamate (2j):** white solid, mp 49-50 °C, 59% yield, new compound, R_f = 0.65 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.1 Hz, 1H), 7.56 (brs, 1H), 7.30-7.21 (m, 1H), 7.16-7.11 (m, 1H), 7.08-7.02 (m, 1H), 3.68 (s, 2H), 2.55 (t, *J* = 7.4 Hz, 2H), 1.62-1.55 (m, 2H), 1.52 (s, 9H), 1.31-1.19 (m, 4H), 0.86 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.5, 153.6, 137.4, 130.5, 128.2, 125.4, 124.2, 123.4, 80.2, 46.9, 42.7, 31.2, 28.4, 23.2, 22.4, 13.9. HRMS Calculated for $C_{18}H_{28}NO_3$ $[M+H]^+$ 306.2064, found: 306.2062.



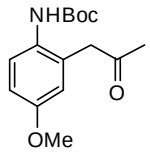
***tert*-Butyl (2-methyl-6-(2-oxopropyl)phenyl)carbamate (2k):** white solid, mp 84-85 °C, 68% yield, new compound, R_f = 0.40 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18-7.08 (m, 2H), 7.01 (d, *J* = 7.1 Hz, 1H), 6.34 (brs, 1H), 3.72 (s, 2H), 2.27 (s, 3H), 2.19 (s, 3H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.1, 153.8, 136.9, 134.7, 132.4, 130.0, 128.2, 127.1, 80.0, 47.7, 29.9, 28.3, 18.4. HRMS Calculated for $C_{15}H_{22}NO_3$ $[M+H]^+$ 264.1594, found: 264.1594.



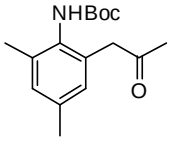
***tert*-Butyl (2-methoxy-6-(2-oxopropyl)phenyl)carbamate (2l):** pale yellow oil, 69% yield, new compound, R_f = 0.40 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (t, *J* = 8.0 Hz, 1H), 6.87-6.76 (m, 2H), 6.09 (brs, 1H), 3.83 (s, 3H), 3.76 (s, 2H), 2.17 (s, 3H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.4, 154.5, 154.4, 133.3, 127.2, 125.2, 122.8, 110.0, 80.2, 55.7, 47.3, 29.8, 28.3. HRMS Calculated for $C_{15}H_{21}NNaO_4$ $[M+Na]^+$ 302.1363, found: 302.1359.



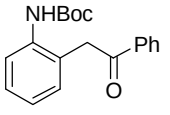
***tert*-Butyl (4-methoxy-2-(2-oxopropyl)phenyl)carbamate (2m):** white solid, mp 82-83 °C, 70% yield, new compound, R_f = 0.35 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.3 Hz, 1H), 6.92-6.78 (m, 1H; brs, 1H), 6.69 (d, *J* = 2.9 Hz, 1H), 3.77 (s, 3H), 3.68 (s, 2H), 2.23 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.3, 156.8, 154.1, 129.9, 128.8, 126.4, 116.2, 113.0, 80.1, 55.5, 47.6, 29.9, 28.4. HRMS Calculated for $C_{15}H_{21}KNO_4$ $[M+K]^+$ 318.1102, found: 318.1106.



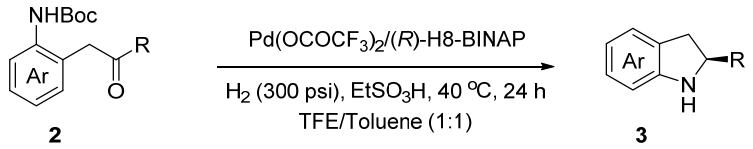
***tert*-Butyl (2,4-dimethyl-6-(2-oxopropyl)phenyl)carbamate (2n):** white solid, mp 81-82 °C, 64% yield, new compound, R_f = 0.50 (hexanes/ethyl acetate 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.97 (s, 1H), 6.82 (s, 1H), 6.16 (brs, 1H), 3.69 (s, 2H), 2.27 (s, 3H), 2.23 (s, 3H), 2.20 (s, 3H), 1.48 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.2, 154.0, 136.7, 136.6, 132.3, 132.0, 130.6, 128.9, 79.7, 47.6, 29.7, 28.3, 20.9, 18.2. HRMS Calculated for $C_{16}H_{24}NO_3$ $[M+H]^+$ 278.1751, found: 278.1752.



***tert*-Butyl (2-(2-oxo-2-phenylethyl)phenyl)carbamate (2o):** white solid, 69% yield, known compound,[1] R_f = 0.50 (hexanes/ethyl acetate 10/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (dd, J = 5.2, 3.3 Hz, 2H), 7.79 (d, J = 7.9 Hz, 1H), 7.71-7.58 (m, 2H), 7.53 (dd, J = 10.5, 4.7 Hz, 2H), 7.31-7.26 (m, 1H), 7.23 (dd, J = 7.6, 1.3 Hz, 1H), 7.07 (td, J = 7.5, 1.2 Hz, 1H), 4.31 (s, 2H), 1.54 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.1, 153.7, 137.6, 136.2, 133.8, 130.7, 128.8, 128.1, 124.4, 80.3, 42.0, 28.4.



3. Asymmetric Hydrogenation of *in situ* Generated Indoles

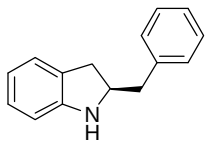


2 **3**

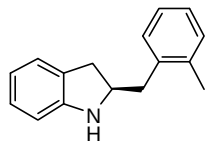
Ligand (*R*)-H8-BINAP (4.8 mg, 0.0076 mmol) and $Pd(OCOCF_3)_2$ (2.1 mg, 0.0063 mmol) were placed in a dried Schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone was added. The mixture was stirred at room temperature for one hour. The solvent was removed under vacuum to give the catalyst. This catalyst was taken into a glove box filled with nitrogen and dissolved in 2,2,2-trifluoroethanol (2 mL). To the mixture of compounds **2** (0.25 mmol) and ethanesulfonic acid (41 μL, 0.50 mmol) in toluene (2 mL), this catalyst solution was added, and then the mixture was transferred to an autoclave, which was charged hydrogen gas (300 psi). The autoclave was stirred at 40 °C for 24 h. After release of the hydrogen, the autoclave was opened and the reaction mixture was evaporated. Then, saturated sodium hydrogencarbonate (5 mL) was added. The mixture was extracted with dichloromethane (5 mL×3), the combined organic layer was dried over sodium sulfate and concentrated in *vacuo*. Purification was performed on silica gel using ethyl acetate/hexanes as the eluent to give the chiral products **3**.

Racemates of **3** were prepared using (±)-BINAP/$Pd(OCOCF_3)_2$ as racemic catalyst.

(+)-(*R*)-2-Benzylindoline (3a): 51 mg, 98% yield, colorless oil, known compound, R_f = 0.65 (hexanes/ethyl acetate 10/1), 95% ee, $[\alpha]^{20}_D$ = +95.09 (*c* 1.02, $CHCl_3$), [lit.[4]: $[\alpha]^{RT}_D$ = +80.2 (c 1.00, $CHCl_3$) for 95% e.e.]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.29 (m, 2H), 7.28-7.20 (m, 3H), 7.07 (d, *J* = 7.3 Hz, 1H), 7.03-6.97 (m, 1H), 6.71-6.65 (m, 1H), 6.55 (d, *J* = 7.7 Hz, 1H), 4.12-4.02 (m, 1H), 3.81 (brs, 1H), 3.18-3.08 (m, 1H), 2.94-2.73 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 139.1, 129.2, 128.7, 128.4, 127.4, 126.5, 124.8, 118.6, 109.1, 61.0, 42.7, 36.0. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 14.8 min (maj), t2 = 16.6 min.



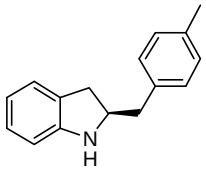
(+)-(*R*)-2-(2-Methylbenzyl)indoline (3b): 51 mg, 91% yield, colorless oil, known compound, R_f = 0.60 (hexanes/ethyl acetate 20/1), 94% ee, $[\alpha]^{20}_D$ = +90.49 (*c* 1.00, $CHCl_3$), [lit.[4]: $[\alpha]^{RT}_D$ = +74.8 (c 1.50, $CHCl_3$) for 94% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19-7.12 (m, 4H), 7.08 (d, *J* = 7.2 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.71-6.67 (m, 1H), 6.54 (d, *J* = 7.7 Hz, 1H), 4.14-4.01 (m, 1H), 3.54 (brs, 1H), 3.13 (dd, *J* = 15.5, 8.4 Hz, 1H), 2.93-2.75 (m, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 137.3, 136.6, 130.6, 129.8, 128.4, 127.4, 126.6, 126.1, 124.9, 118.6, 109.2, 59.7, 39.7, 36.1, 19.7. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 13.8 min (maj), t2 = 15.7 min.



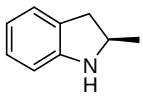
(+)-(*R*)-2-(3-Methylbenzyl)indoline (3c): 50 mg, 90% yield, colorless oil, known compound, R_f = 0.55 (hexanes/ethyl acetate 20/1). 95% ee, $[\alpha]^{20}_D$ = +86.69 (*c* 1.00, $CHCl_3$), [lit.[4]: $[\alpha]^{20}_D$ = +75.4 (c 1.60, $CHCl_3$) for 94% e.e.]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.16 (m, 1H), 7.10-7.95 (m, 5H), 6.70-6.65 (m, 1H), 6.54 (d, *J* = 7.7 Hz, 1H), 4.10-3.99 (m, 1H), 3.57 (brs, 1H), 3.16-3.07 (m, 1H), 2.88-2.72 (m, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 139.1, 138.3, 130.0, 128.6, 128.5, 127.4, 127.2, 126.2, 124.9, 118.6, 109.2, 61.0, 42.7, 36.0, 21.5. HPLC (OD-H,

elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 11.6 min (maj), t2 = 12.9 min.

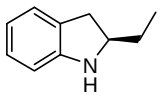
(+)-(*R*)-2-(4-Methylbenzyl)indoline (3d): 52 mg, 93% yield, colorless oil, known compound, R_f = 0.50 (hexanes/ethyl acetate 20/1). 95% ee, $[\alpha]^{20}_D$ = +87.95 (*c* 0.98, $CHCl_3$), [lit.[4]: $[\alpha]^{RT}_D$ = +75.5 (c 1.50, $CHCl_3$) for 93% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16-7.04 (m, 5H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.70-6.65 (m, 1H), 6.54 (d, *J* = 7.7 Hz, 1H), 4.10-3.99 (m, 1H), 3.71 (brs, 1H), 3.16-3.06 (m, 1H), 2.89-2.73 (m, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 136.0, 136.0, 129.4, 129.0, 128.5, 127.4, 124.9, 118.5, 109.1, 61.1, 42.3, 35.9, 21.1. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 11.6 min (maj), t2 = 12.9 min.



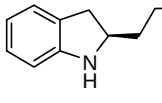
(+)-(*R*)-2-Methylindoline (3e): 32 mg, 96% yield, colorless oil, known compound, R_f = 0.75 (hexanes/ethyl acetate 10/1). 90% ee, $[\alpha]^{20}_D$ = +3.98 (*c* 0.56, benzene), [lit.[4]: $[\alpha]^{RT}_D$ = +6.96 (c 0.63, benzene) for 91% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 7.1 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.71-6.65 (m, 1H), 6.59 (d, *J* = 7.7 Hz, 1H), 4.05-3.92 (m, 1H), 3.67 (brs, 1H), 3.18-3.07 (m, 1H), 2.67-5-2.59 (m, 1H), 1.27 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 128.9, 127.3, 124.7, 118.6, 109.2, 55.2, 37.8, 22.3. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 97/3, detector: 254 nm, 30 °C, flow rate: 0.8 mL/min), t1 = 11.0 min (maj), t2 = 12.5 min.



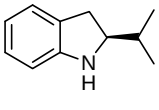
(+)-(*R*)-2-Ethylindoline (3f): 30 mg, 82% yield, colorless oil, known compound, R_f = 0.60 (hexanes/ethyl acetate 20/1). 94% ee, $[\alpha]^{20}_D$ = +5.88 (*c* 0.34, $CHCl_3$), [lit.[5]: $[\alpha]^{22}_D$ = -5.5 (c 0.2, $CHCl_3$) for 97% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.67 (t, *J* = 7.2 Hz, 1H), 6.59 (d, *J* = 7.7 Hz, 1H), 3.81-3.72 (m, 1H), 3.45-3.05 (m, 1H; brs, 1H), 2.72-2.62 (m, 1H), 1.68-1.56 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 128.9, 127.2, 124.7, 118.4, 109.1, 61.5, 35.8, 29.6, 10.7. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 95/5, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 8.0 min (maj), t2 = 9.0 min.



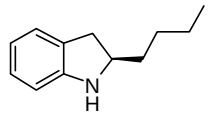
(+)-(*R*)-2-Propylindoline (3g): 34 mg, 84% yield, colorless oil, known compound, R_f = 0.60 (hexanes/ethyl acetate 20/1). 94% ee, $[\alpha]^{20}_D$ = +11.50 (*c* 0.40, $CHCl_3$), [lit.[6]: $[\alpha]^{25}_D$ = +9.3 (c 0.3, $CHCl_3$) for 96% e.e.]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.67 (t, *J* = 7.3 Hz, 1H), 6.59 (d, *J* = 7.7 Hz, 1H), 3.91-3.79 (m, 1H), 3.47-3.05 (m, 1H; brs, 1H), 2.71-2.62 (m, 1H), 1.65-1.51 (m, 2H), 1.47-1.34 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 128.9, 127.2, 124.7, 118.5, 109.1, 59.8, 39.1, 36.2, 19.8, 14.2. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 95/5, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 7.7 min (maj), t2 = 9.2 min.



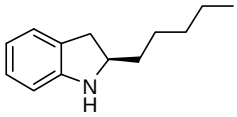
(+)-(*S*)-2-Isopropylindoline (3h): 38 mg, 94% yield, colorless oil, known compound,[7] R_f = 0.80 (hexanes/ethyl acetate 10/1). 96% ee, $[\alpha]^{20}_D$ = -12.86 (*c* 0.70, $CHCl_3$), ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.04 (d, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.65 (t, *J* = 7.4 Hz, 1H), 6.57 (d, *J* = 7.7 Hz, 1H), 3.95-3.50 (m, 1H; brs, 1H), 3.10-3.00 (m, 1H), 2.76-2.66 (m, 1H), 1.80-1.70(m, 1H), 0.98 (d, *J* = 6.6 Hz, 3H), 0.93 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 129.2, 127.2, 124.5, 118.3, 108.8, 66.6, 34.2, 34.0, 19.6, 19.1. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 10.7 min (maj), t2 = 17.5 min.



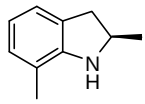
(+)-(*R*)-2-Butylindoline (3i): 43 mg, 98% yield, colorless oil, known compound, R_f = 0.85 (hexanes/ethyl acetate 10/1). 94% ee, $[\alpha]^{20}_D$ = +16.28 (*c* 0.86, $CHCl_3$), [lit.[4]: $[\alpha]^{RT}_D$ = +12.6 (c 1.1, $CHCl_3$) for 93% e.e.]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.67 (t, *J* = 7.4 Hz, 1H), 6.59 (d, *J* = 7.7 Hz, 1H), 4.01-3.63 (m, 1H; brs, 1H), 3.16-3.06 (m, 1H), 2.70-2.62 (m, 1H), 1.66-1.54 (m, 2H), 1.41-1.30 (m, 4H), 0.92 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 128.9, 127.2, 124.7, 118.4, 109.1, 60.1, 36.6, 36.2, 28.8, 22.8, 14.1. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 9.5 min (maj), t2 = 13.1 min.



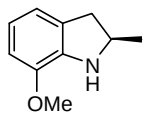
(+)-(*R*)-2-Pentylindoline (3j): 46 mg, 97% yield, colorless oil, known compound, R_f = 0.85 (hexanes/ethyl acetate 10/1). 93% ee, $[\alpha]^{20}_D$ = +15.87 (*c* 0.92, $CHCl_3$), [lit.[4]: $[\alpha]^{RT}_D$ = +15.5 (c 1.40, $CHCl_3$) for 92% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08-6.96 (m, 2H), 6.69-6.56 (m, 2H), 4.10-3.3.54 (m, 1H; brs, 1H), 3.15-3.05 (m, 1H), 2.70-2.60 (m, 1H), 1.66-1.52 (m, 2H), 1.47-1.24 (m, 6H), 0.90 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 128.9, 127.2, 124.6, 118.4, 109.1, 60.1, 36.8, 36.2, 31.9, 26.3, 22.7, 14.0. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 9.6 min (maj), t2 = 12.4 min.



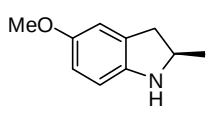
(+)-(*R*)-2,7-Dimethylindoline (3k): 35 mg, 94% yield, colorless oil, known compound, R_f = 0.65 (hexanes/ethyl acetate 10/1). 96% ee, $[\alpha]^{20}_D$ = +8.12 (*c* 0.48, $CHCl_3$), ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96-6.82 (m, 2H), 6.63 (t, *J* = 7.4 Hz, 1H), 4.05-3.92 (m, 1H), 3.50 (brs, 1H), 3.20-3.10 (m, 1H), 2.70-2.60 (m, 1H), 2.12 (s, 3H), 1.30 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.6, 128.3, 128.2, 122.2, 118.7, 118.6, 55.2, 38.1, 22.5, 16.9. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 11.1 min, t2 = 12.4 min (maj).



(+)-(*R*)-7-Methoxy-2-methylindoline (3l): 37 mg, 91% yield, colorless oil, new compound, R_f = 0.40 (hexanes/ethyl acetate 20/1). 80% ee, $[\alpha]^{20}_D$ = +8.09 (*c* 0.68, $CHCl_3$), ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (d, *J* = 7.0 Hz, 1H), 6.71-6.61 (m, 2H), 4.07-3.96 (m, 1H), 3.81 (s, 3H), 3.59 (brs, 1H), 3.20-3.12 (m, 1H), 2.70-2.62 (m, 1H), 1.30 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 139.9, 129.9, 119.1, 117.3, 109.2, 55.7, 55.3, 38.4, 22.3. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 13.9 min, t2 = 16.6 min (maj). HRMS Calculated for $C_{10}H_{14}NO$ $[M+H]^+$ 164.1070, found: 164.1072.

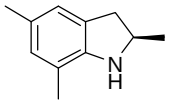


(+)-(*R*)-5-Methoxy-2-methylindoline (3m): 33 mg, 81% yield, yellow oil, known compound, R_f = 0.20 (hexanes/ethyl acetate 20/1). 84% ee, $[\alpha]^{20}_D$ = +7.00 (*c* 0.60, $CHCl_3$), [lit.[6]: $[\alpha]^{25}_D$ = +10.2 (c 0.50, $CHCl_3$) for 95% ee]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.71 (s, 1H), 6.61-6.51 (m, 2H), 4.02-3.91 (m, 1H), 3.73 (s, 3H), 3.21 (brs, 1H), 3.14-3.06 (m, 1H), 2.66-2.56 (m, 1H), 1.28 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5, 144.7, 130.8, 112.1, 111.7, 109.9, 56.0, 55.7, 38.3, 22.2. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 95/5, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 9.2 min (maj), t2 = 18.4 min.



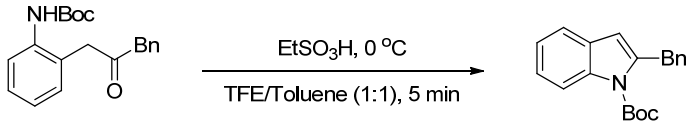
(+)-(*R*)-2,5,7-Trimethylindoline (3n): 36 mg, 90% yield, colorless oil, new compound, R_f = 0.70 (hexanes/ethyl acetate 10/1). 94% ee, $[\alpha]^{20}_D$ = +10.48 (*c* 0.42, $CHCl_3$), ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.76 (s, 1H), 6.67 (s, 1H), 4.02-3.89 (m, 1H), 3.35 (brs, 1H), 3.15-3.05 (m, 1H),

2.65-2.55 (m, 1H), 2.22 (s, 3H), 2.09 (s, 3H), 1.29 (d, J = 6.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.2, 128.8, 128.7, 128.2, 122.9, 118.6, 55.4, 38.2, 22.4, 20.8, 16.8. HPLC (OD-H, elute: *n*-hexane/ *i*-PrOH = 99/1, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 7.3 min, t2 = 8.1 min (maj). HRMS Calculated for $C_{11}H_{16}N$ $[M+H]^+$ 162.1277, found: 162.1282.



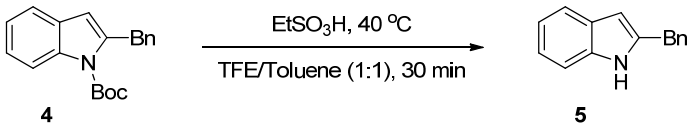
(-)-(*S*)-2-Phenylindoline (3o): 27 mg, 55% yield, white solid, known compound, R_f = 0.70 (hexanes/ethyl acetate 10/1). 68% ee, $[\alpha]^{20}_D$ = -45.9 (*c* 0.54, $CHCl_3$), [lit.[8]: $[\alpha]^{25}_{D=}$ -80.1 (c 1.0, $CHCl_3$) for > 99% ee of the (*S*)-enantiomer]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.31 (m, 2H), 7.29-7.22 (m, 2H), 7.22-7.16 (m, 1H), 7.00 (dd, J = 12.6, 7.3 Hz, 2H), 6.68 (t, J = 7.3 Hz, 1H), 6.61 (d, J = 7.7 Hz, 1H), 4.88 (t, J = 9.0 Hz, 1H), 3.60 (br, 1H), 3.36 (dd, J = 15.6, 9.2 Hz, 1H), 2.92 (dd, J = 15.6, 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 144.5, 128.8, 128.5, 127.8, 127.7, 126.5, 124.8, 119.1, 109.5, 63.7, 39.7. HPLC (OD-H, elute: *n*-hexane/*i*-PrOH = 90/10, detector: 254 nm, 30 °C, flow rate: 1.0 mL/min), t1 = 12.7 min (maj), t2 = 21.3 min.

4. The Mechanistic Investigation



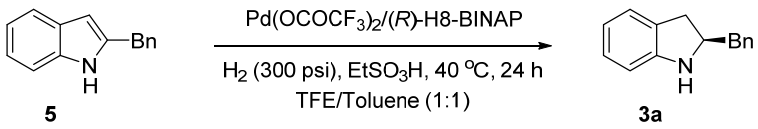
2a **4**

Under nitrogen, to a solution of compound **2a** (32 mg, 0.1 mmol) in 2,2,2-trifluoroethanol/ toluene (1 mL/1 mL) was added ethanesulfonic acid (16 uL, 0.2 mmol). After stirring for five minutes, the mixture was quenched with saturated sodium hydrogencarbonate (5 mL). Then the mixture was extracted with ethyl acetate (5 mL×3), the combined organic layer was washed with brine, dried over anhydrous sodium sulfate, and concentrated in *vacuo*. The residue was purified by flash chromatography to give *tert*-butyl 2-benzyl-1*H*-indole-1-carboxylate **4** (24 mg, 78% yield, colorless oil, known compound,[1] R_f = 0.80 (hexanes/ethyl acetate 20/1). 1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.4 Hz, 1H), 7.35-7.30 (m, 1H), 7.26-7.20 (m, 2H), 7.19-7.07 (m, 5H), 6.05 (d, J = 0.5 Hz, 1H), 4.29 (s, 2H), 1.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.5, 140.5, 139.2, 136.9, 129.1, 129.0, 128.4, 126.3, 123.5, 122.7, 119.9, 115.6, 109.3, 83.9, 36.4, 28.1.



4 **5**

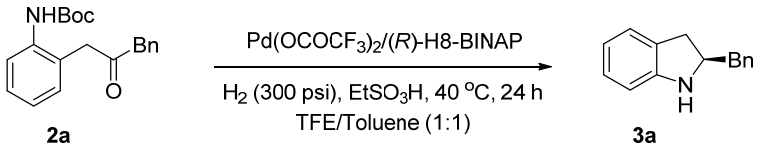
Under nitrogen, to a solution of compound **4** (46 mg, 0.15 mmol) in 2,2,2-trifluoroethanol/ toluene (2 mL/2 mL) was added ethanesulfonic acid (24 uL, 0.30 mmol). After stirring for thirty minutes, the mixture was quenched with saturated sodium hydrogencarbonate (5 mL). Then the mixture was extracted with ethyl acetate (10 mL×3), the combined organic layer was washed with brine, dried over sodium sulfate, and concentrated in *vacuo*. The residue was purified by flash chromatography to give the 2-benzyl-1*H*-indole **5** (30 mg, 97% yield, pale yellow solid, known compound,[6] R_f = 0.70 (hexanes/ethyl acetate 20/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (brs, 1H), 7.53 (d, J = 7.6 Hz, 1H), 7.34-7.28 (m, 2H), 7.26-7.19 (m, 4H), 7.12-7.03 (m, 2H), 6.30 (d, J = 0.6 Hz, 1H), 4.09 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 137.8, 136.3, 128.9, 128.8, 128.7, 126.8, 121.4, 120.0, 119.8, 110.5, 101.2, 34.8.



5 **3a**

Ligand (*R*)-H8-BINAP (4.8 mg, 0.0076 mmol) and $Pd(OCOCF_3)_2$ (2.1 mg, 0.0063 mmol) were placed in a dried Schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone was added. The mixture was stirred at room temperature for one hour. The solvent was removed under vacuum to give the catalyst. This catalyst was taken into a glove box filled with nitrogen and dissolved in 2,2,2-trifluoroethanol (2.0 mL). To the mixture of compound **5** (51.8 mg, 0.25 mmol) and ethanesulfonic acid (41 μL, 0.50 mmol) in toluene (2 mL), this catalyst solution was added, and then the mixture was transferred to an autoclave, which was charged hydrogen gas (300 psi). The autoclave was stirred at 40 °C for 24 h. After release of the hydrogen, the autoclave was opened and the reaction mixture was evaporated. Purification was performed on silica gel using hexanes/ethyl acetate (20:1) as the eluent to give chiral products **3a** (51 mg, 98% yield, 95% ee).

5. The Scale-up Experiment



2a **3a**

Ligand (*R*)-H8-BINAP (47.9 mg, 0.076 mmol) and $Pd(OCOCF_3)_2$ (20.9 mg, 0.063 mmol) were placed in a dried Schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone was added. The mixture was stirred at room temperature for one hour. The solvent was removed under vacuum to give the catalyst. This catalyst was taken into a glove box filled with nitrogen and dissolved in 2,2,2-trifluoroethanol (10 mL). To the mixture of compound **2a** (814 mg, 2.5 mmol) and ethanesulfonic acid (0.41 mL, 5 mmol,) in toluene (10 mL), this catalyst solution was added, and then the mixture was transferred to an autoclave, which was charged hydrogen gas (300 psi). The autoclave was stirred at 40 °C for 24 h. After release of the hydrogen, the autoclave was opened and the reaction mixture was evaporated. Then, saturated sodium hydrogencarbonate (10 mL) was added. The mixture was extracted with dichloromethane (10 mL×3), and the combined organic layer was dried over anhydrous sodium sulfate and concentrated in *vacuo*. Purification was performed on silica gel using ethyl acetate/hexanes (10:1) as the eluent to give chiral product **3a** (477 mg, 91% yield, 94% ee).

6. References

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4. Wang, D.-S.; Chen, Q.-A.; Li, W.; Yu, C.-B.; Zhou, Y.-G.; Zhang, X. *J. Am. Chem. Soc.* **2010**, *132*, 8909.
5. Wen, J.; Fan, X.; Tan, R.; Chien, H.-C.; Zhou, Q.; Chung, L. W.; Zhang, X. *Org. Lett.* **2018**, *20*, 2143.
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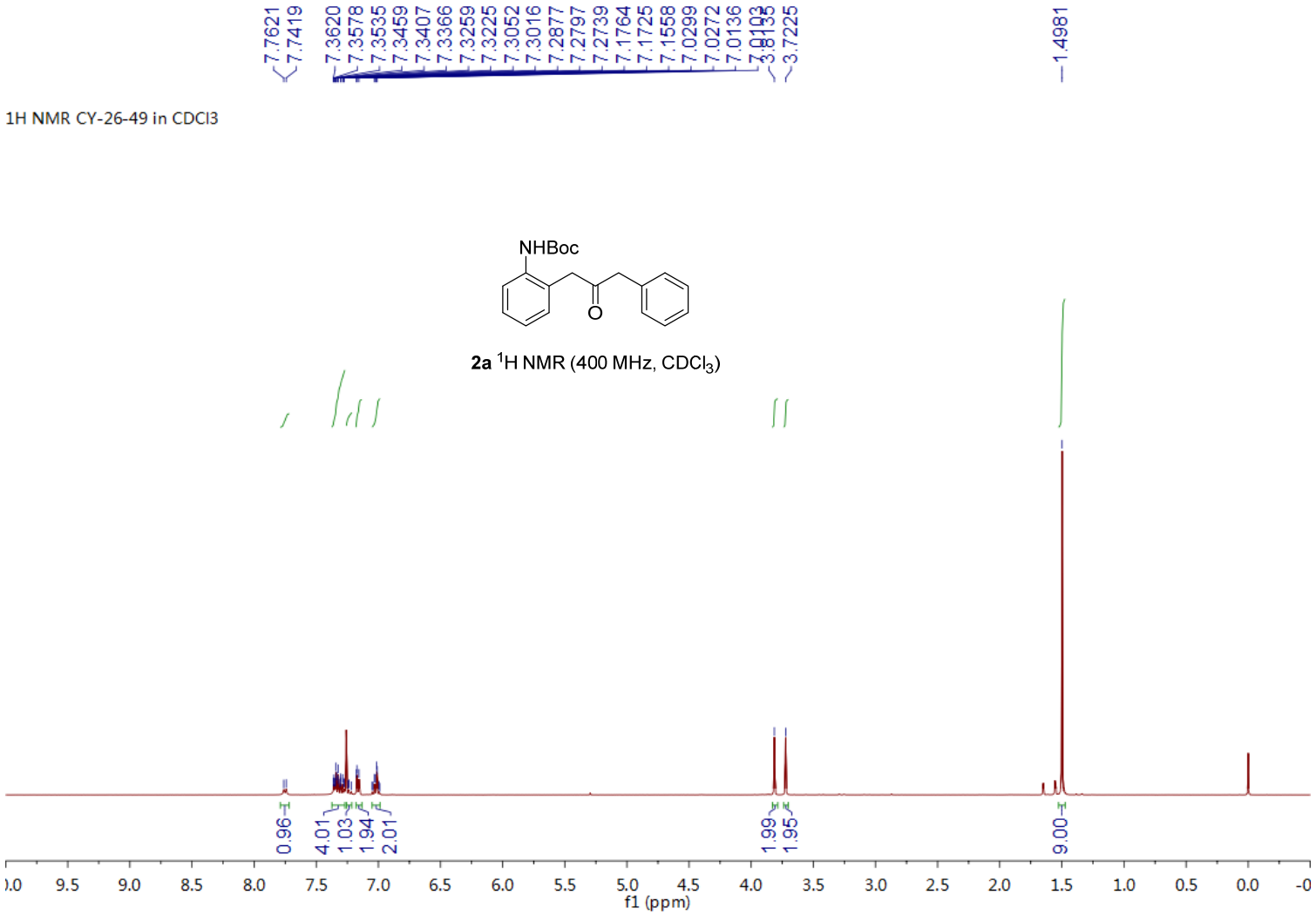
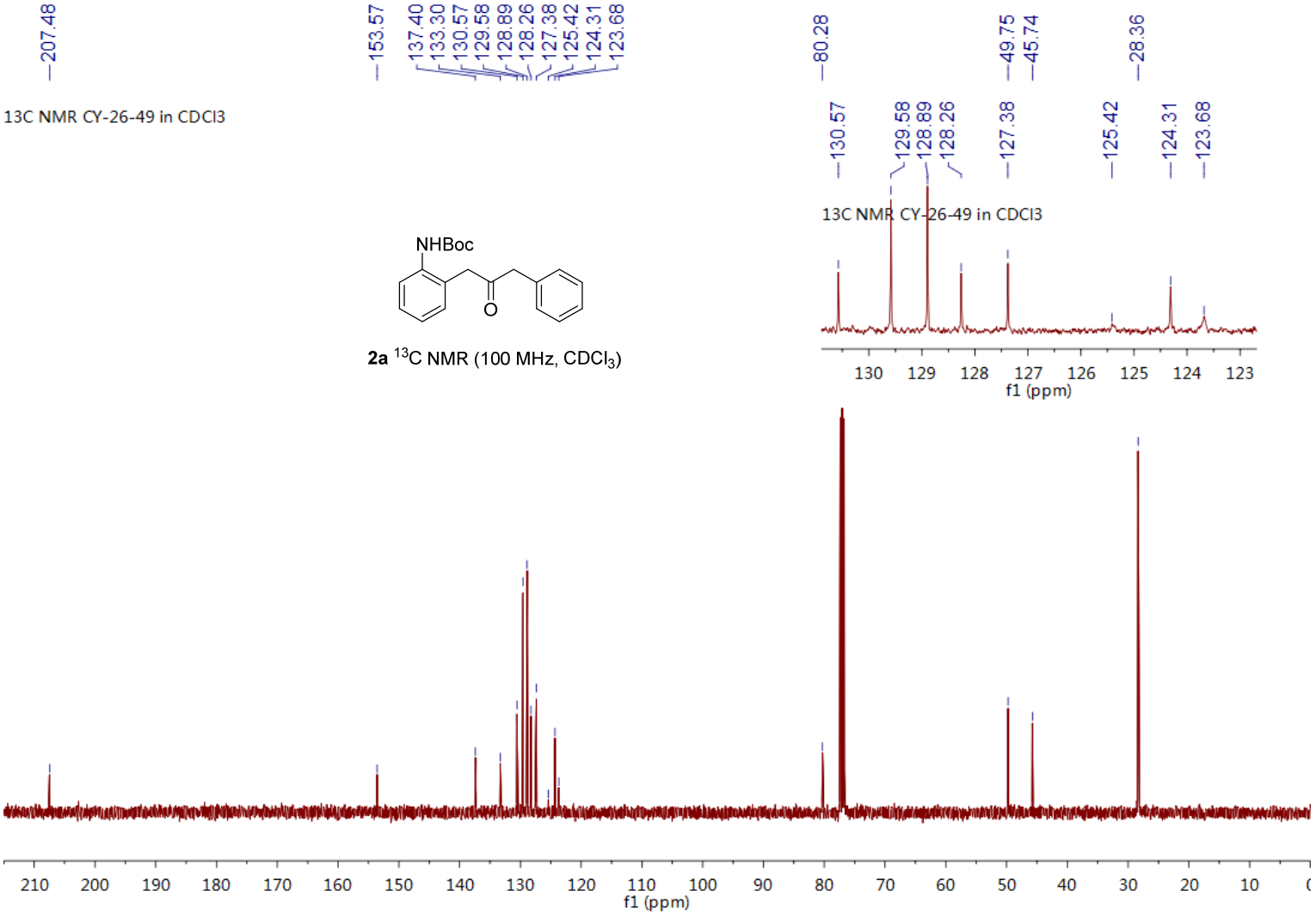
8. Saito, K.; Shibata, Y.; Yamanaka, M.; Akiyama, T. *J. Am. Chem. Soc.* **2013**, *135*, 11740.

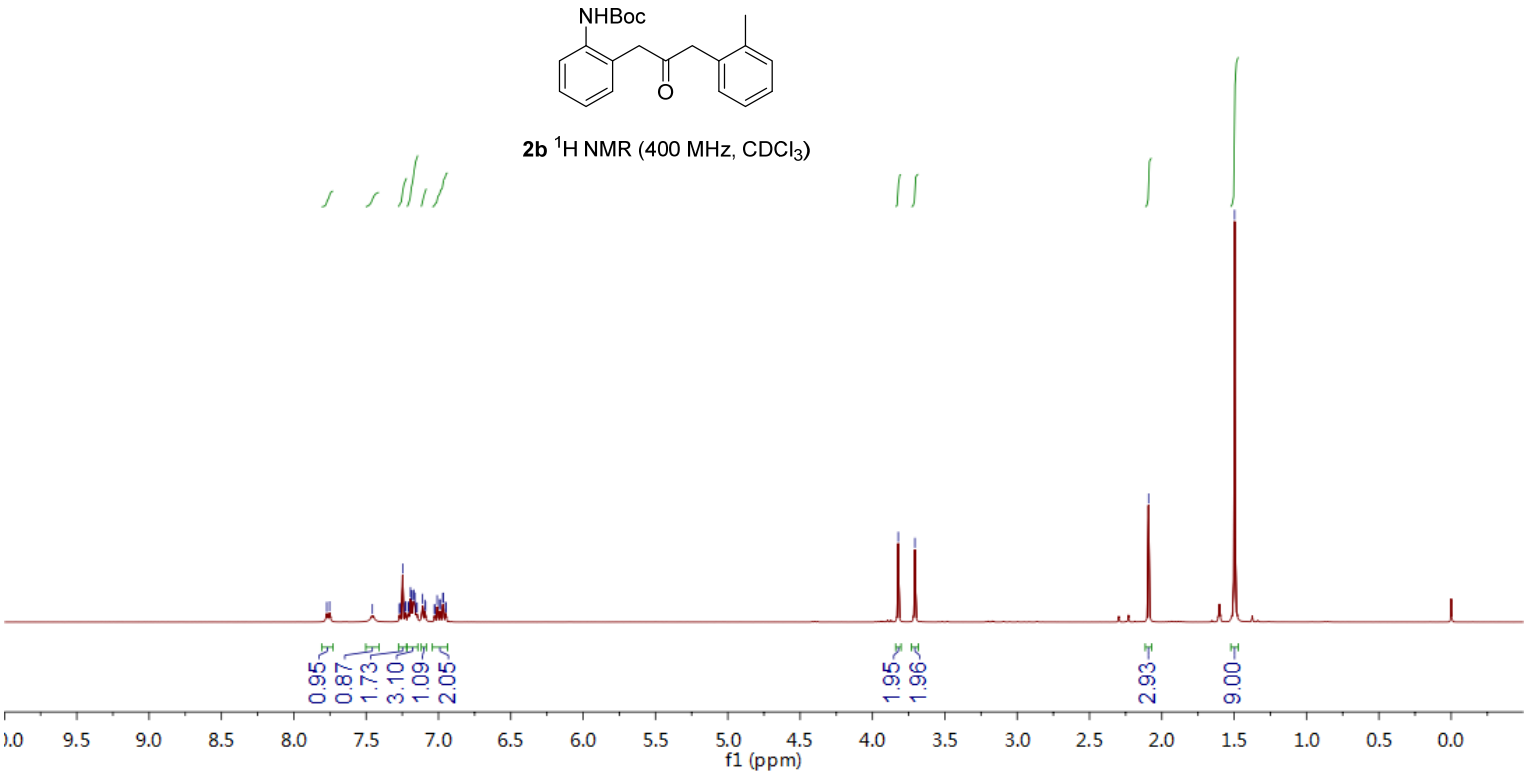
7. Copy of NMR, HPLC for Compounds



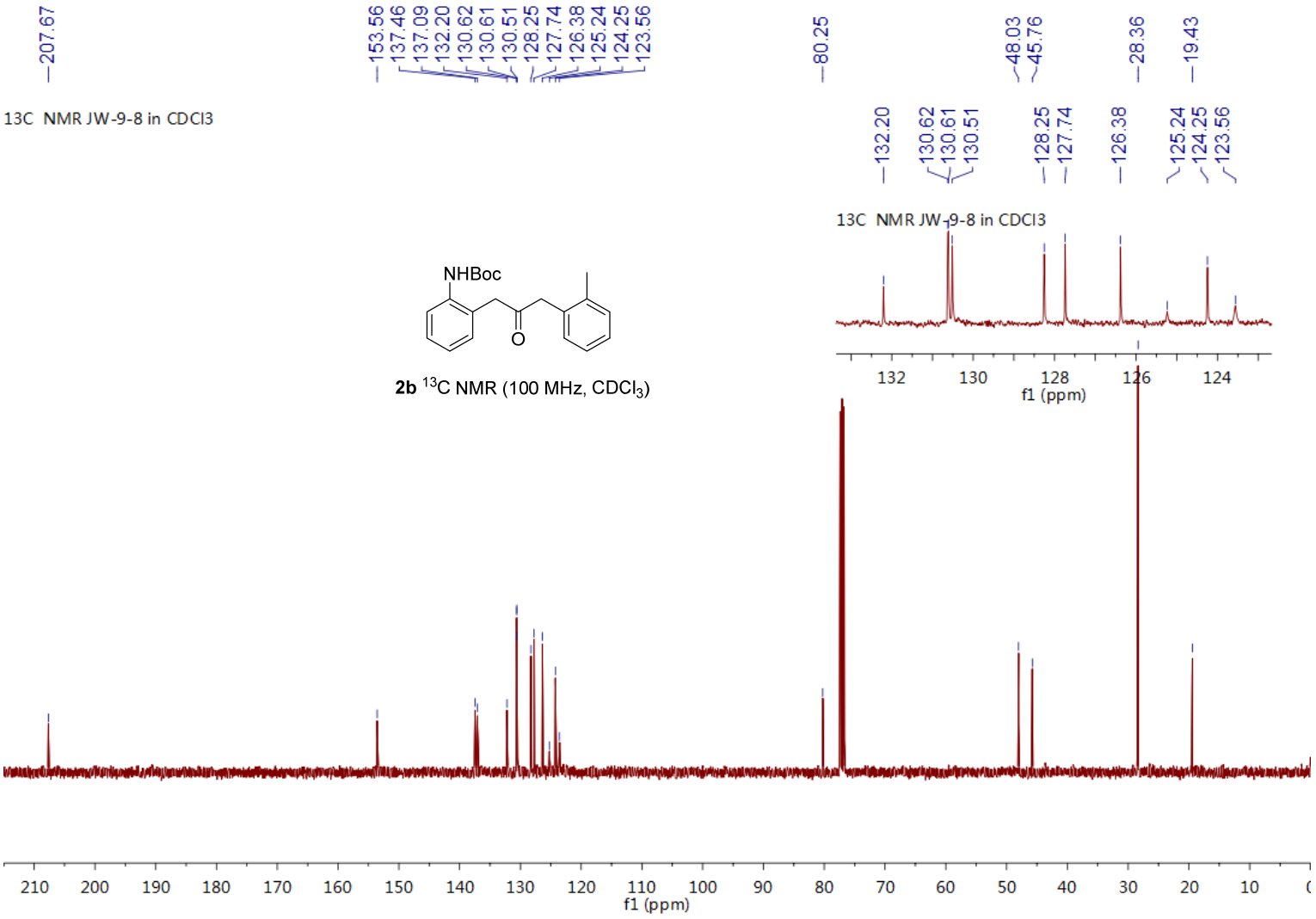


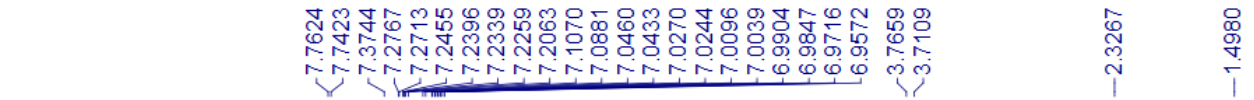
2a ^{13}C NMR (100 MHz, $CDCl_3$)

1H NMR JW-9-8 in CDCl3

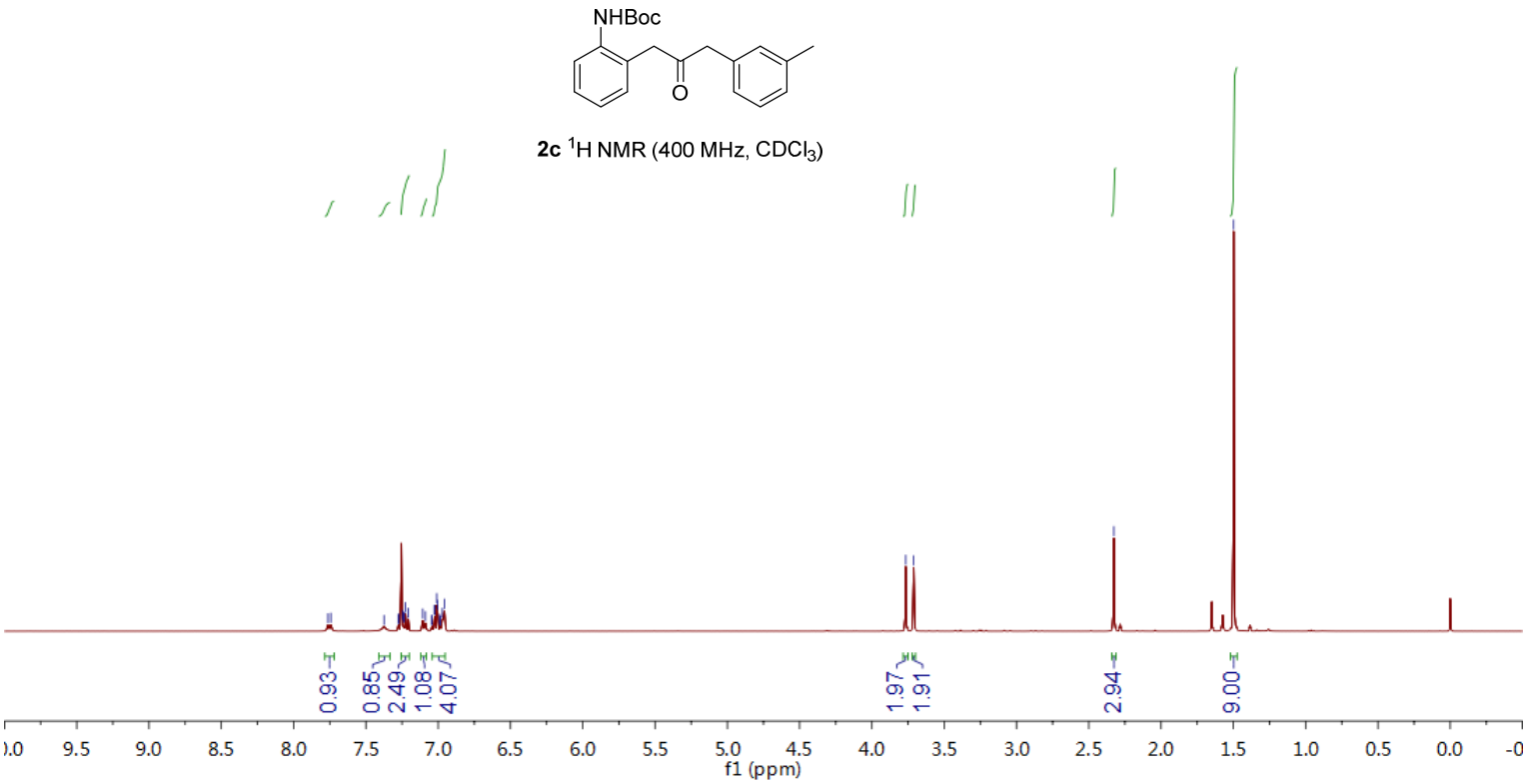


2b ^{1}H NMR (400 MHz, $CDCl_3$)

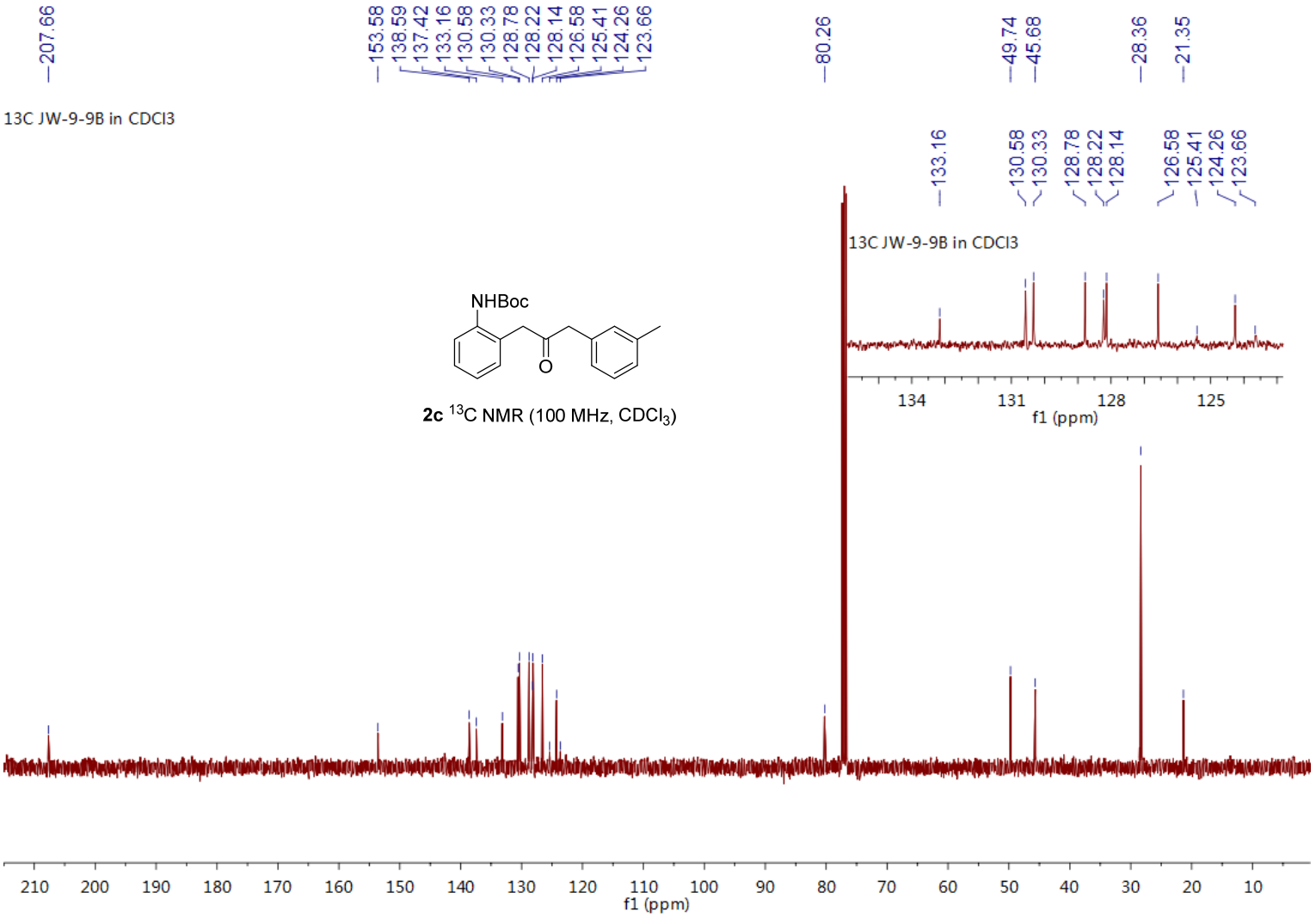

207.67
153.56
137.46
137.09
132.20
130.62
130.61
130.51
128.25
127.74
126.38
125.24
124.25
123.56
80.25
48.03
45.76
28.36
19.43
13C NMR JW-9-8 in CDCl3
NHBoc
O
2b 13C NMR (100 MHz, CDCl3)
132.20
130.62
130.61
130.51
128.25
127.74
126.38
125.24
124.25
123.56
13C NMR JW-9-8 in CDCl3
132
130
128
126
124
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



1H JW-9-9B in CDCl3



2c ^{1}H NMR (400 MHz, $CDCl_3$)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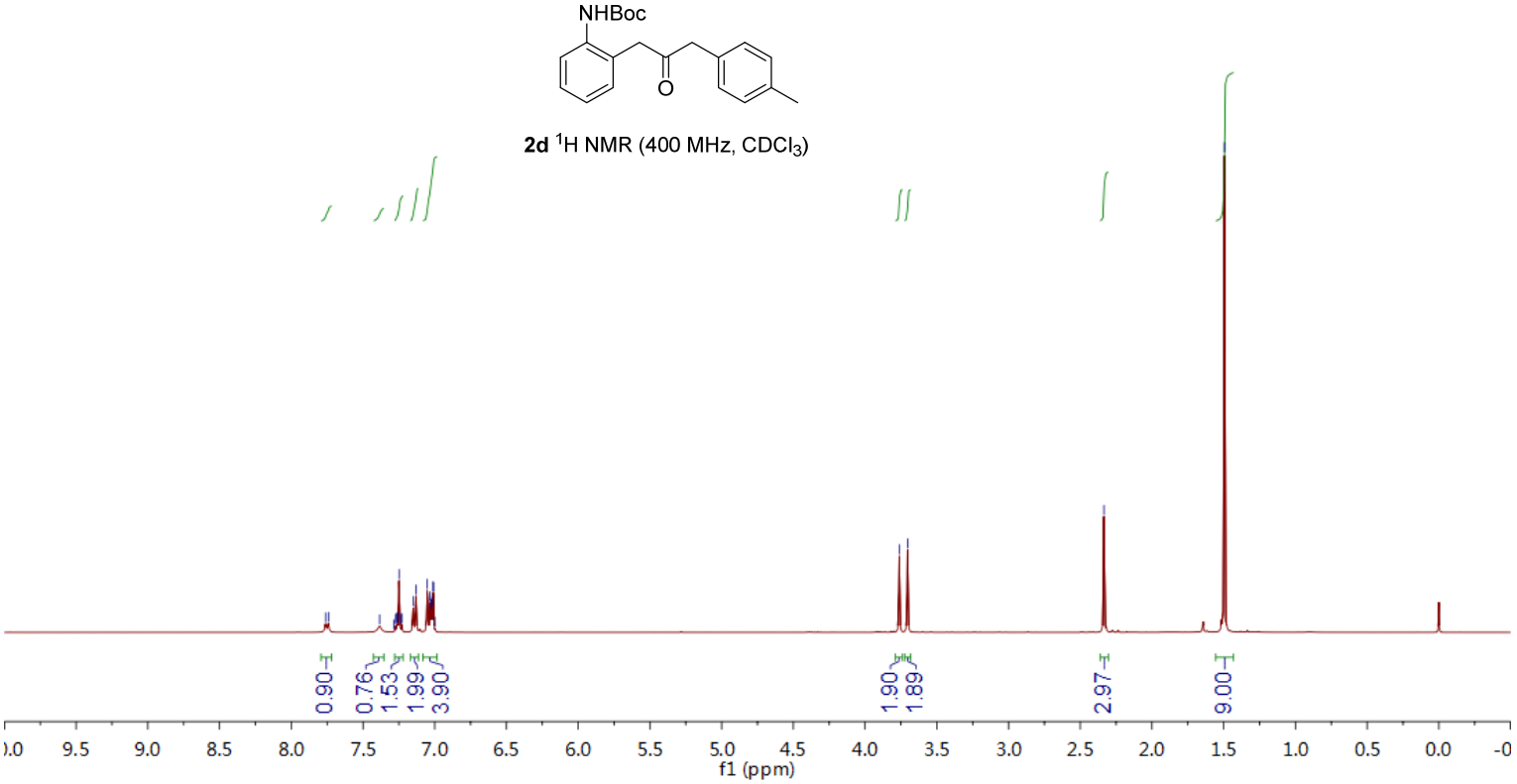
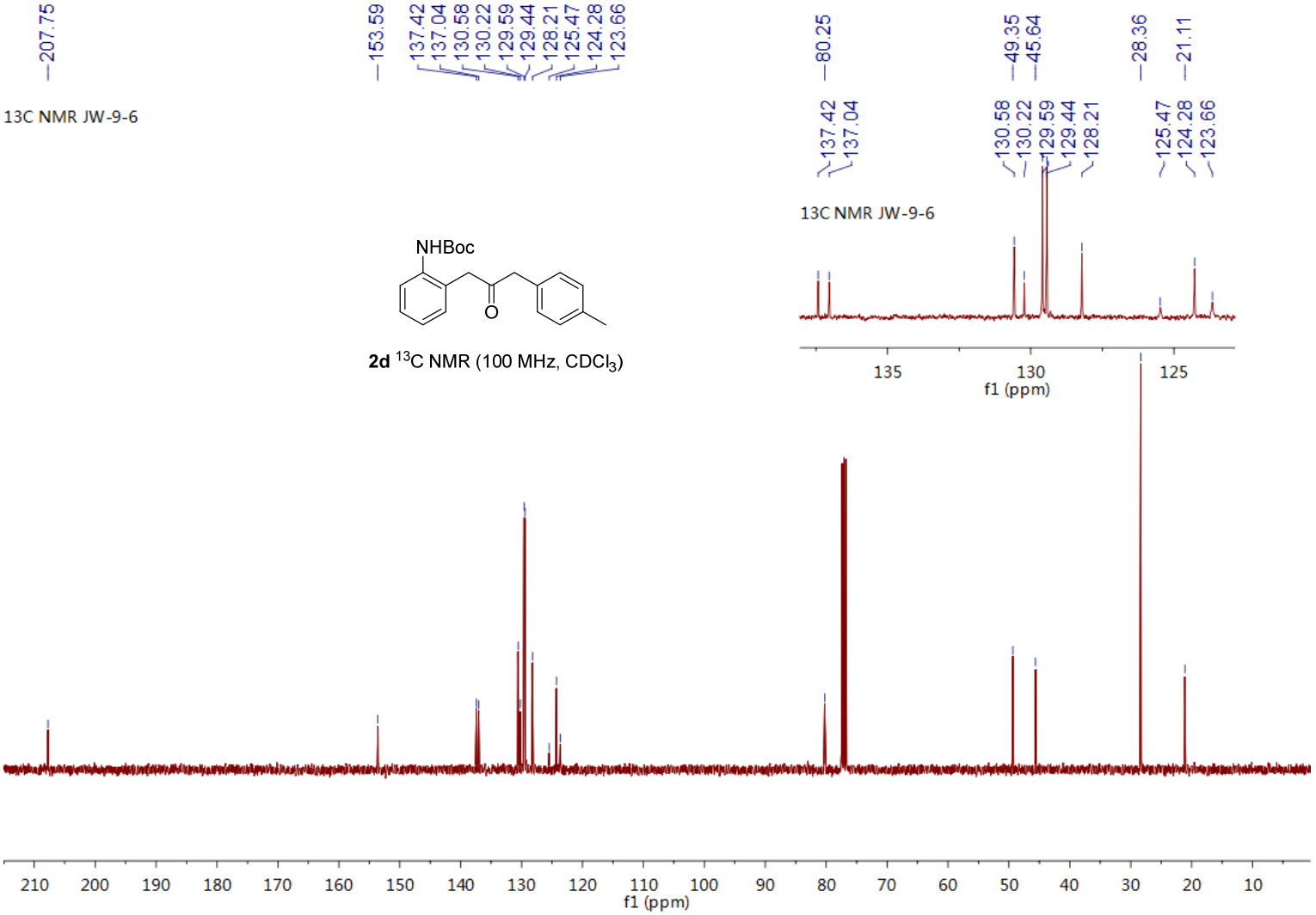
153.58
138.59
137.42
133.16
130.58
130.33
128.78
128.22
128.14
126.58
125.41
124.26
123.66
80.26
49.74
45.68
28.36
21.35
13C JW-9-9B in CDCl3
NHBoc
O
2c 13C NMR (100 MHz, CDCl3)
133.16
130.58
130.33
128.78
128.22
128.14
126.58
125.41
124.26
123.66
13C JW-9-9B in CDCl3
134
131
128
125
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

1H NMR JW-9-6 in CDCl3



2d ^{1}H NMR (400 MHz, $CDCl_3$)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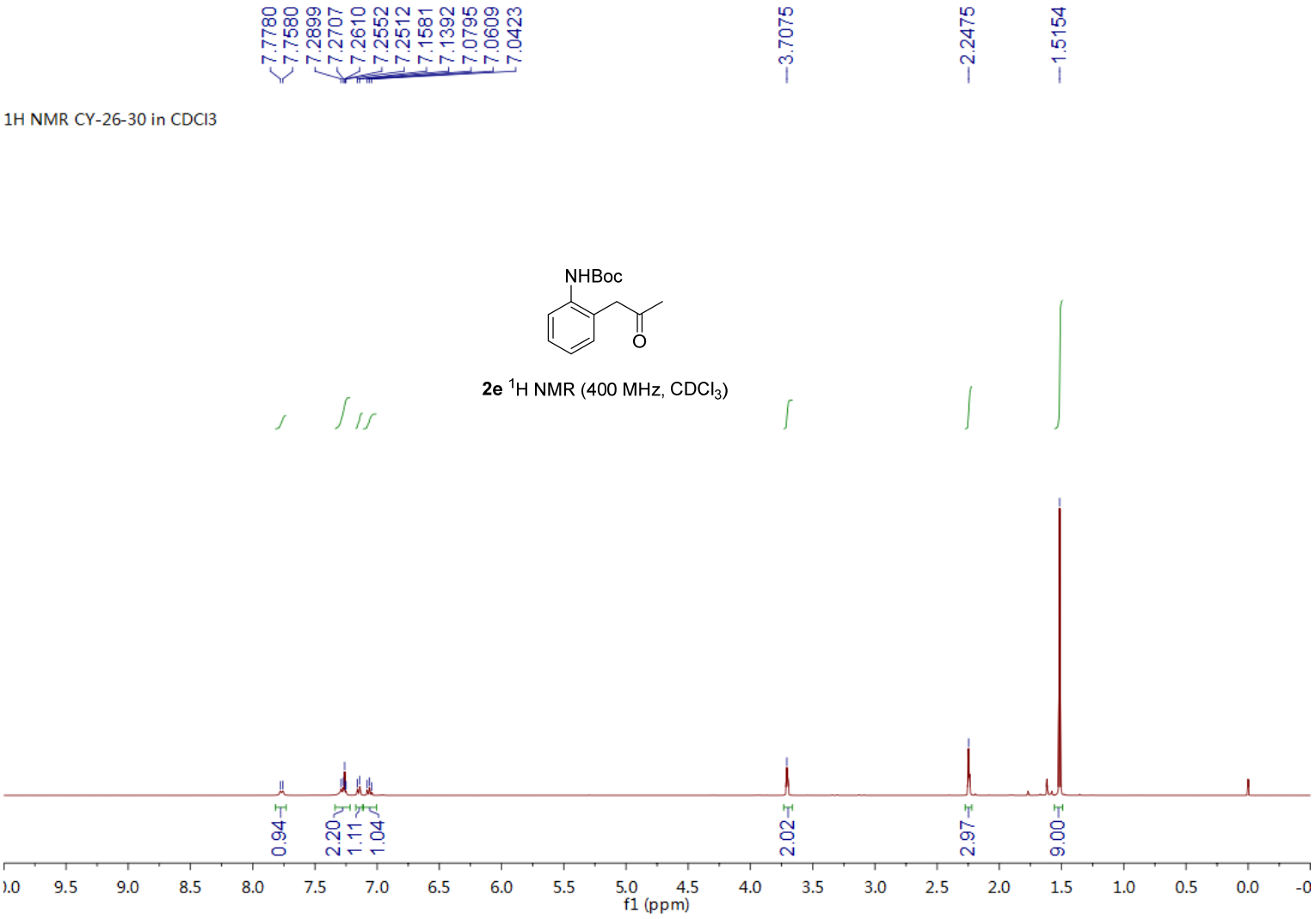
153.59
137.42
137.04
130.58
130.22
129.59
129.44
128.21
125.47
124.28
123.66
80.25
49.35
45.64
28.36
21.11
13C NMR JW-9-6
NHBoc
O
2d 13C NMR (100 MHz, CDCl3)
13C NMR JW-9-6
135
130
125
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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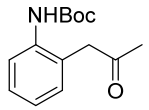
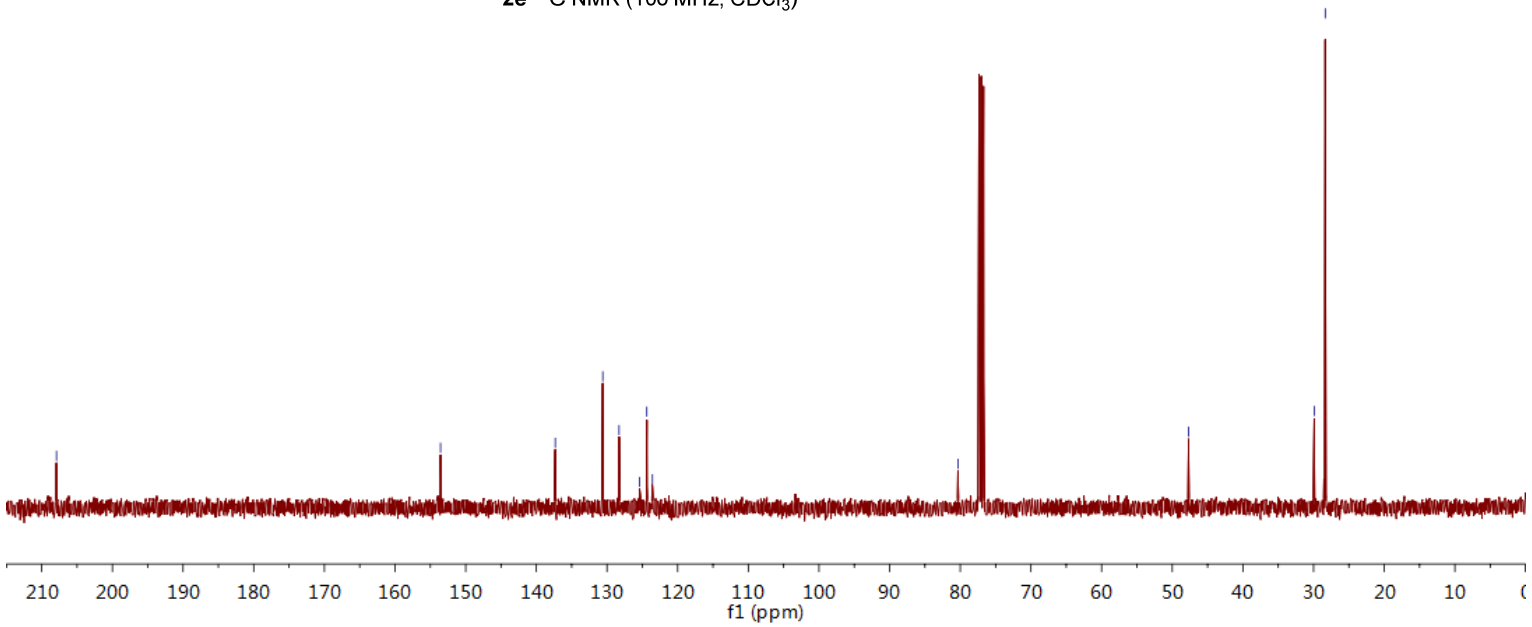
7.7580
7.2899
7.2707
7.2610
7.2552
7.2512
7.1581
7.1392
7.0795
7.0609
7.0423
3.7075
2.2475
1.5154
1H NMR CY-26-30 in CDCl3
NHBoc
O
2e 1H NMR (400 MHz, CDCl3)
0.94
2.20
1.11
1.04
2.02
2.97
9.00
f1 (ppm)

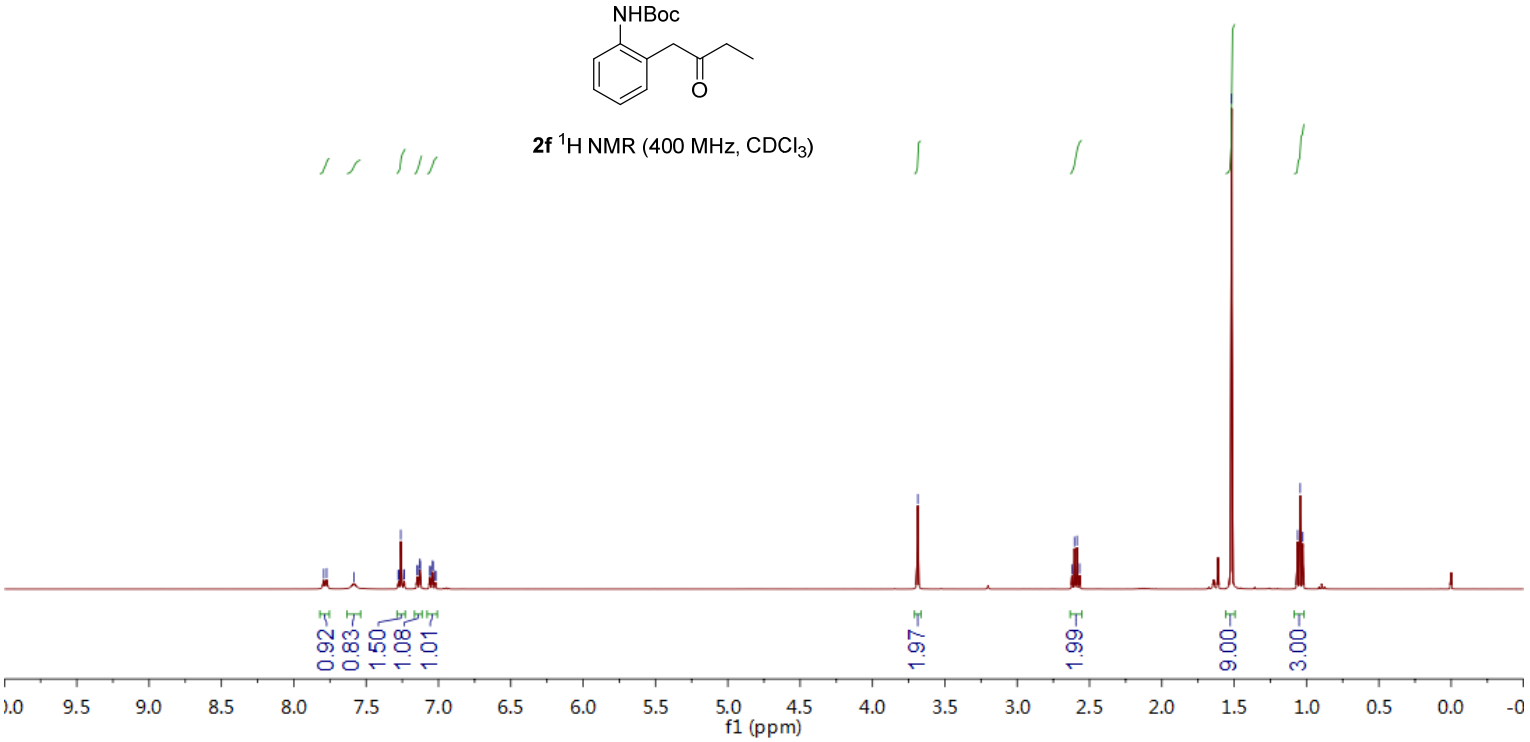
13C NMR CY-26-30 in CDCl3



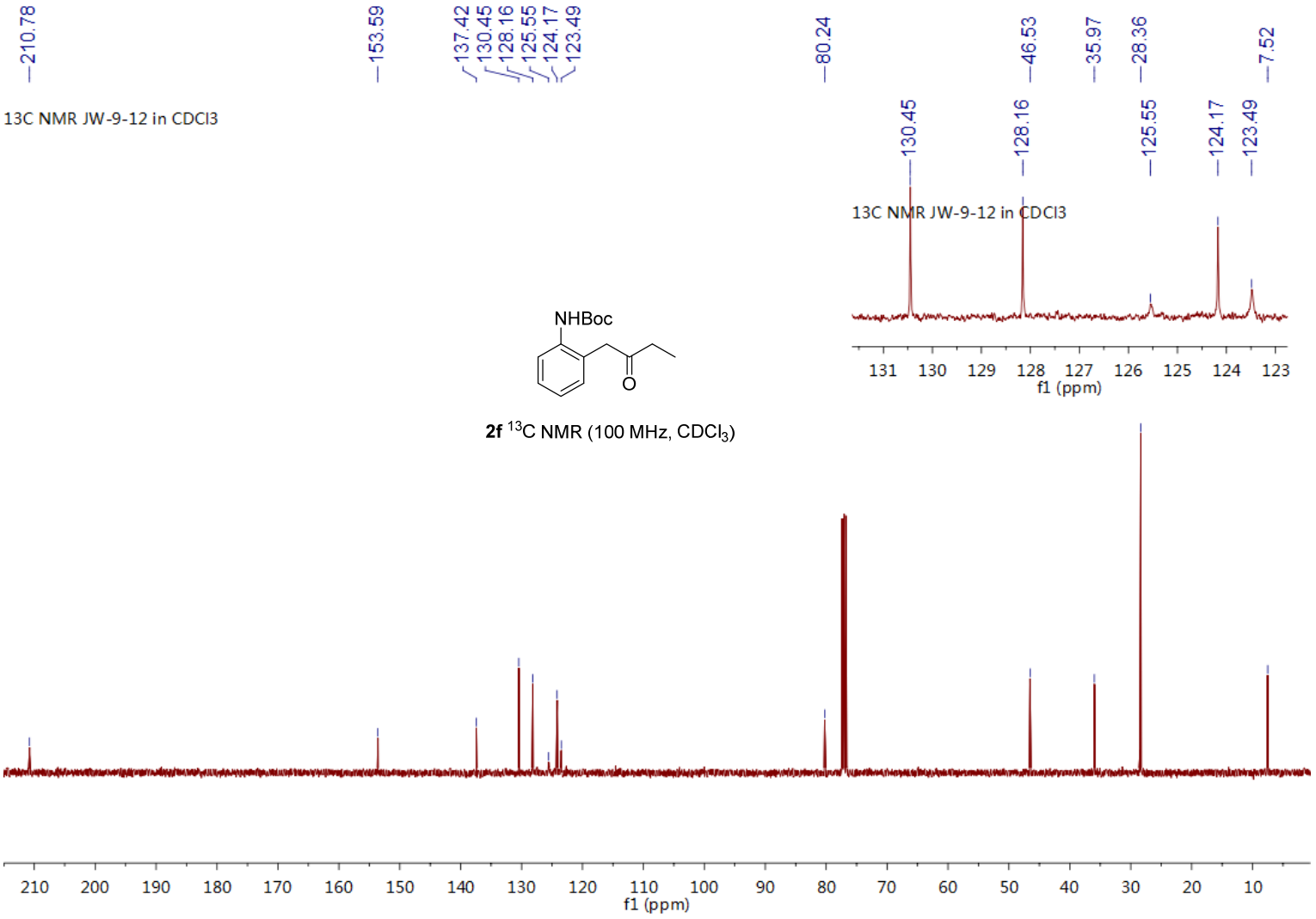
2e ^{13}C NMR (100 MHz, $CDCl_3$)



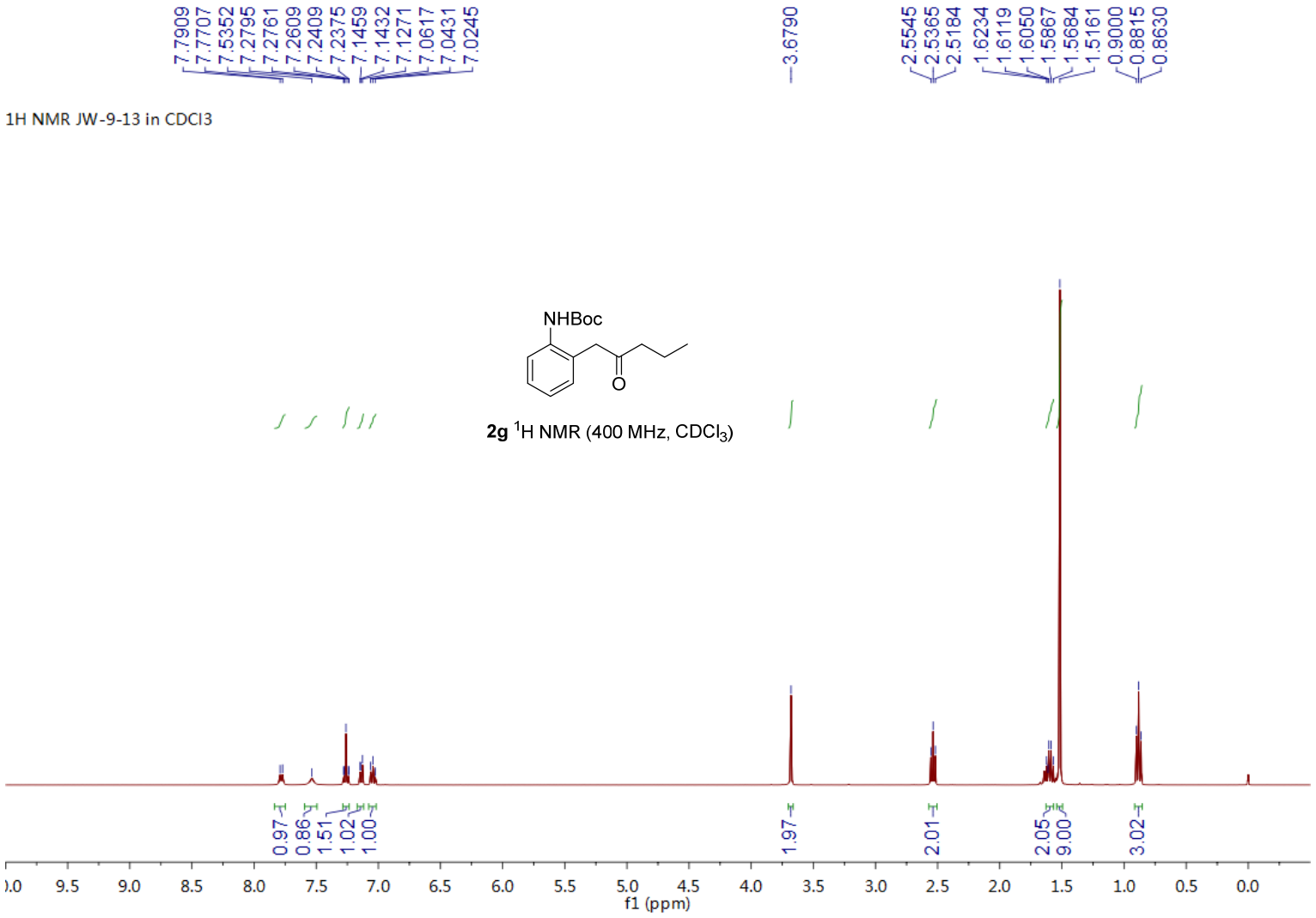
1H NMR JW-9-12 in CDCl3



2f ^{1}H NMR (400 MHz, $CDCl_3$)



2f ^{13}C NMR (100 MHz, $CDCl_3$)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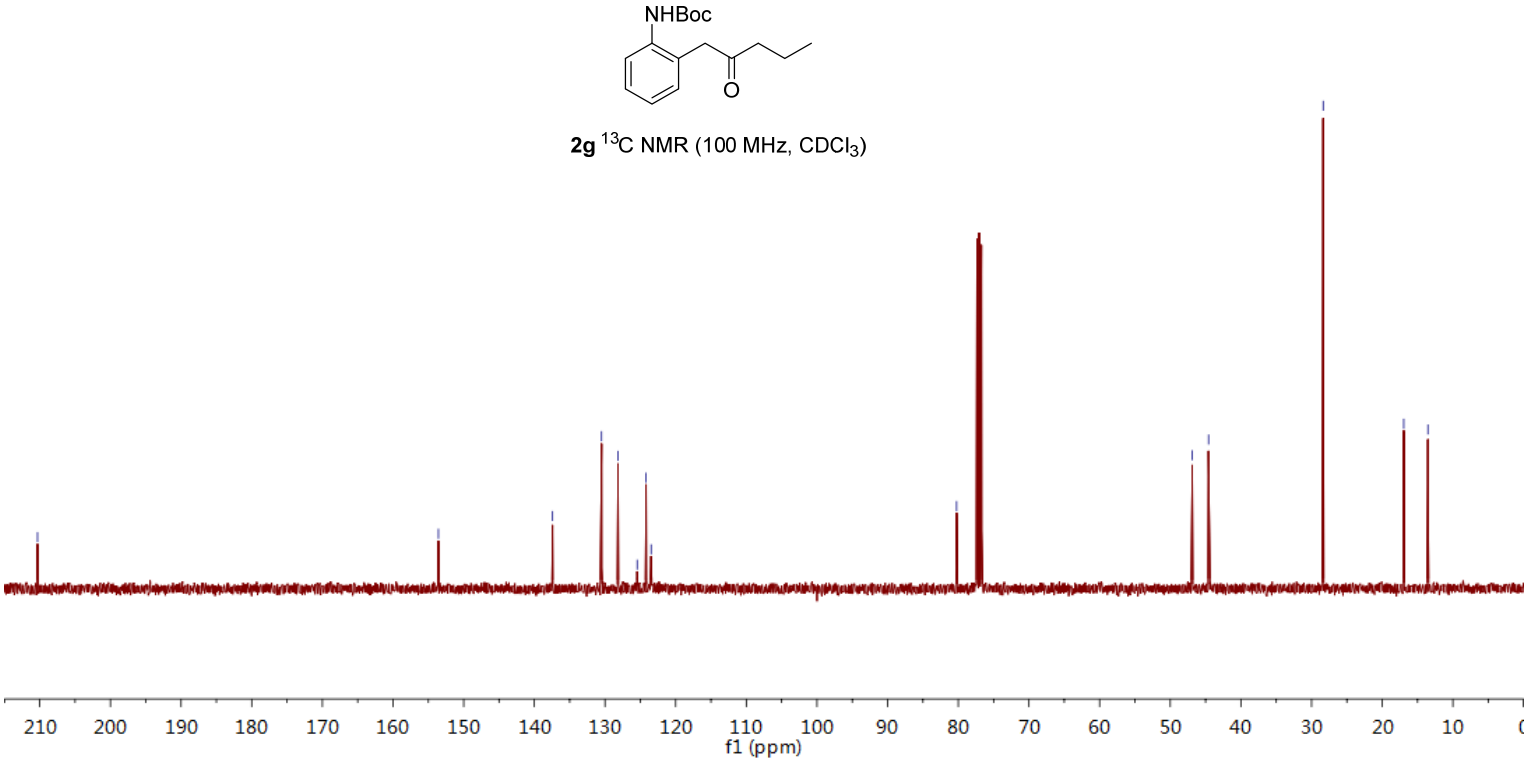
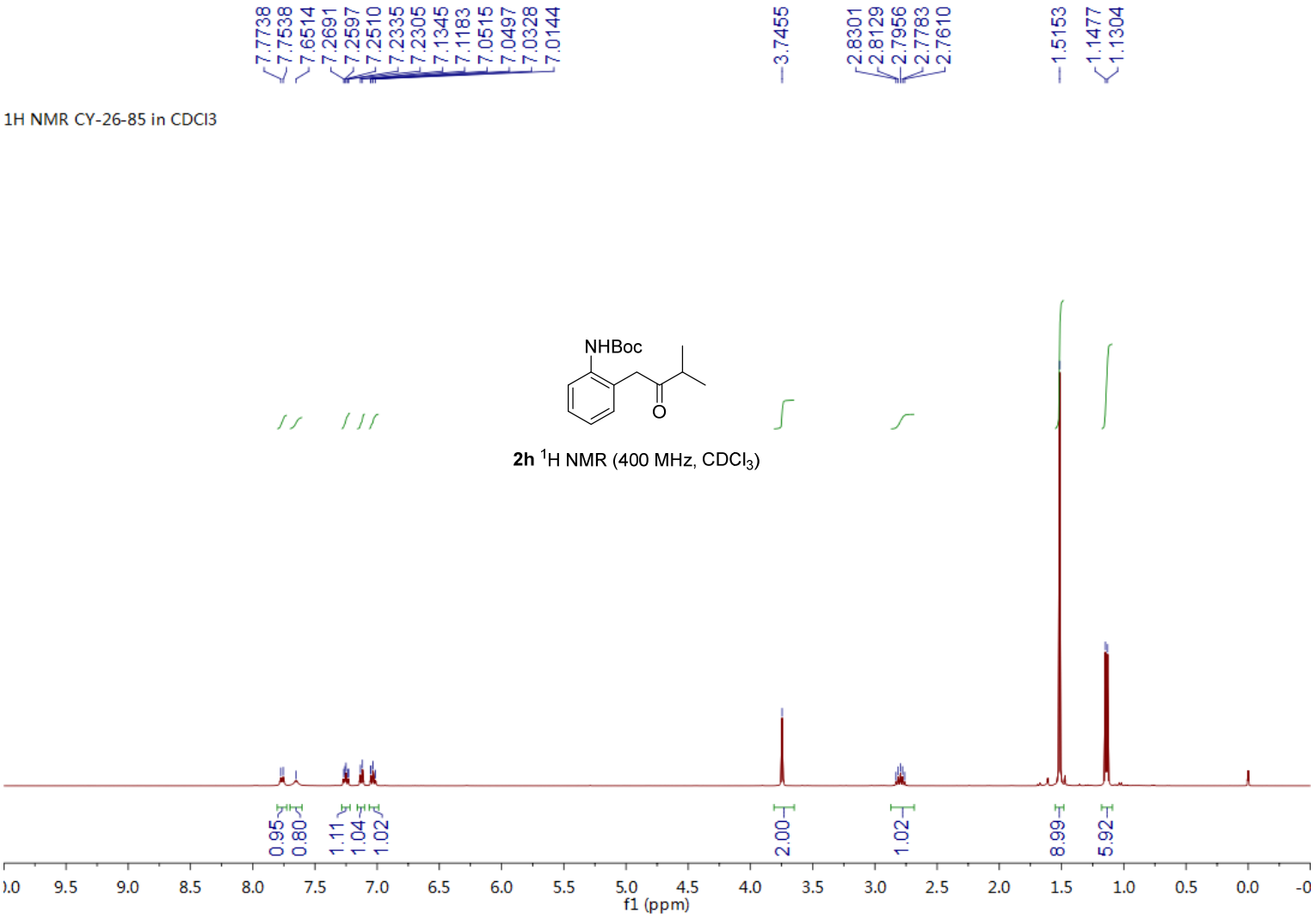
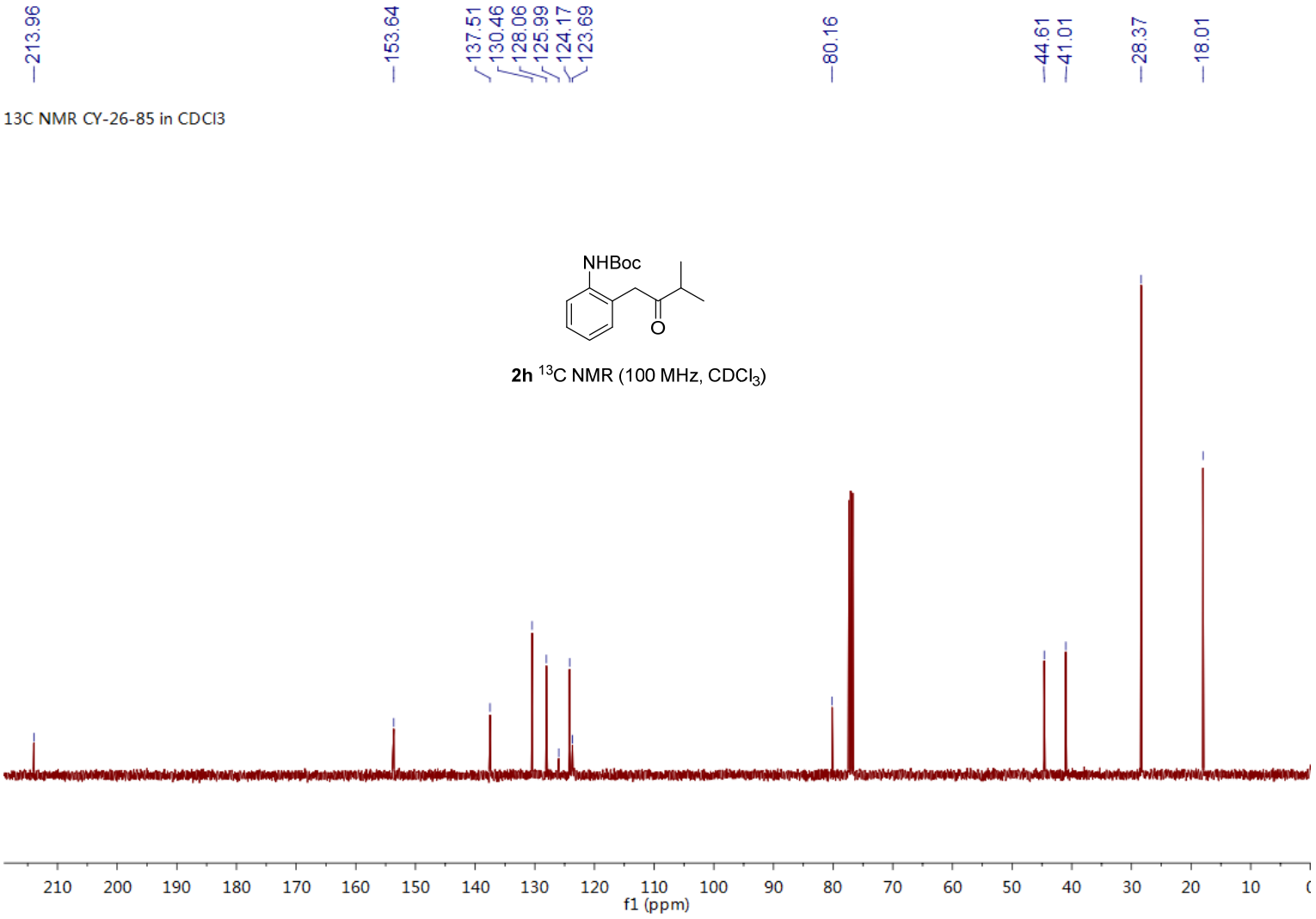
7.2795
7.2761
7.2609
7.2409
7.2375
7.1459
7.1432
7.1271
7.0617
7.0431
7.0245
3.6790
2.5545
2.5365
2.5184
1.6234
1.6119
1.6050
1.5867
1.5684
1.5161
0.9000
0.8815
0.8630
1H NMR JW-9-13 in CDCl3
NHBoc
O
2g 1H NMR (400 MHz, CDCl3)
0.97
0.86
1.51
1.02
1.00
1.97
2.01
2.05
9.00
3.02
f1 (ppm)

13C NMR JW-9-13 in CDCl3

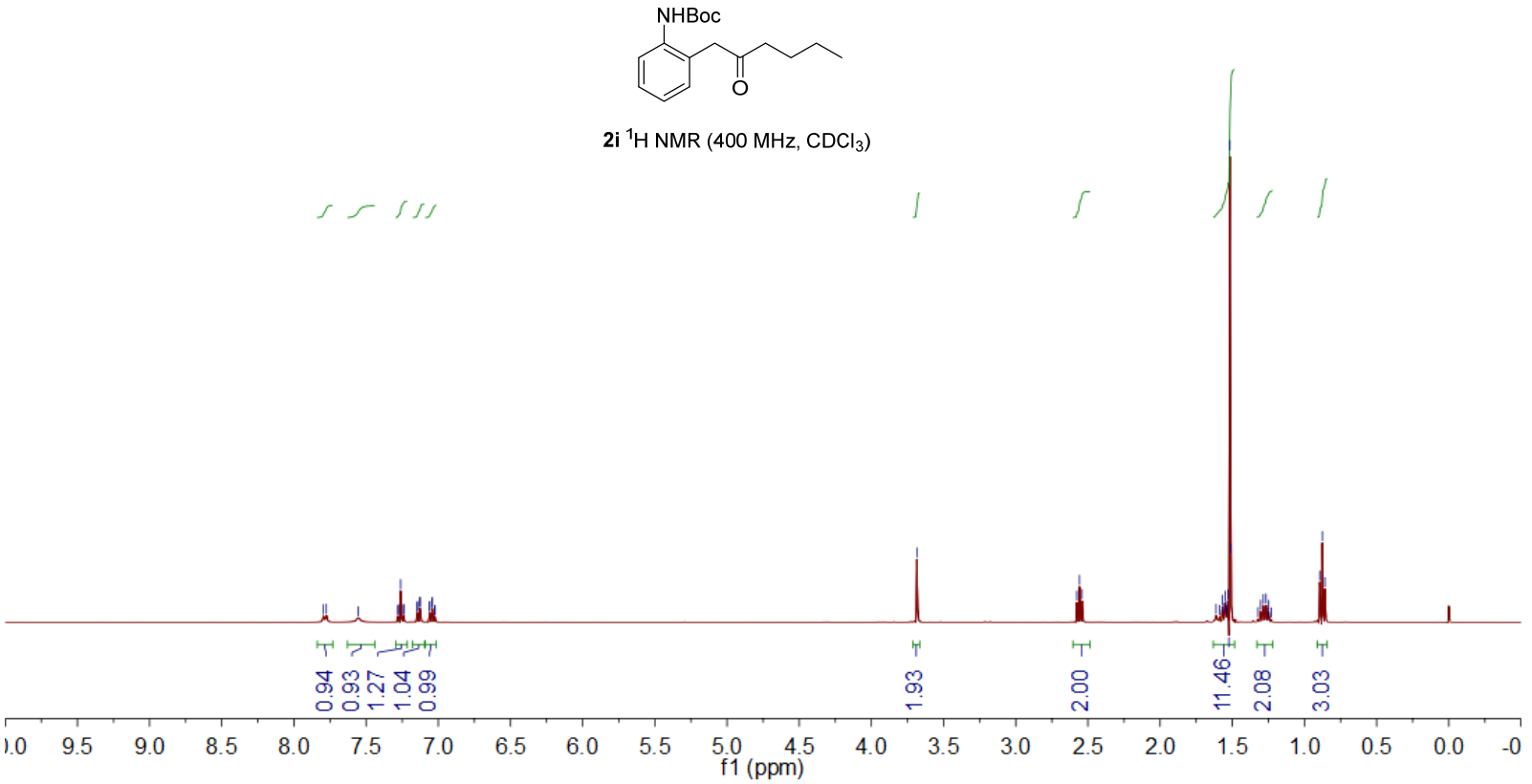


2g ^{13}C NMR (100 MHz, $CDCl_3$)

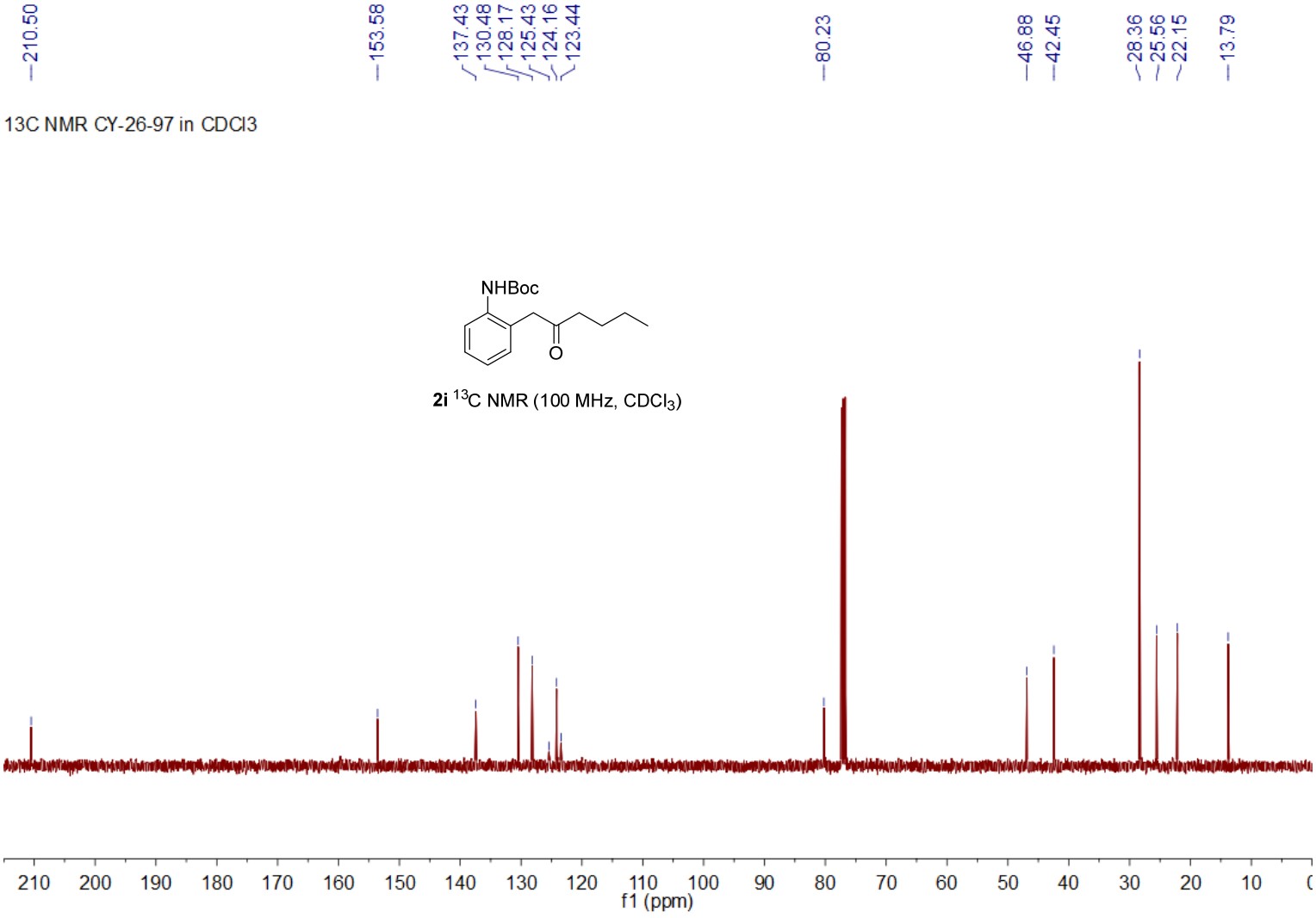

7.7738
7.7538
7.6514
7.2691
7.2597
7.2510
7.2335
7.2305
7.1345
7.1183
7.0515
7.0497
7.0328
7.0144
3.7455
2.8301
2.8129
2.7956
2.7783
2.7610
1.5153
1.1477
1.1304
1H NMR CY-26-85 in CDCl3
NHBoc
O
2h 1H NMR (400 MHz, CDCl3)
0.95
0.80
1.11
1.04
1.02
2.00
1.02
8.99
5.92
f1 (ppm)


213.96
153.64
137.51
130.46
128.06
125.99
124.17
123.69
80.16
44.61
41.01
28.37
18.01
13C NMR CY-26-85 in CDCl3
NHBoc
O
2h 13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

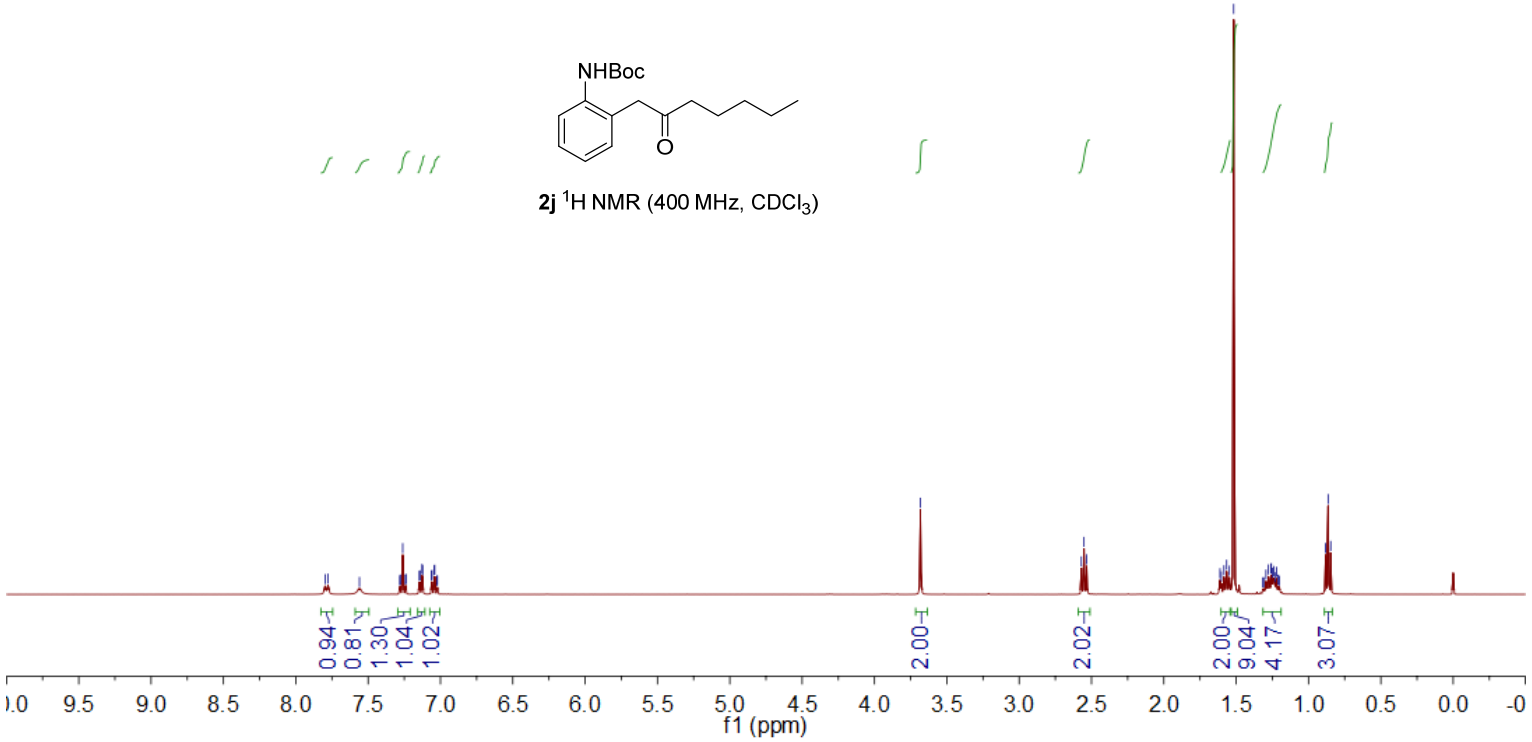
1H NMR CY-26-97 in CDCl3



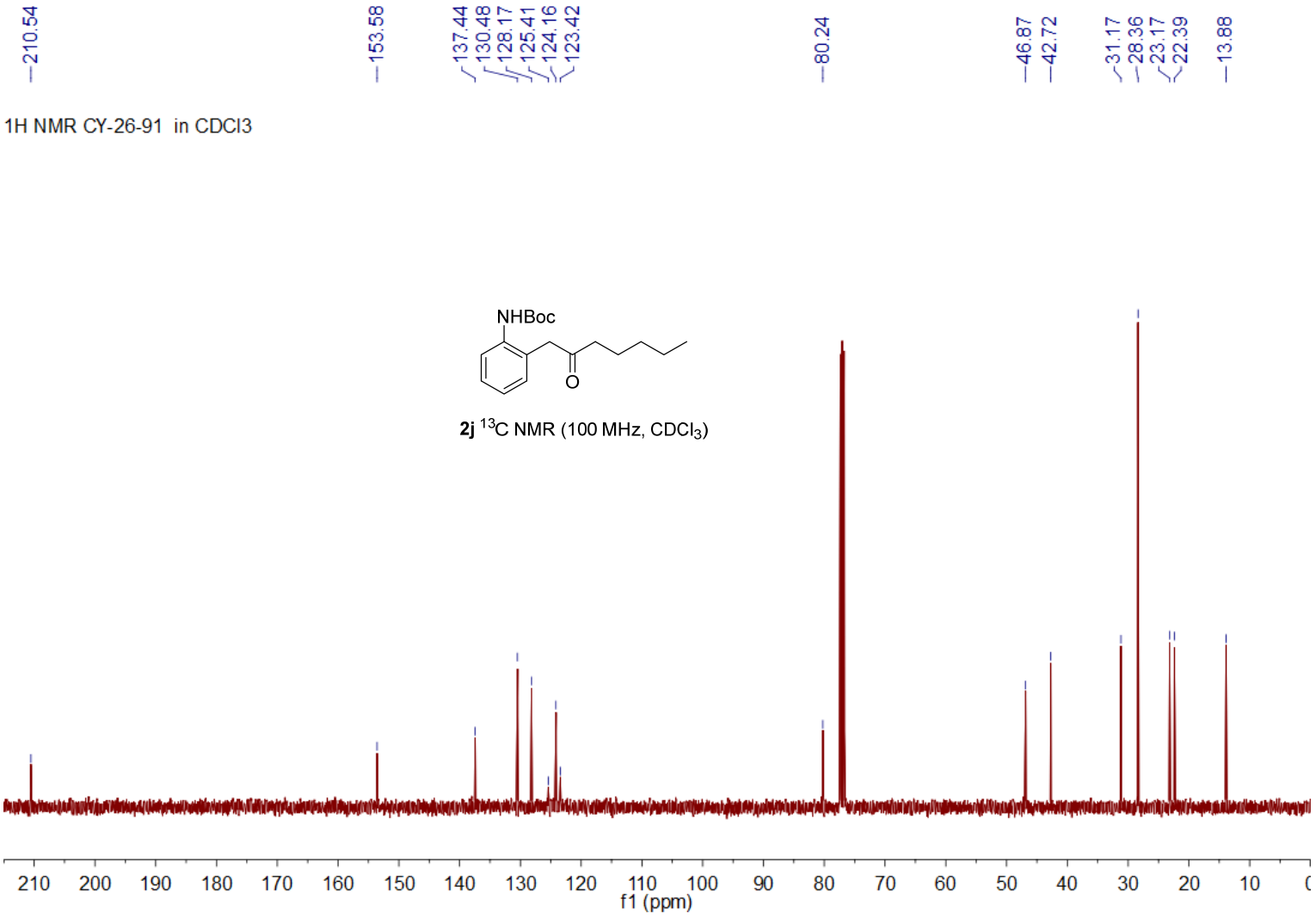
2i ^{1}H NMR (400 MHz, $CDCl_3$)

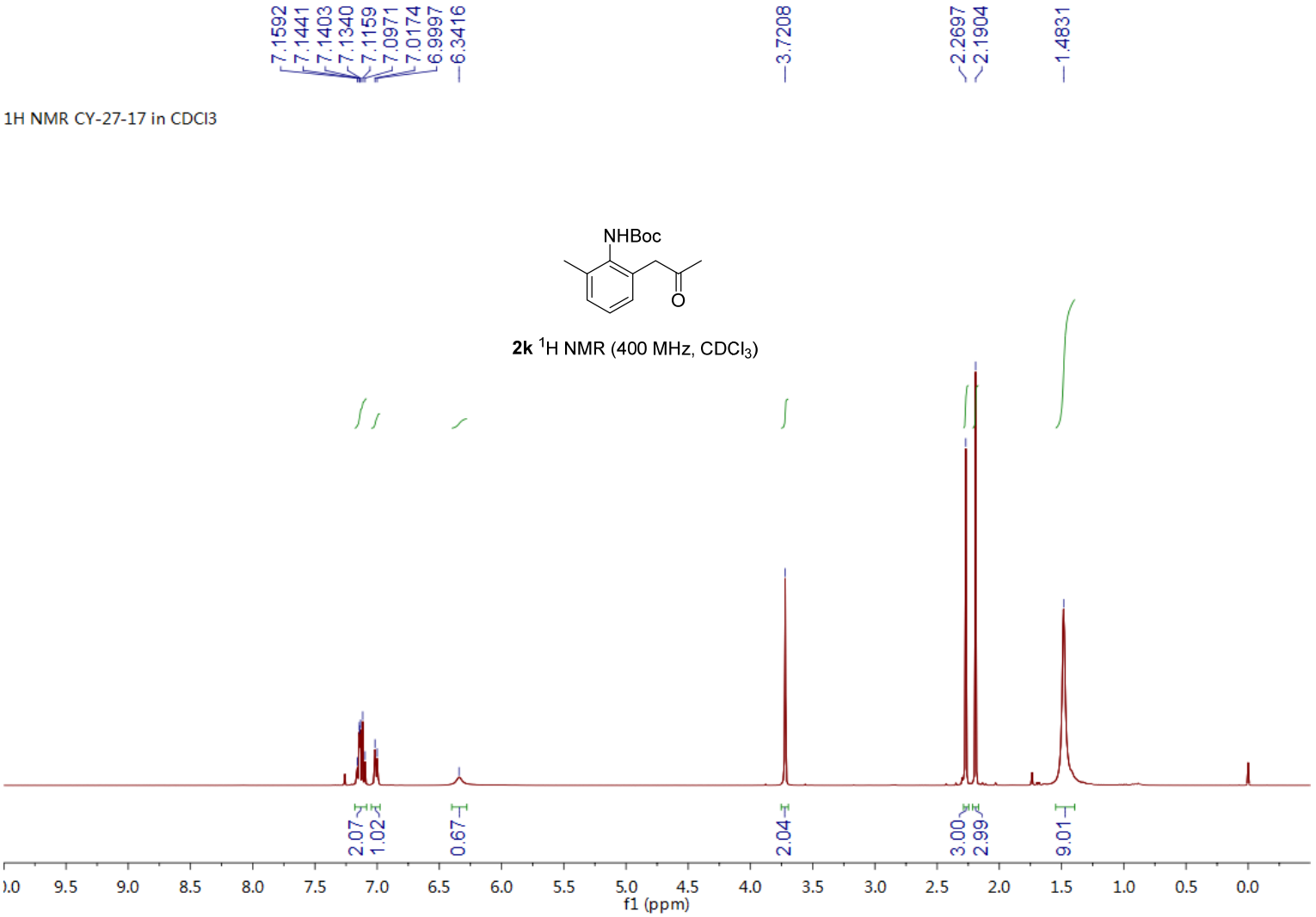

210.50
153.58
137.43
130.48
128.17
125.43
124.16
123.44
80.23
46.88
42.45
28.36
25.56
22.15
13.79
13C NMR CY-26-97 in CDCl3
NHBoc
O
2i 13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

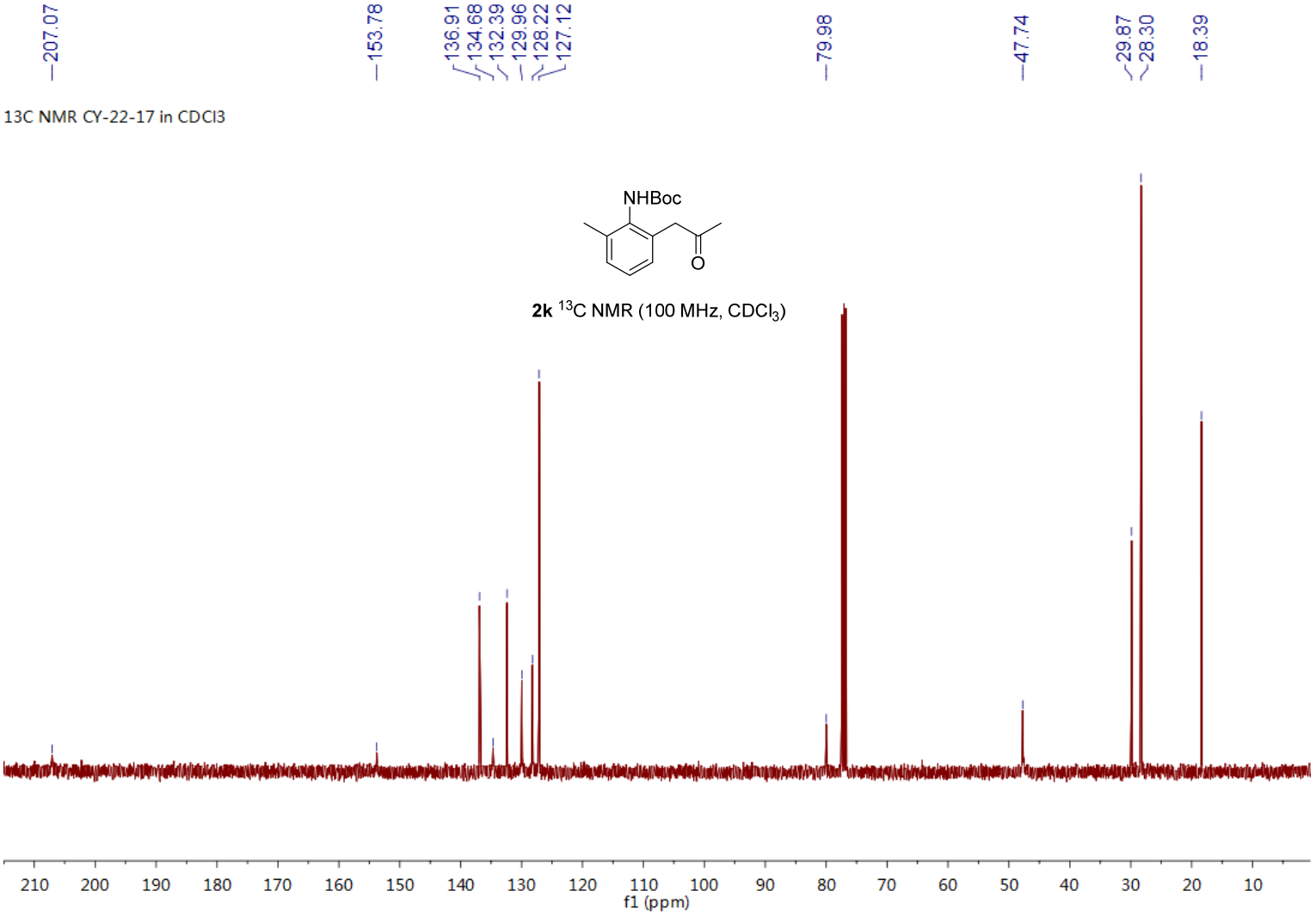
1H NMR CY-26-91 in CDCl3

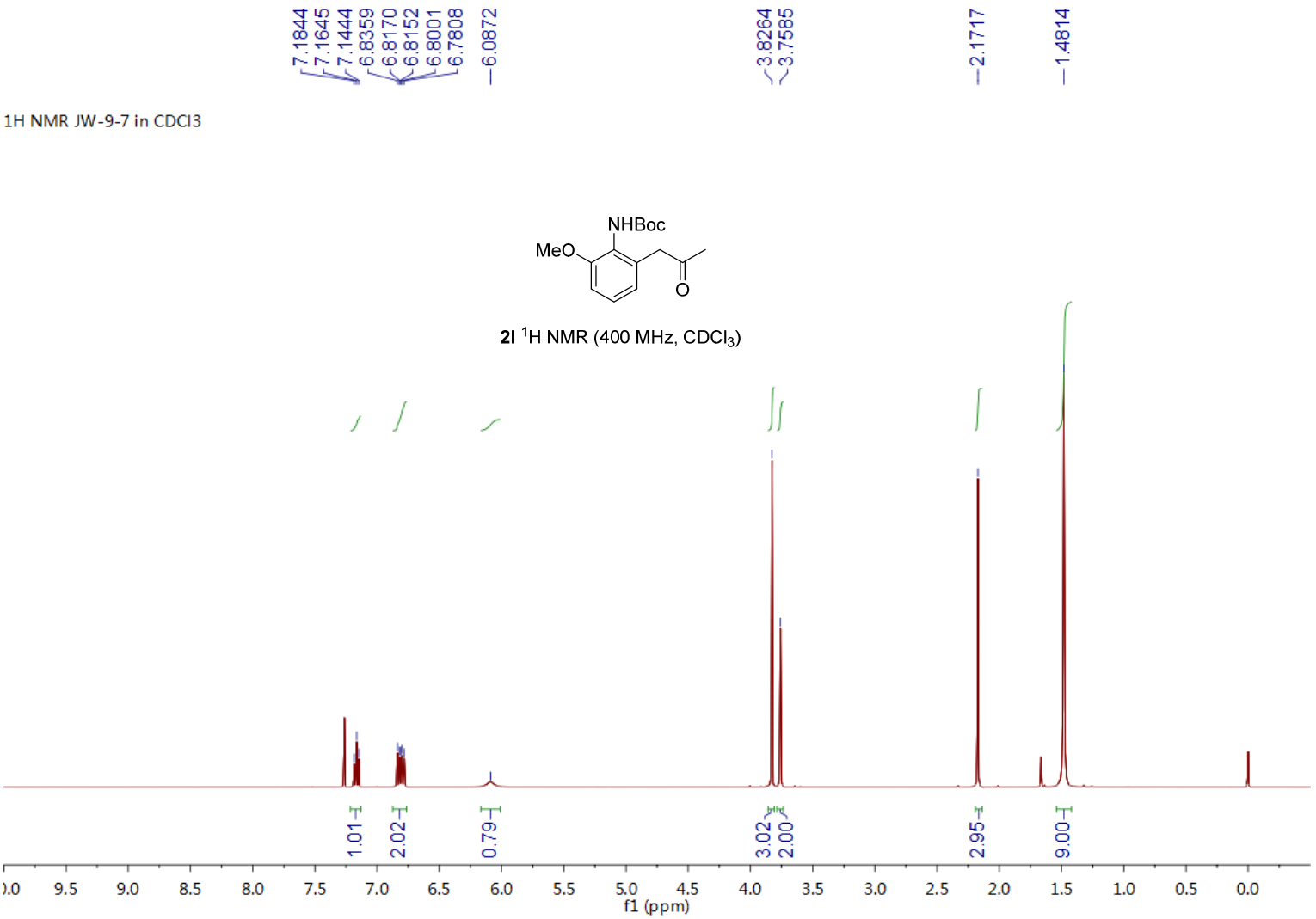


2j ^{1}H NMR (400 MHz, $CDCl_3$)


210.54
153.58
137.44
130.48
128.17
125.41
124.16
123.42
80.24
46.87
42.72
31.17
28.36
23.17
22.39
13.88
1H NMR CY-26-91 in CDCl3
NHBoc
O
2j ^{13}C NMR (100 MHz, $CDCl_3$)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.1592
7.1441
7.1403
7.1340
7.1159
7.0971
7.0174
6.9997
6.3416
3.7208
2.2697
2.1904
1.4831
1H NMR CY-27-17 in CDCl3
NHBoc
O
2k 1H NMR (400 MHz, CDCl3)
2.07
1.02
0.67
2.04
3.00
2.99
9.01
f1 (ppm)


207.07
153.78
136.91
134.68
132.39
129.96
128.22
127.12
79.98
47.74
29.87
28.30
18.39
13C NMR CY-22-17 in CDCl3
NHBoc
O
2k 13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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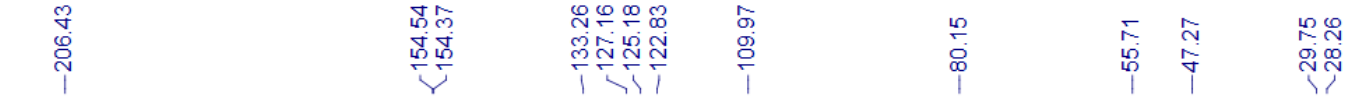
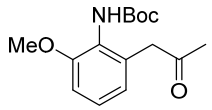
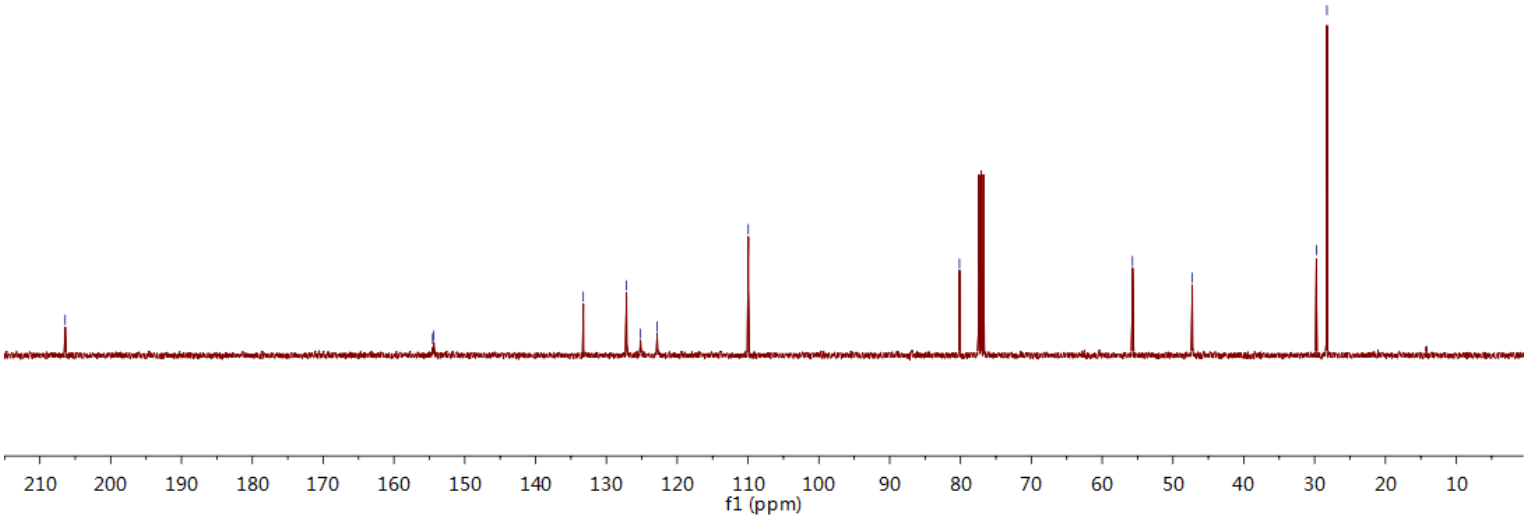
7.1645
7.1444
6.8359
6.8170
6.8152
6.8001
6.7808
6.0872
3.8264
3.7585
2.1717
1.4814
1H NMR JW-9-7 in CDCl3
NHBoc
MeO
O
2l 1H NMR (400 MHz, CDCl3)
1.01
2.02
0.79
3.02
2.00
2.95
9.00
f1 (ppm)

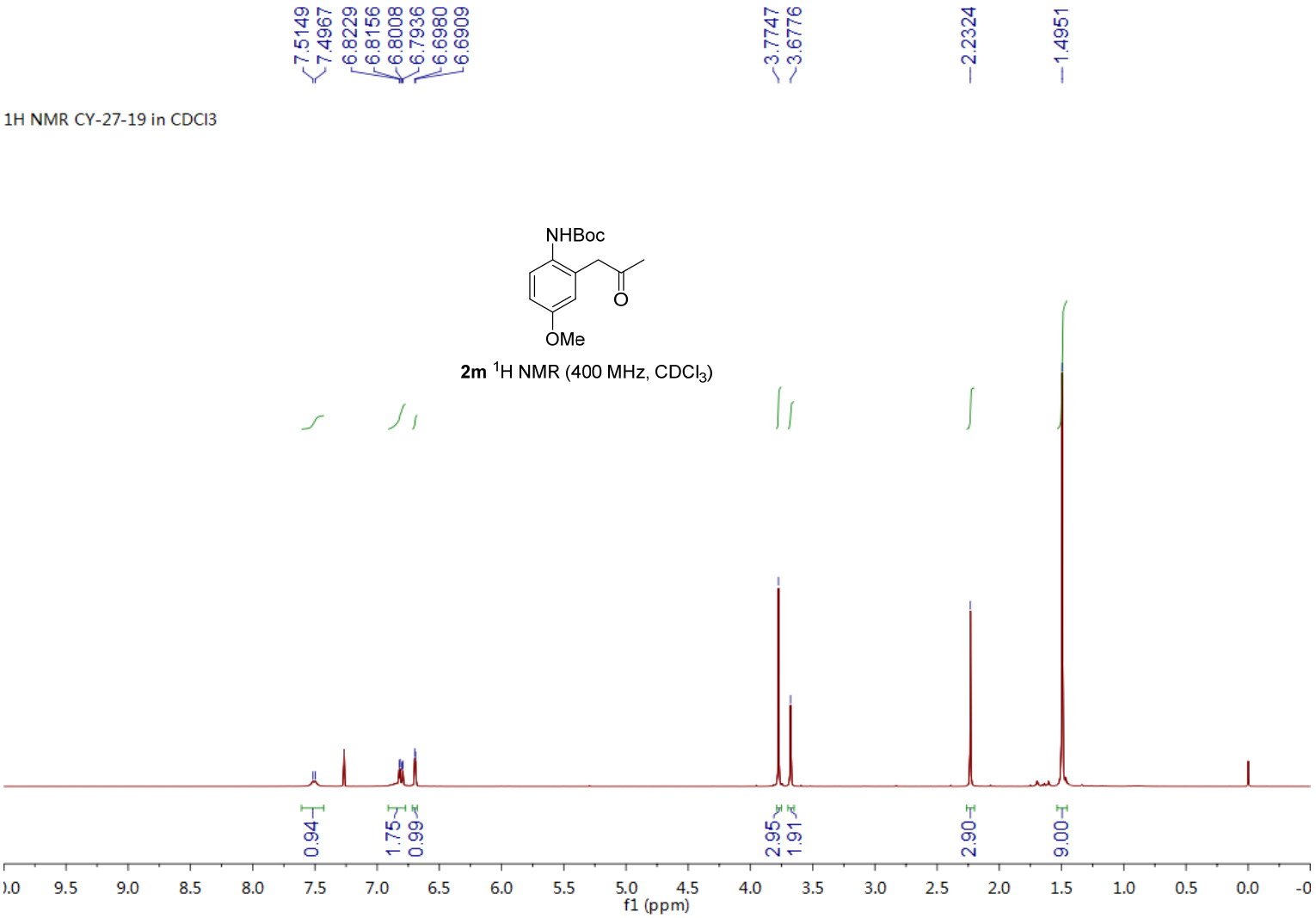


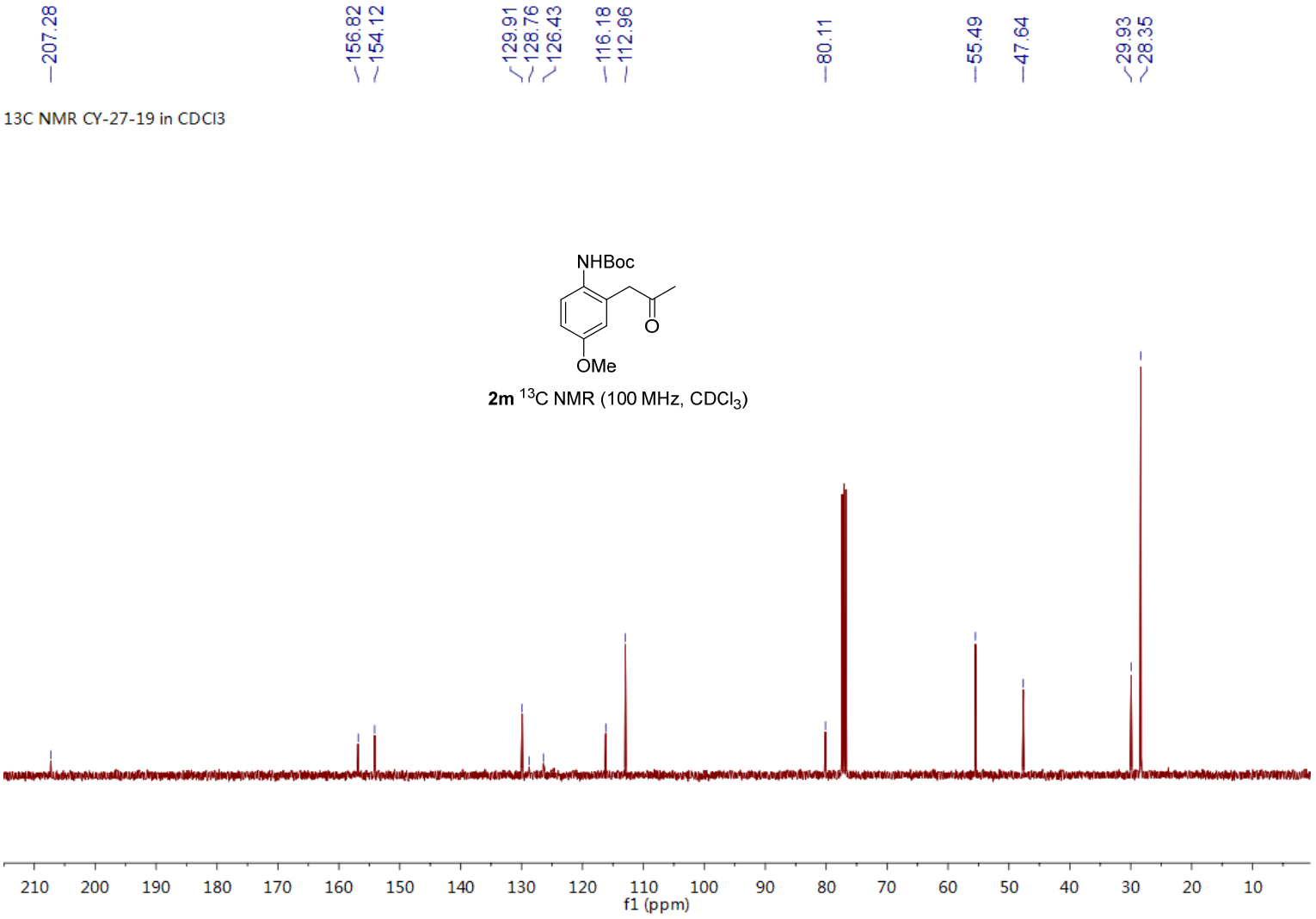
13C NMR JW-9-7

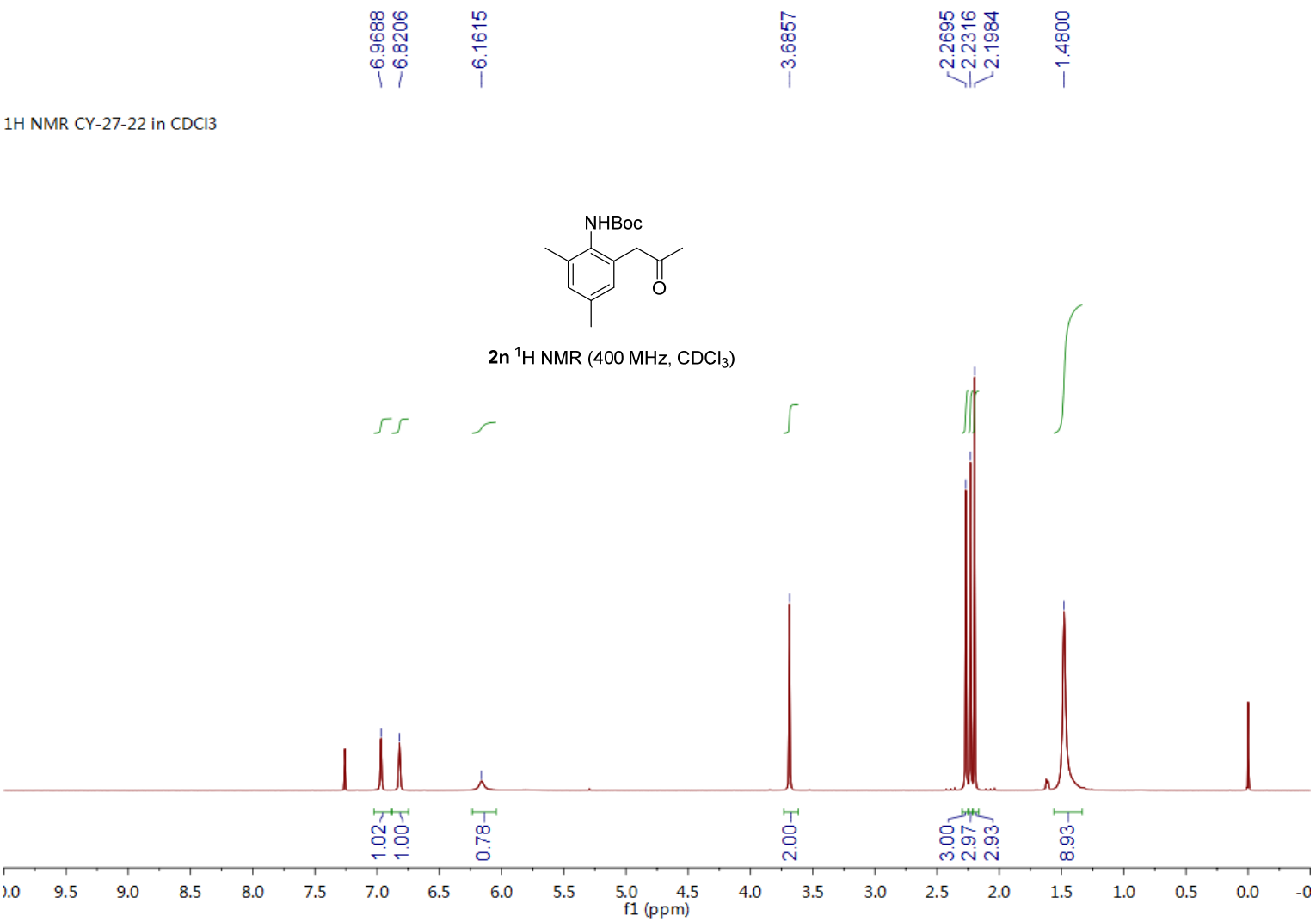


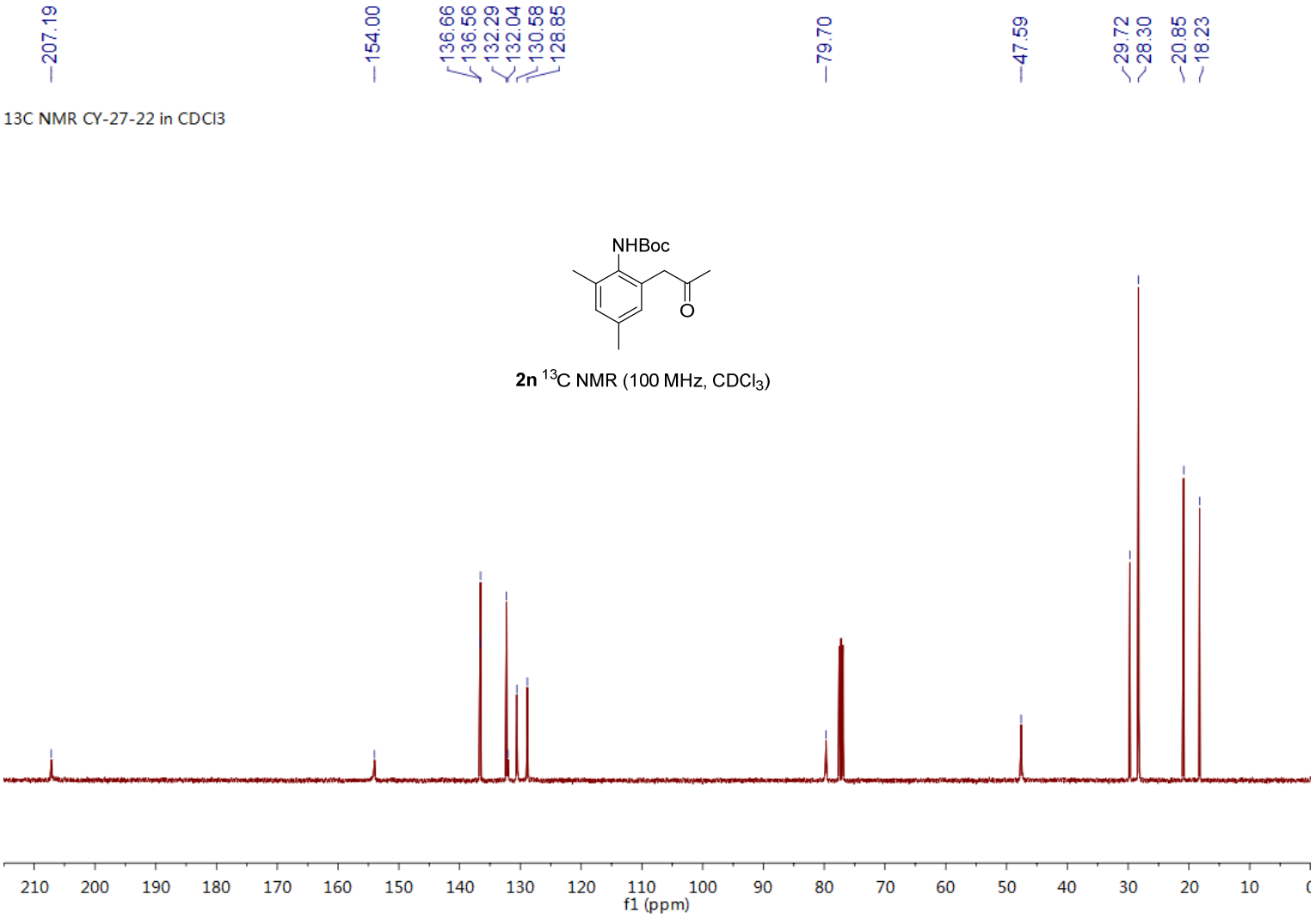
2l ^{13}C NMR (100 MHz, $CDCl_3$)

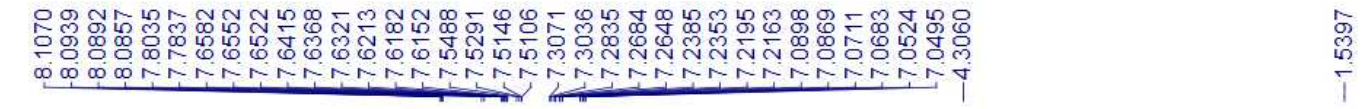



7.5149
7.4967
6.8229
6.8156
6.8008
6.7936
6.6980
6.6909
3.7747
3.6776
2.2324
1.4951
1H NMR CY-27-19 in CDCl3
NHBoc
O
OMe
2m 1H NMR (400 MHz, CDCl3)
0.94
1.75
0.99
2.95
1.91
2.90
9.00
f1 (ppm)

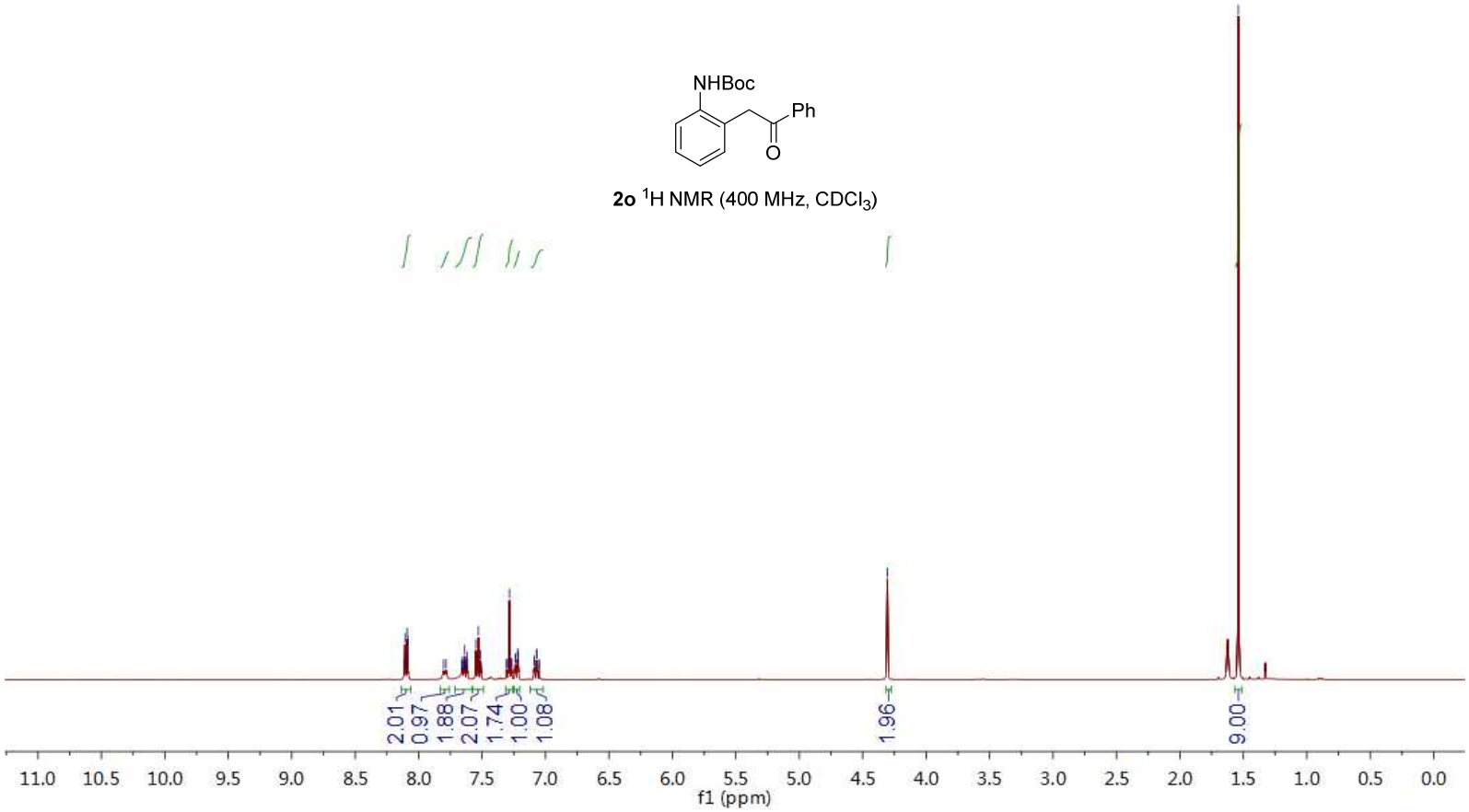

207.28
156.82
154.12
129.91
128.76
126.43
116.18
112.96
80.11
55.49
47.64
29.93
28.35
13C NMR CY-27-19 in CDCl3
NHBoc
O
OMe
2m 13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


6.9688
6.8206
6.1615
3.6857
2.2695
2.2316
2.1984
1.4800
1H NMR CY-27-22 in CDCl3
NHBoc
O
2n 1H NMR (400 MHz, CDCl3)
1.02
1.00
0.78
2.00
3.00
2.97
2.93
8.93
f1 (ppm)

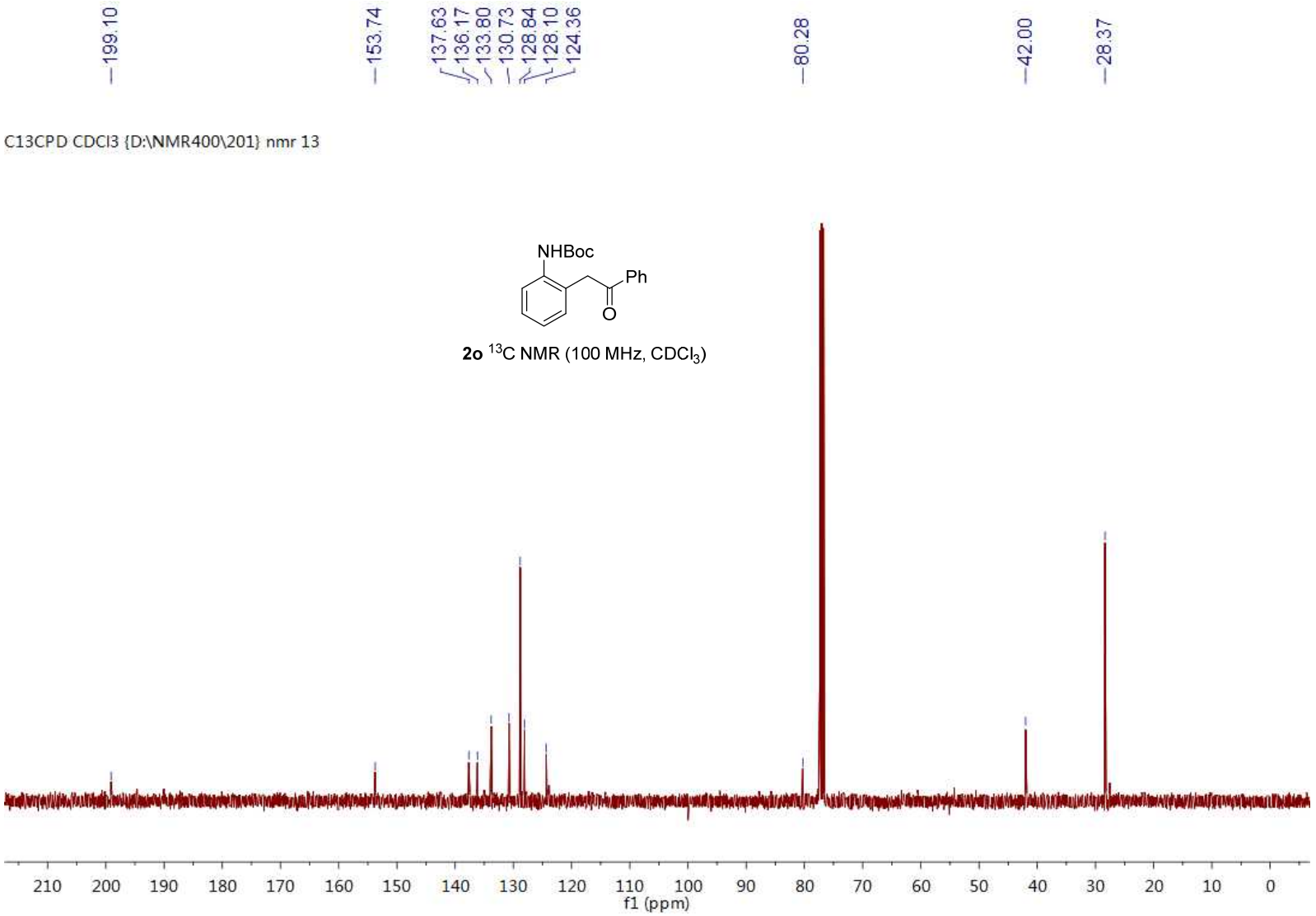

207.19
154.00
136.66
136.56
132.29
132.04
130.58
128.85
79.70
47.59
29.72
28.30
20.85
18.23
13C NMR CY-27-22 in CDCl3
NHBoc
O
2n 13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



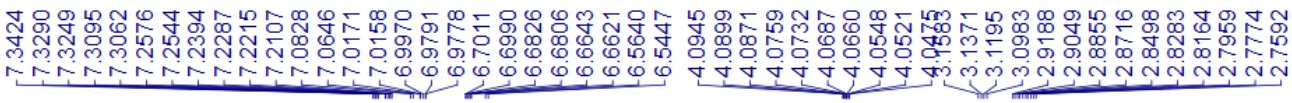
PROTON CDCl3 {D:\NMR400\201} nmr 13



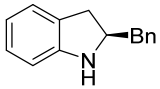
2o ^{1}H NMR (400 MHz, $CDCl_3$)



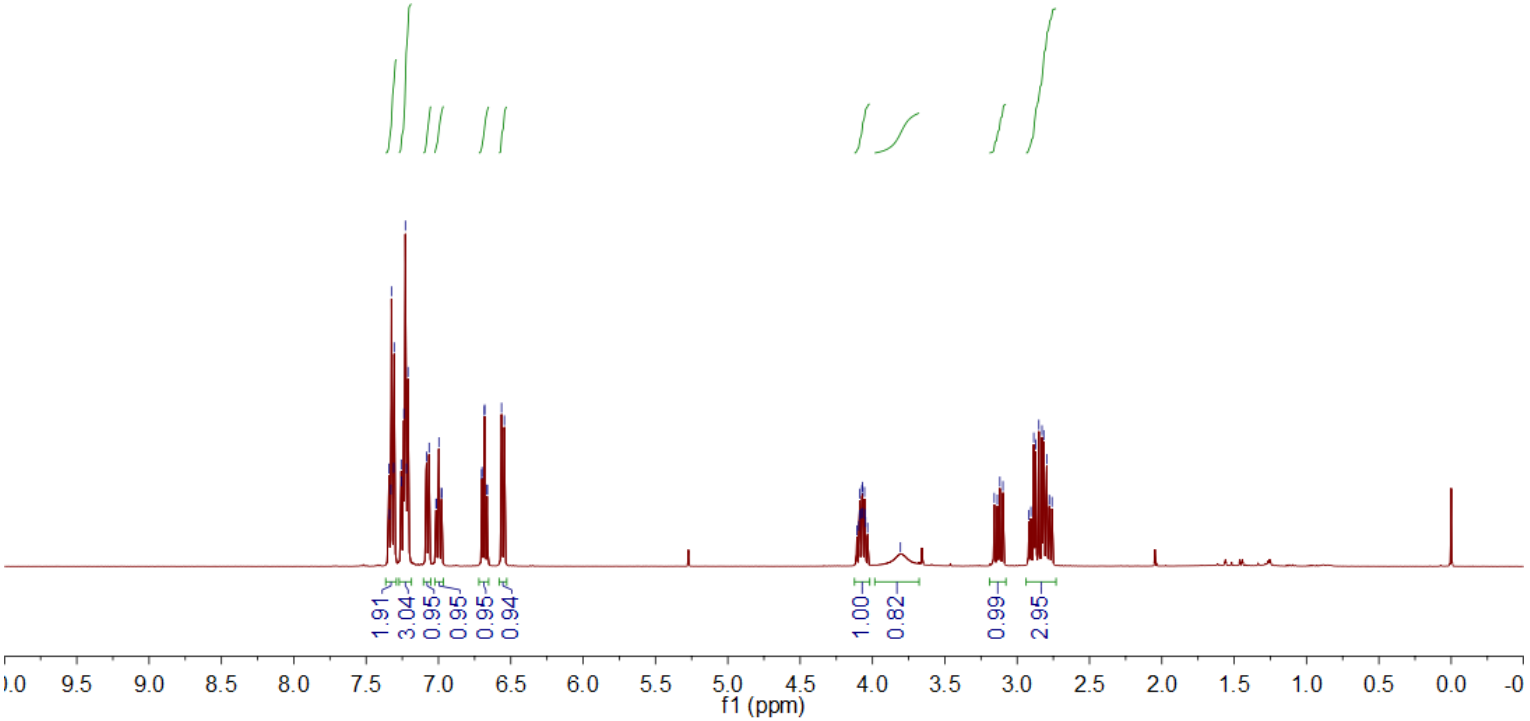
2o ^{13}C NMR (100 MHz, $CDCl_3$)



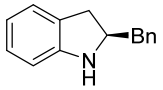
1H NMR CY-26-101 in CDCl3



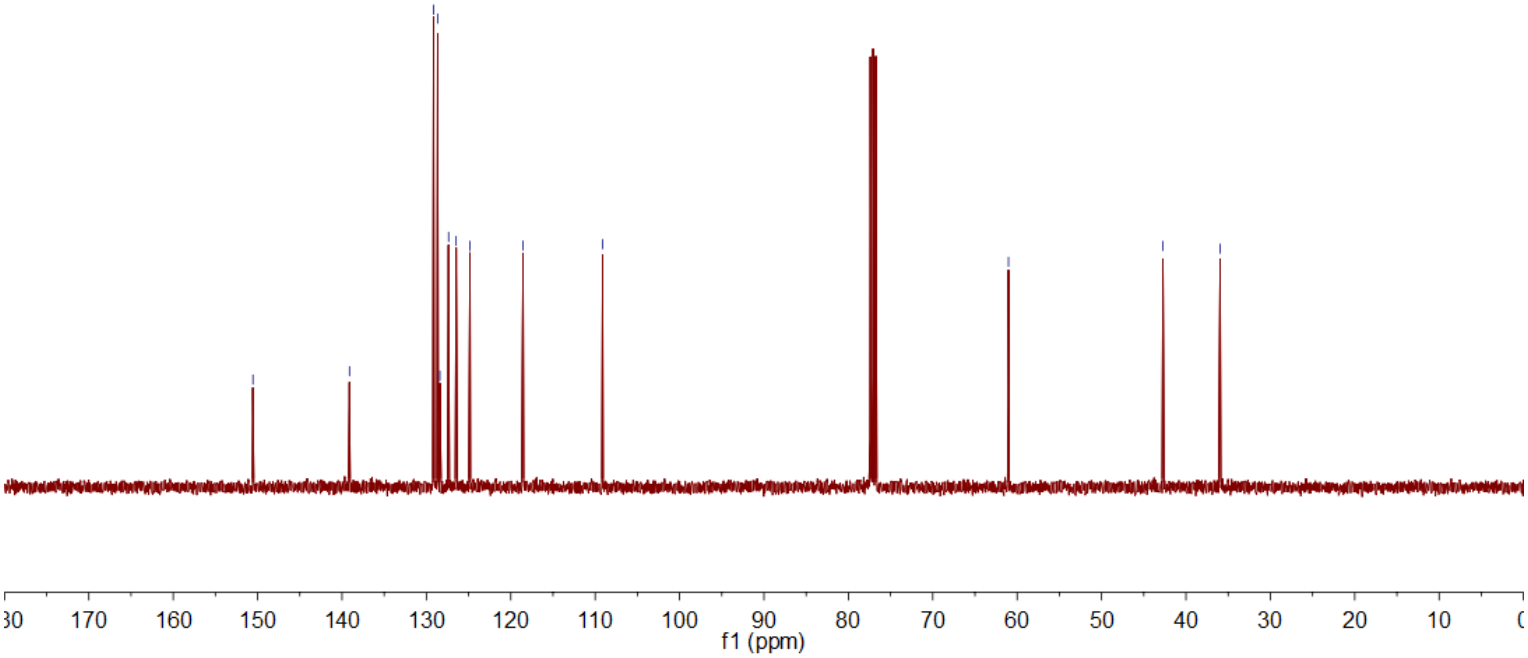
3a ^{1}H NMR (400 MHz, $CDCl_3$)



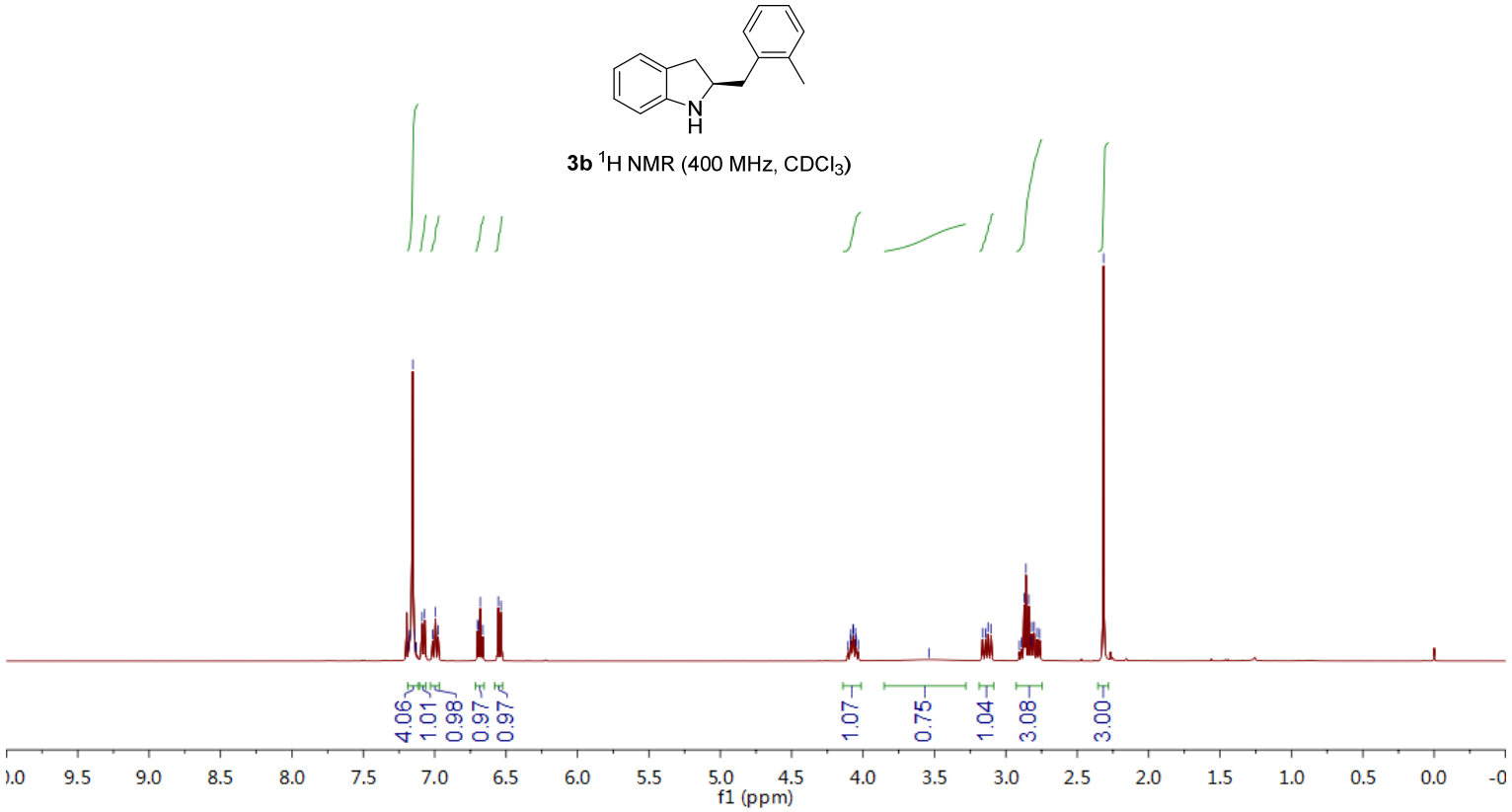
13C NMR CY-26-101 in CDCl3



3a ^{13}C NMR (100 MHz, $CDCl_3$)

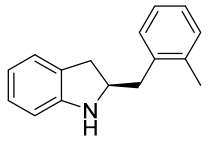


1H NMR JW-9-11A in CDCl3

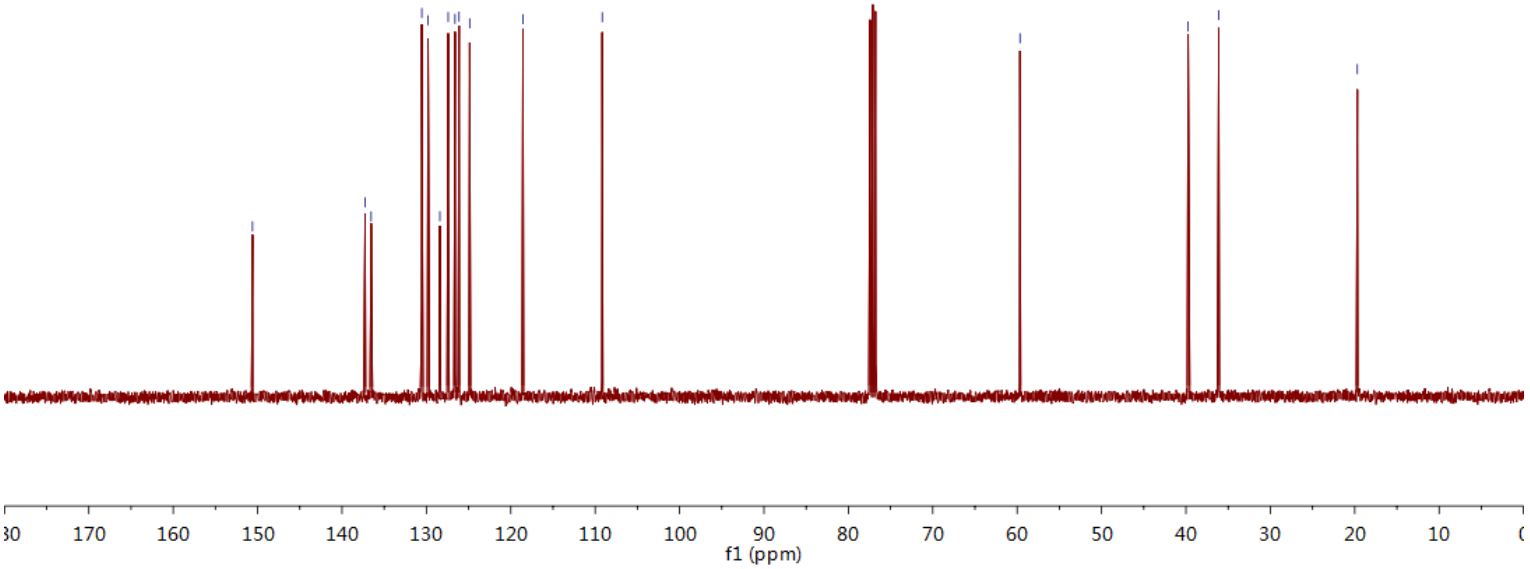


3b ^{1}H NMR (400 MHz, $CDCl_3$)

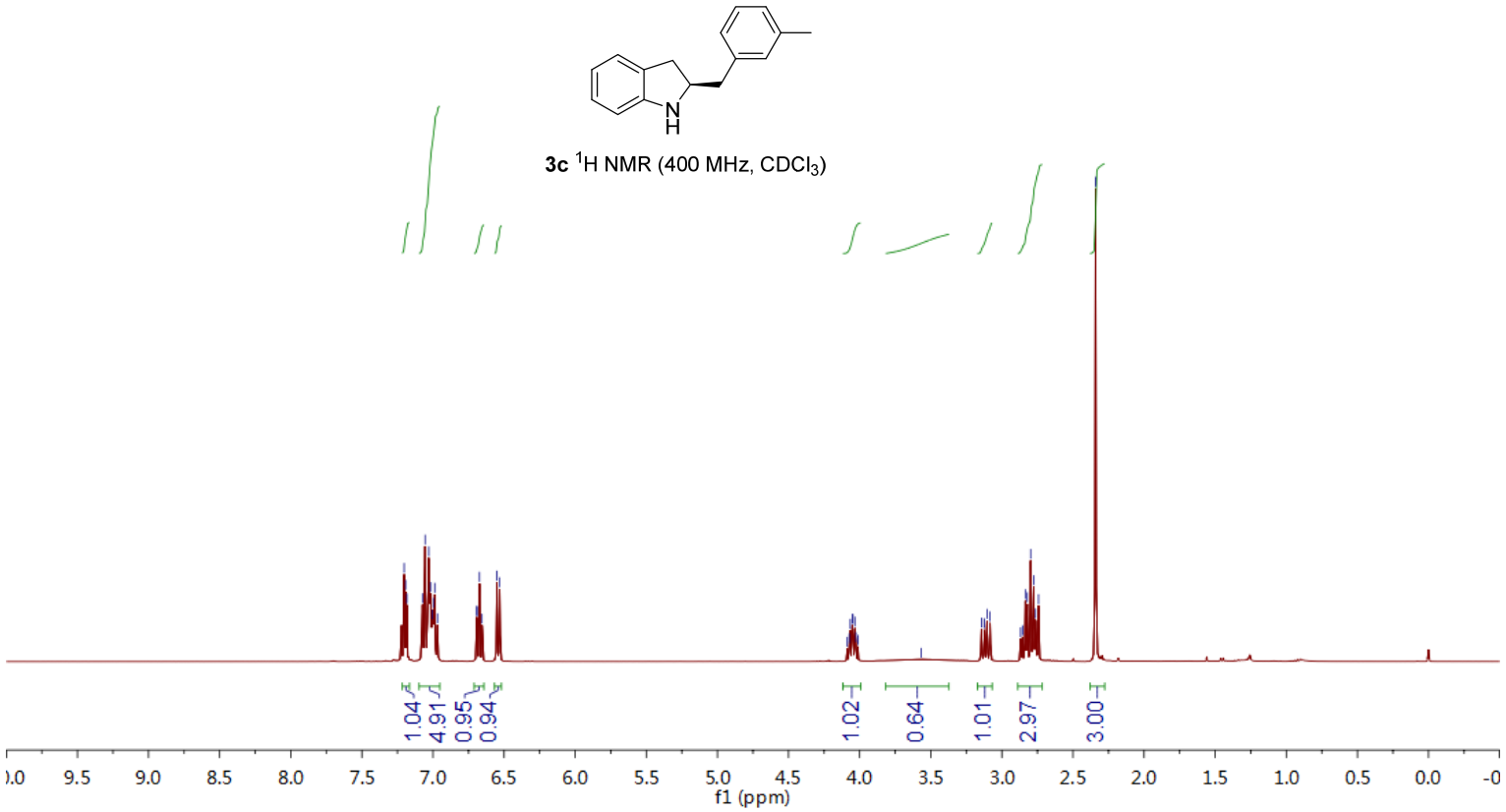
13C NMR JW-9-11A in CDCl3



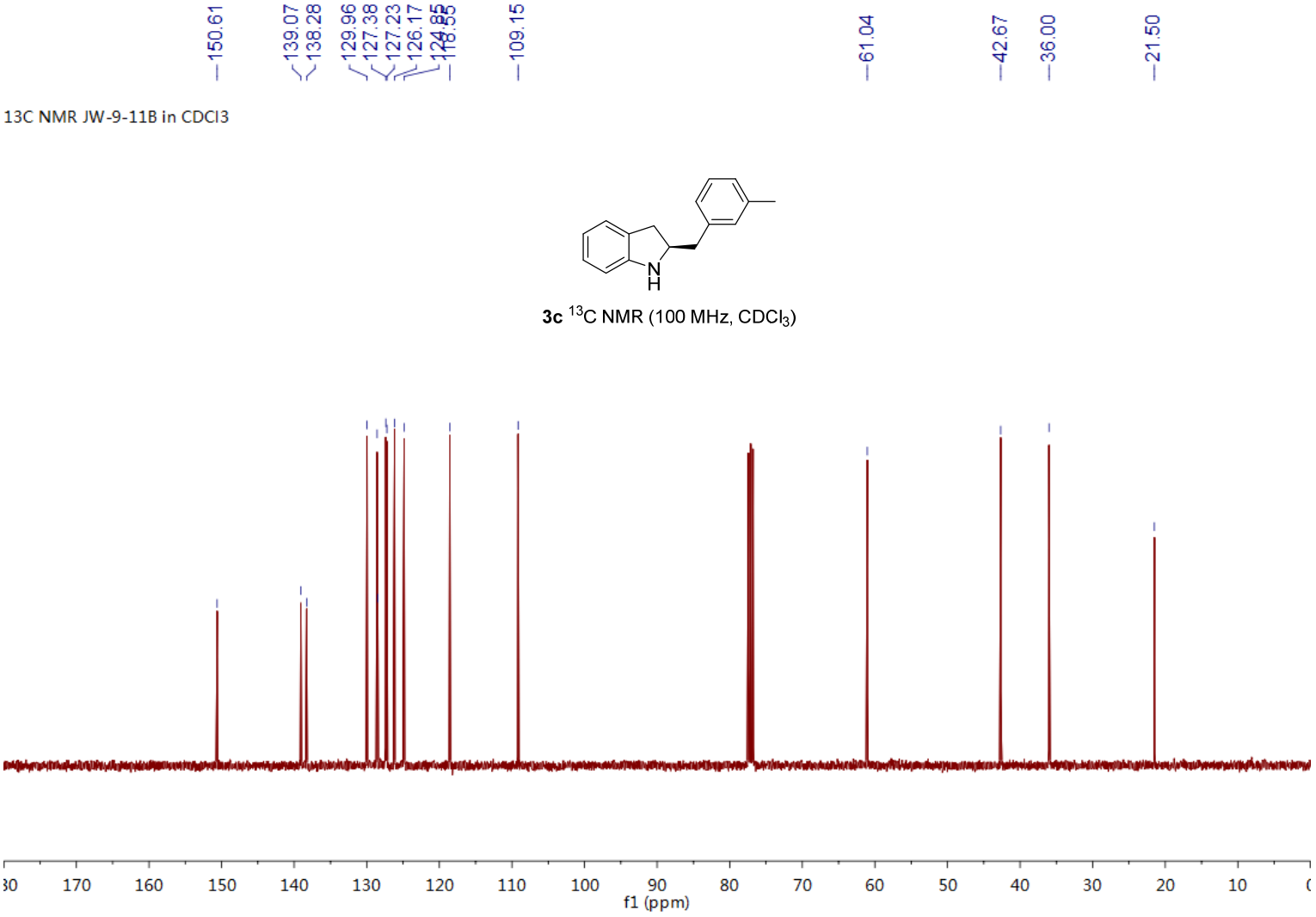
3b ^{13}C NMR (100 MHz, $CDCl_3$)



1H NMR JW-9-11B in CDCl3

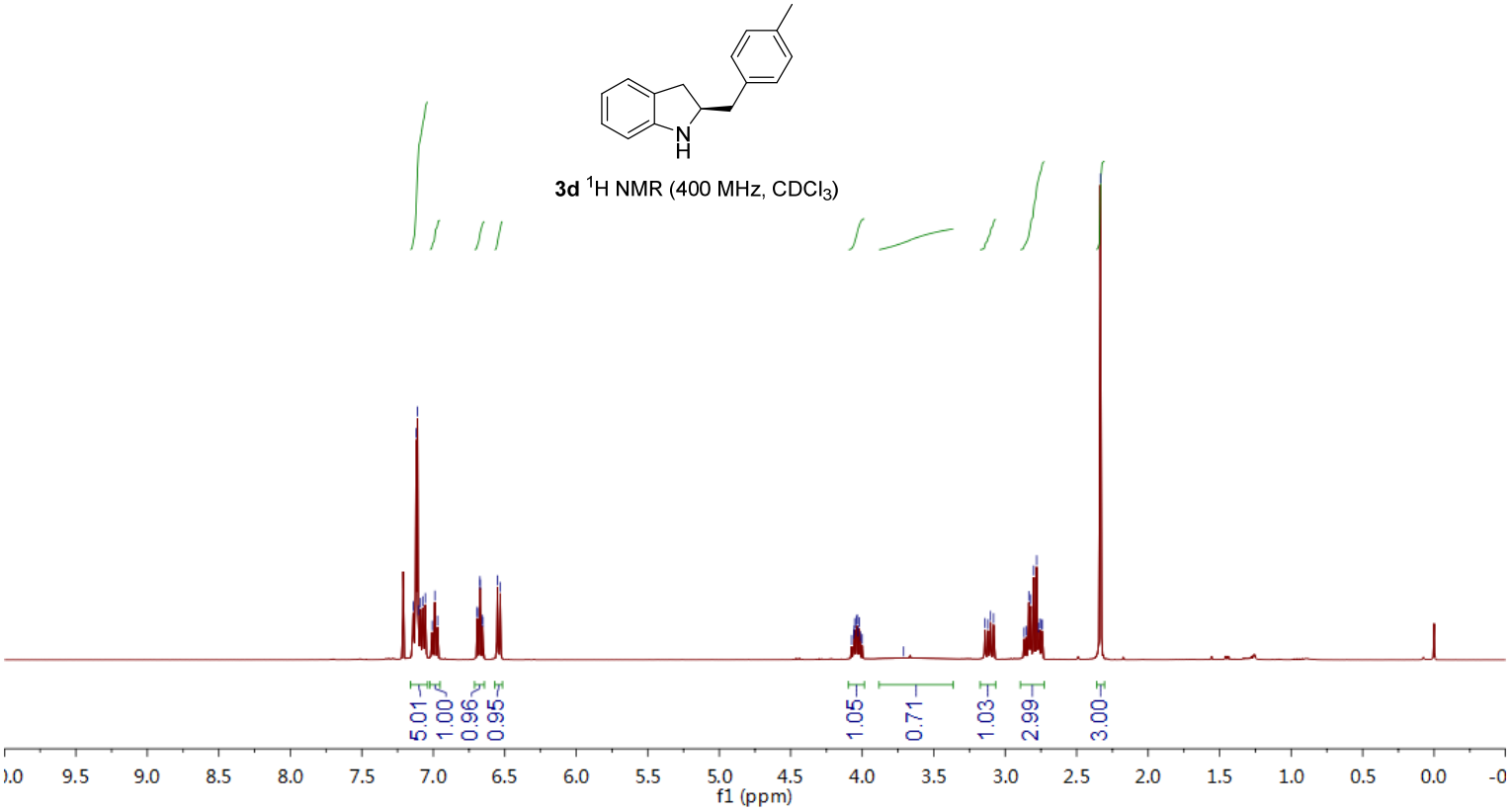


3c ^{1}H NMR (400 MHz, $CDCl_3$)

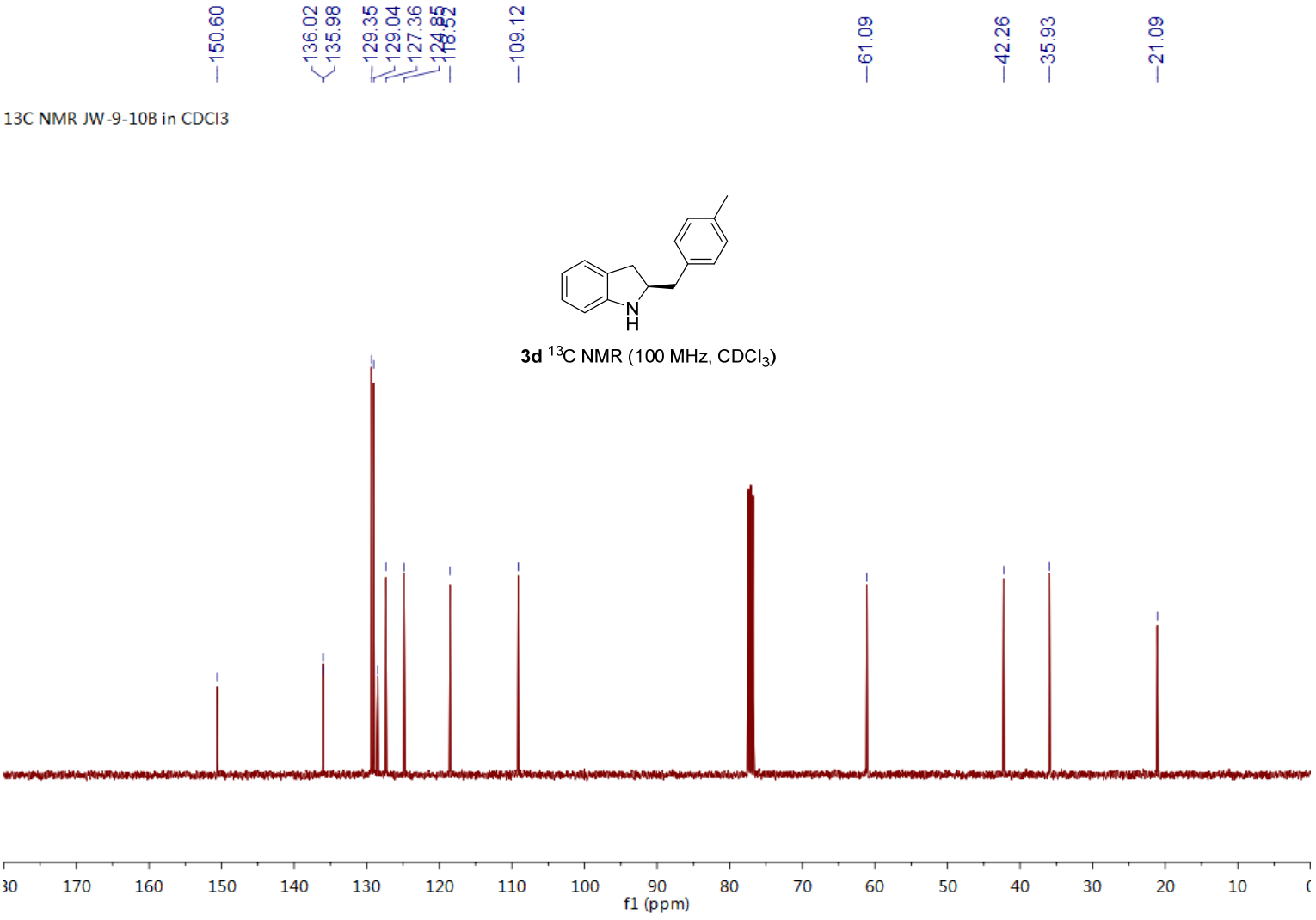


3c ^{13}C NMR (100 MHz, $CDCl_3$)

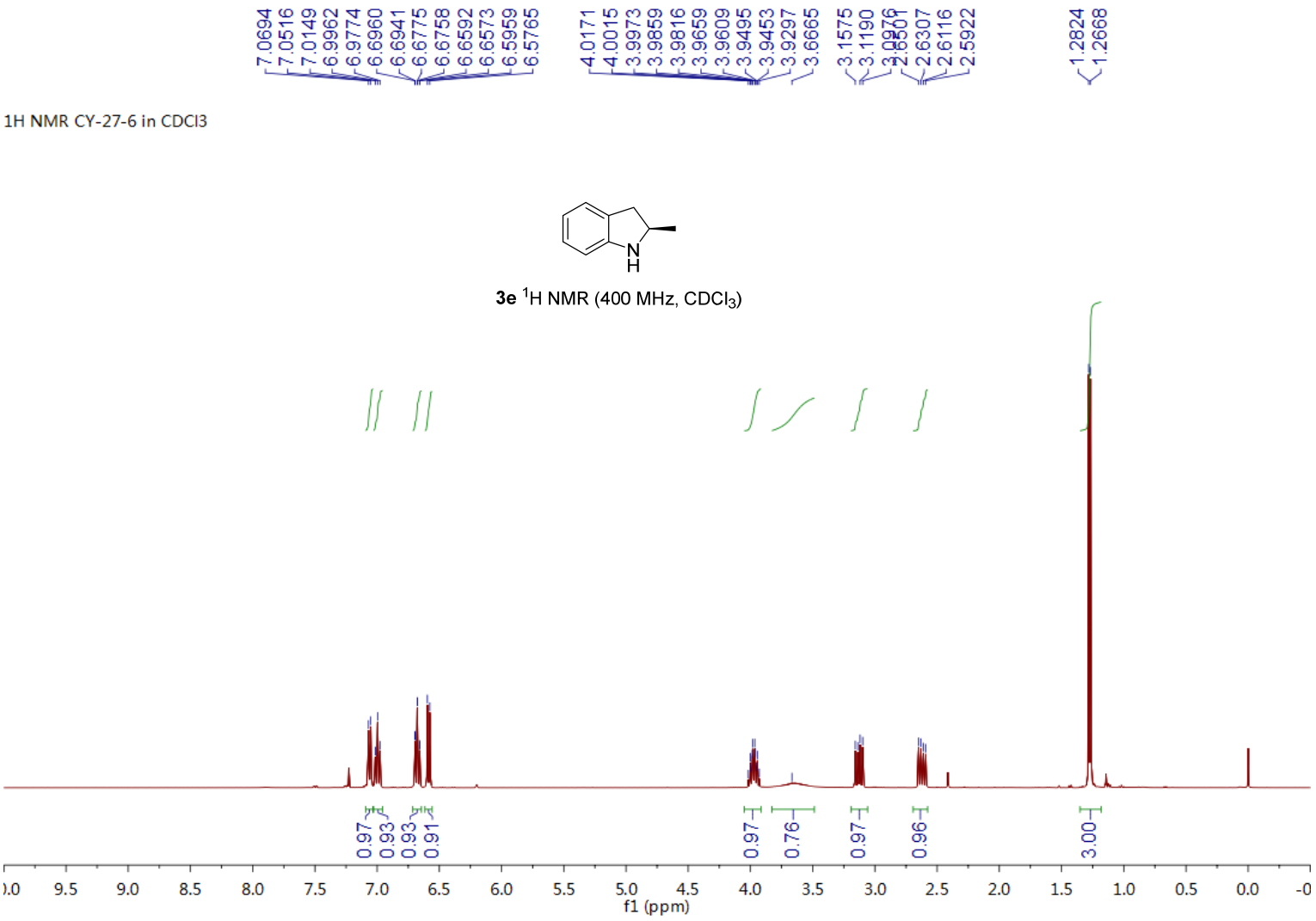
1H NMR JW-9-10B in CDCl3



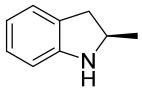
3d ^{1}H NMR (400 MHz, $CDCl_3$)



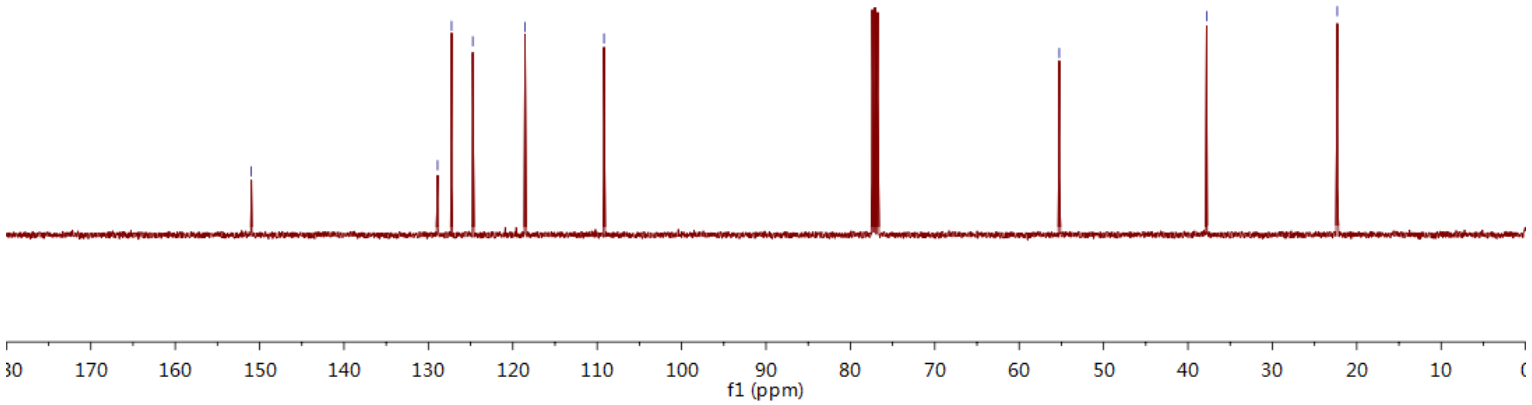
3d ^{13}C NMR (100 MHz, $CDCl_3$)


7.0694
7.0516
7.0149
6.9962
6.9774
6.6960
6.6941
6.6775
6.6758
6.6592
6.6573
6.5959
6.5765
4.0171
4.0015
3.9973
3.9859
3.9816
3.9659
3.9609
3.9495
3.9453
3.9297
3.6665
3.1575
3.1190
2.6307
2.6116
2.5922
1.2824
1.2668
1H NMR CY-27-6 in CDCl3
N
H
3e 1H NMR (400 MHz, CDCl3)
0.97
0.93
0.93
0.91
0.97
0.76
0.97
0.96
3.00
f1 (ppm)

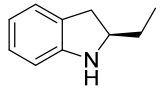
13C NMR CY-27-6 in CDCl3



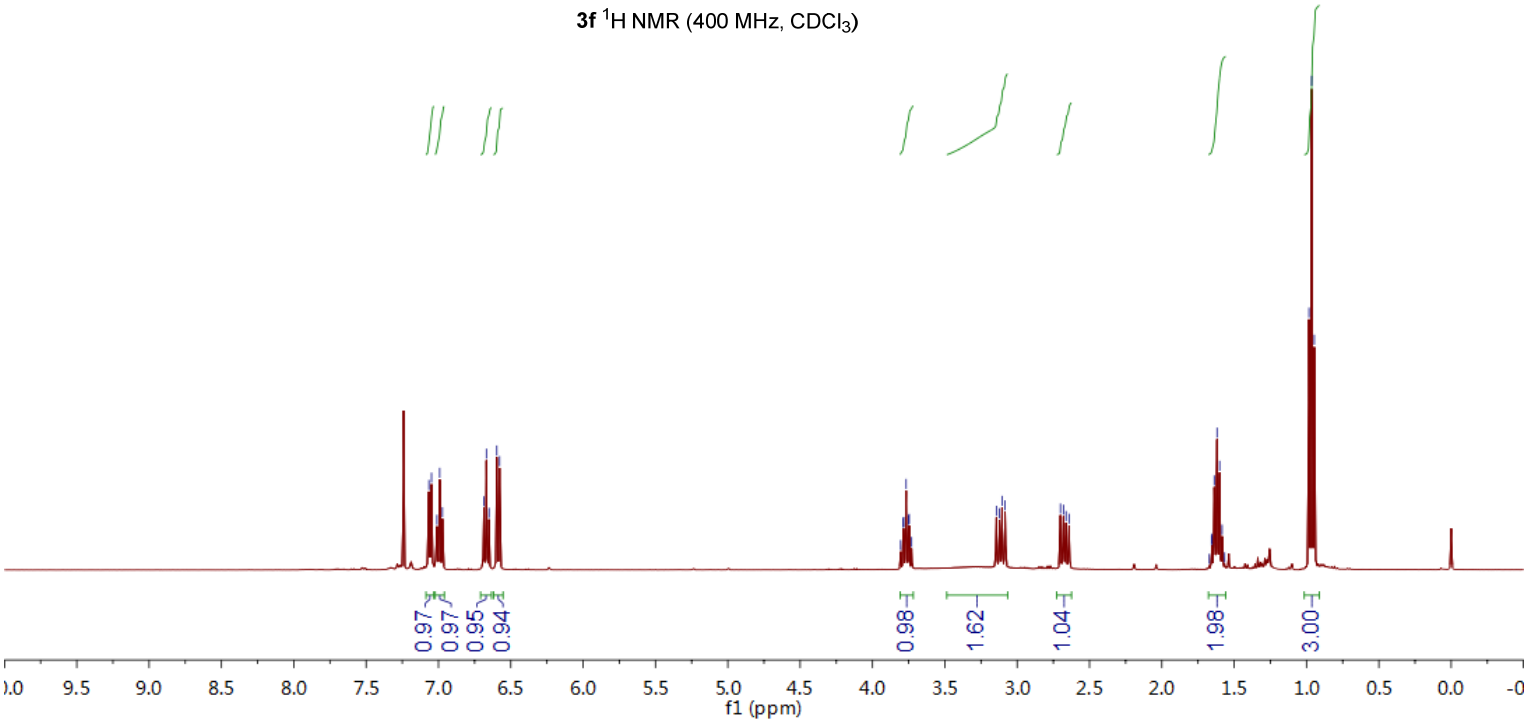
3e ^{13}C NMR (100 MHz, $CDCl_3$)

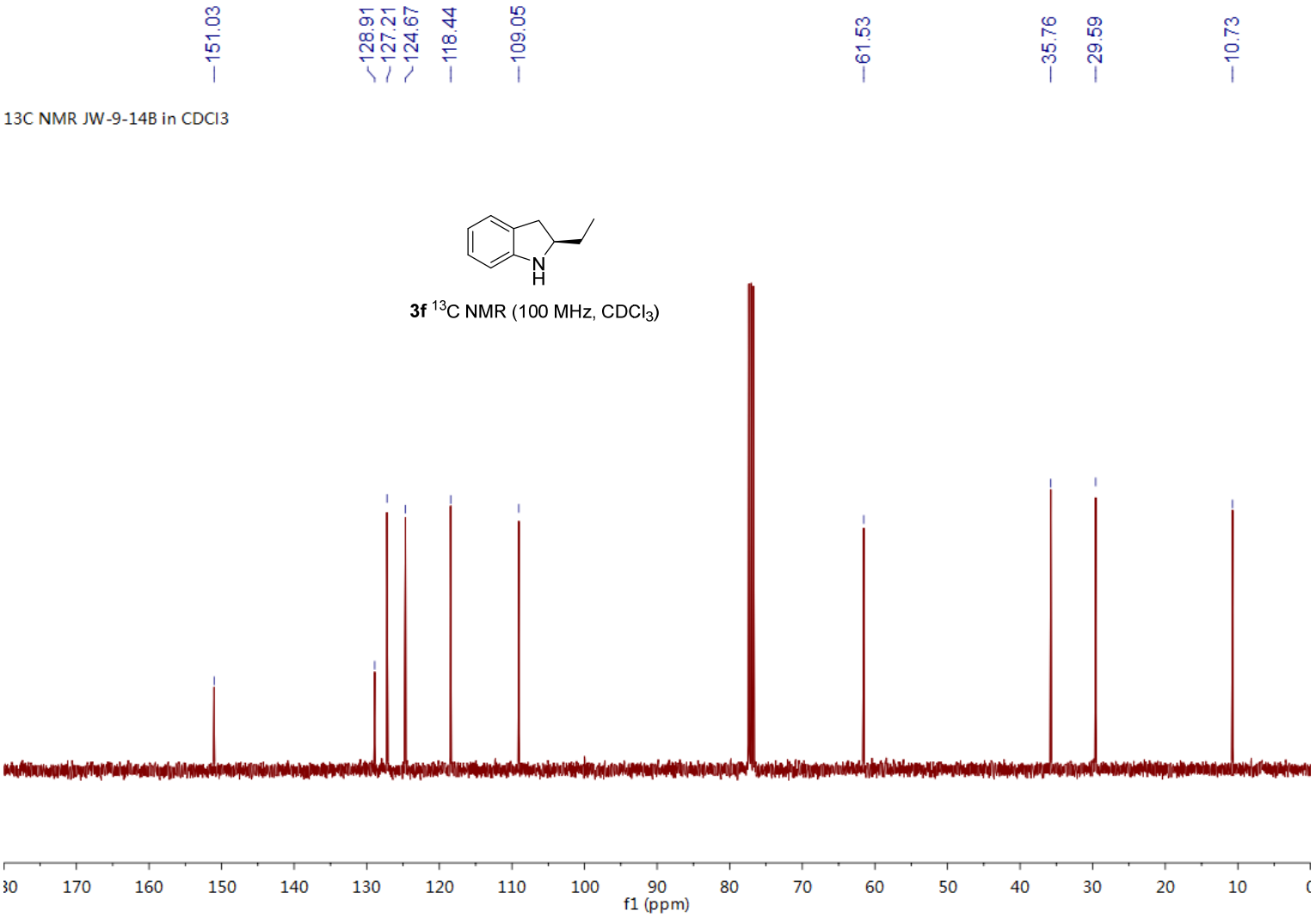


1H NMR JW-9-14B in CDCl3

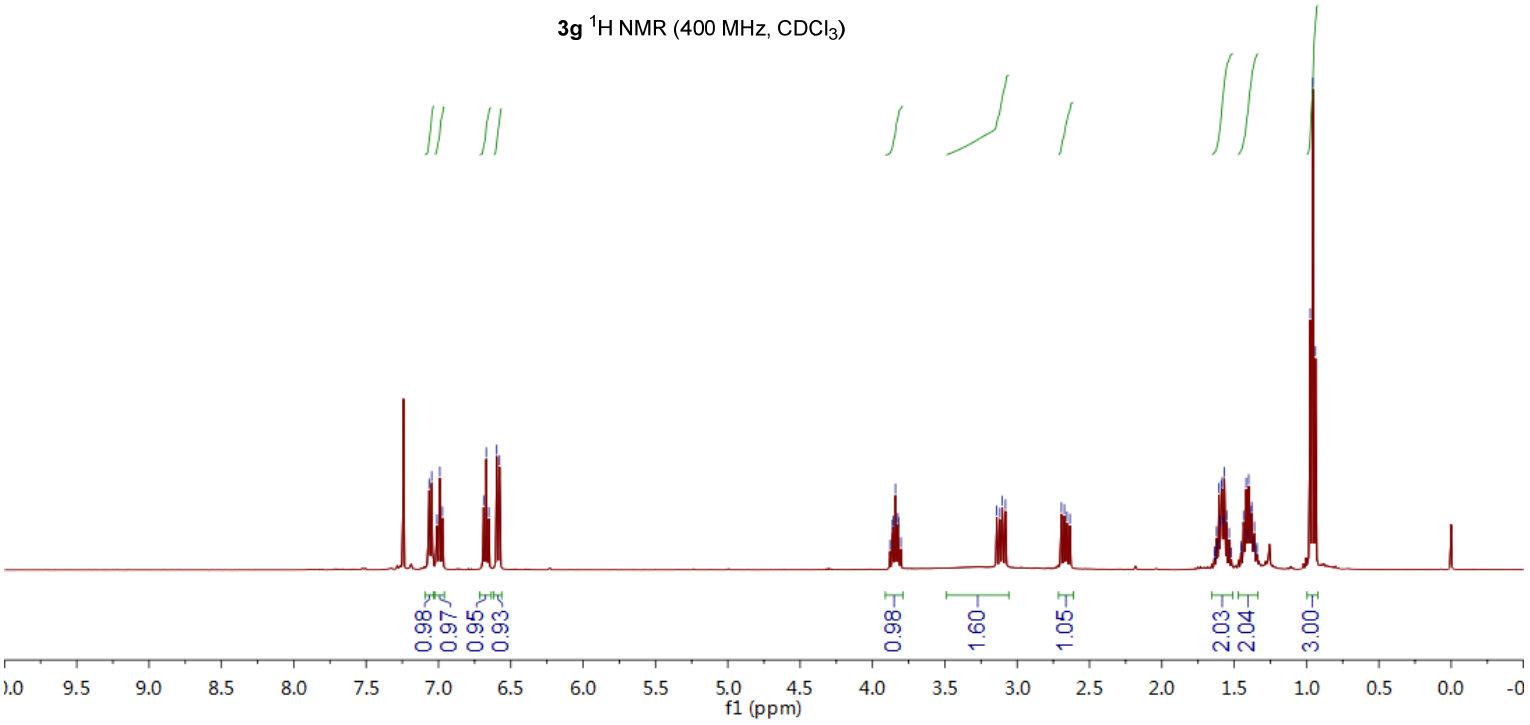


3f ^{1}H NMR (400 MHz, $CDCl_3$)

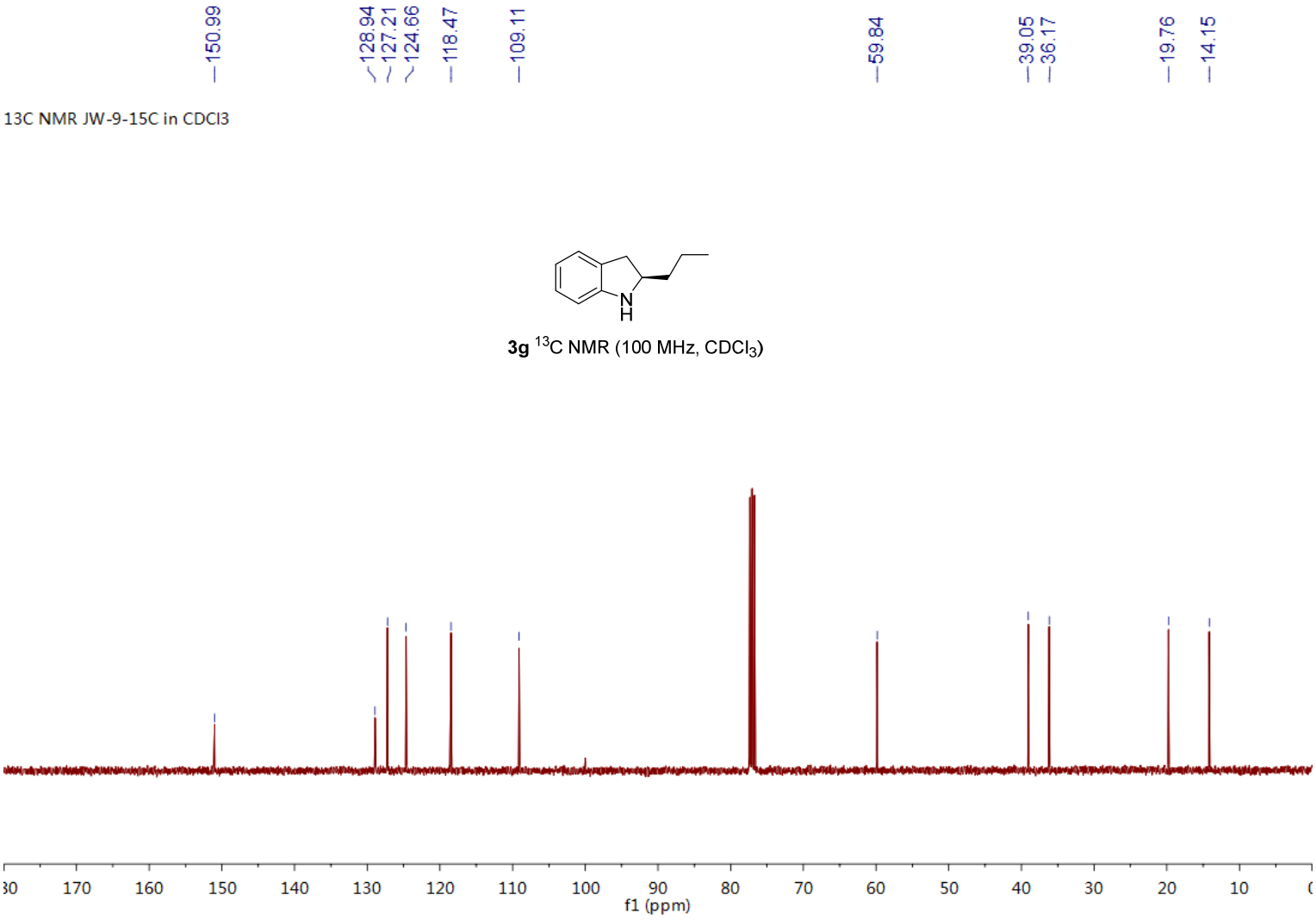



151.03
128.91
127.21
124.67
118.44
109.05
61.53
35.76
29.59
10.73
13C NMR JW-9-14B in CDCl3
N
H
3f ^{13}C NMR (100 MHz, $CDCl_3$)
f1 (ppm)

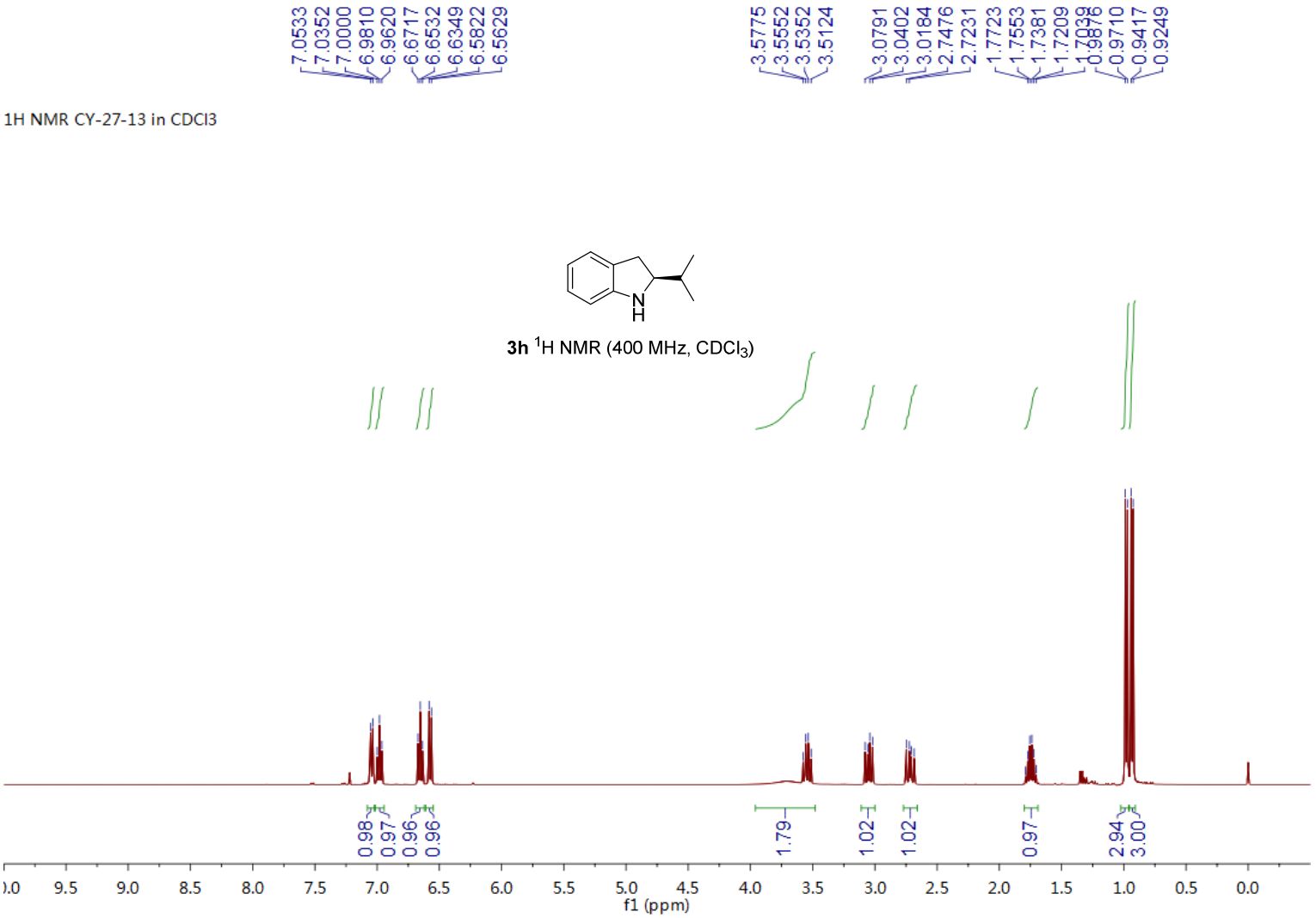
1H NMR JW-9-15C in CDCl3

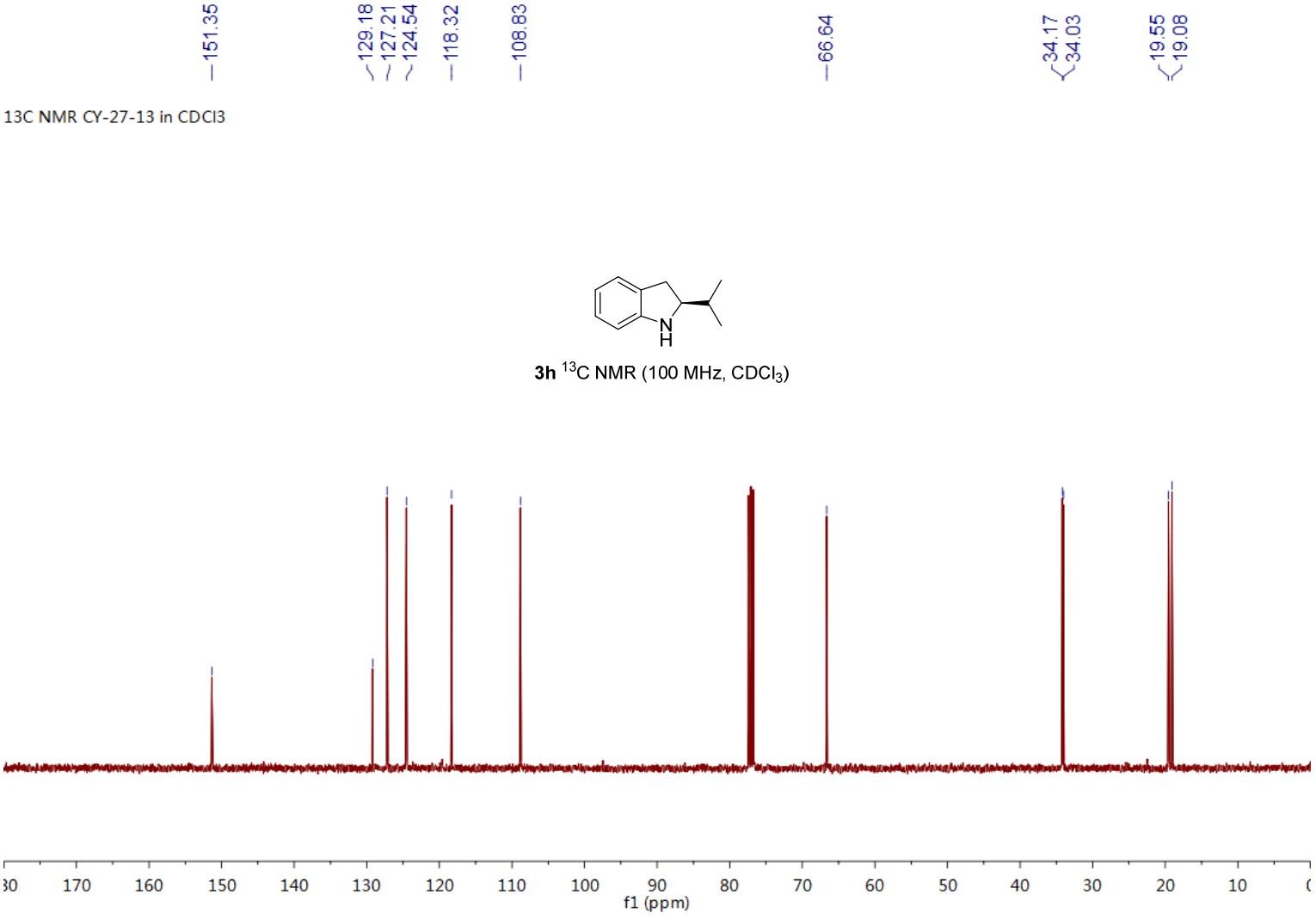


3g ^{1}H NMR (400 MHz, $CDCl_3$)



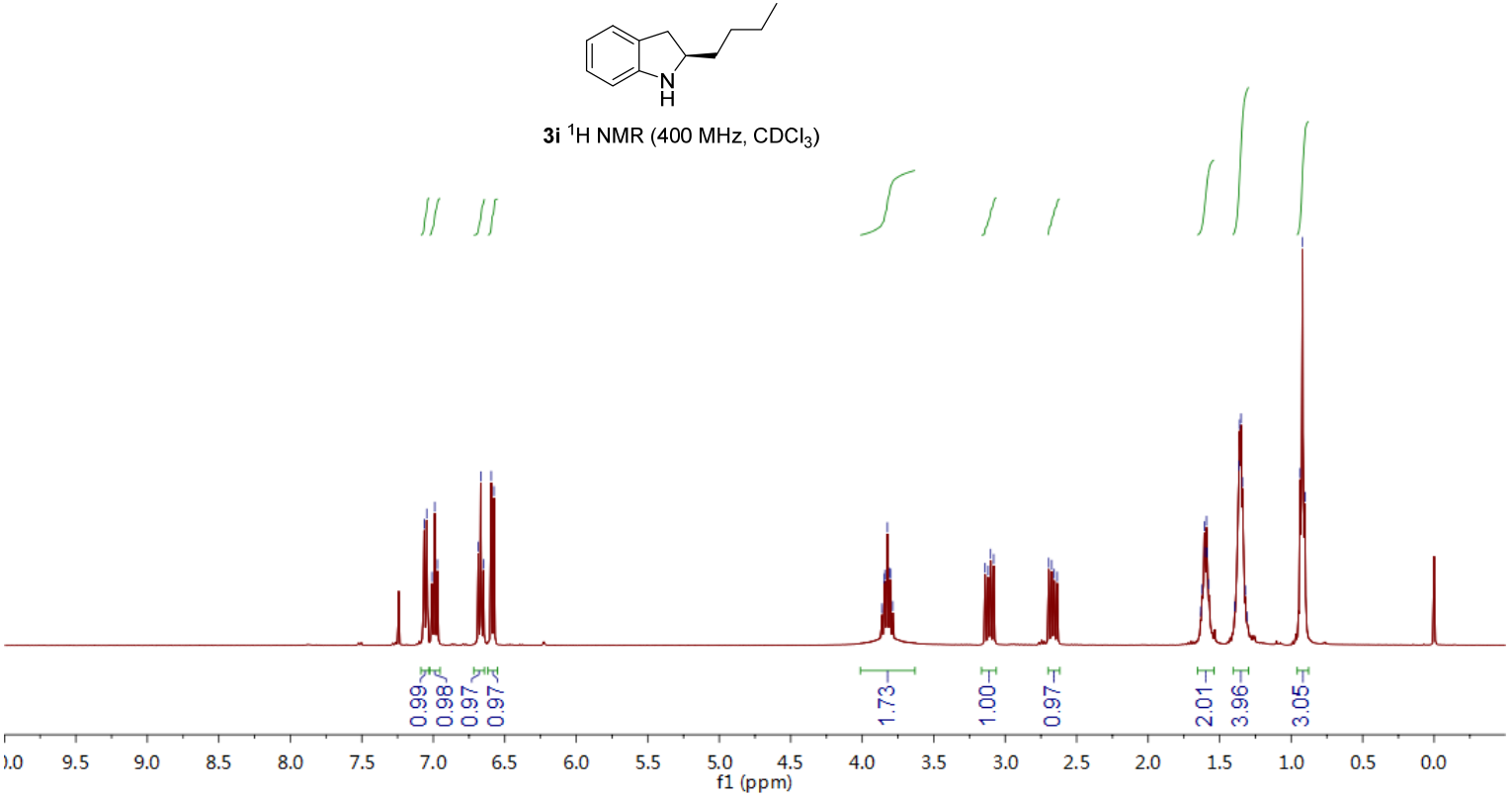
3g ^{13}C NMR (100 MHz, $CDCl_3$)


7.0533
7.0352
7.0000
6.9810
6.9620
6.6717
6.6532
6.6349
6.5822
6.5629
3.5775
3.5552
3.5352
3.5124
3.0791
3.0402
3.0184
2.7476
2.7231
1.7723
1.7553
1.7381
1.7209
1.7039
0.9878
0.9710
0.9417
0.9249
1H NMR CY-27-13 in CDCl3
N
H
3h 1H NMR (400 MHz, CDCl3)
0.98
0.97
0.96
0.96
1.79
1.02
1.02
0.97
2.94
3.00
f1 (ppm)

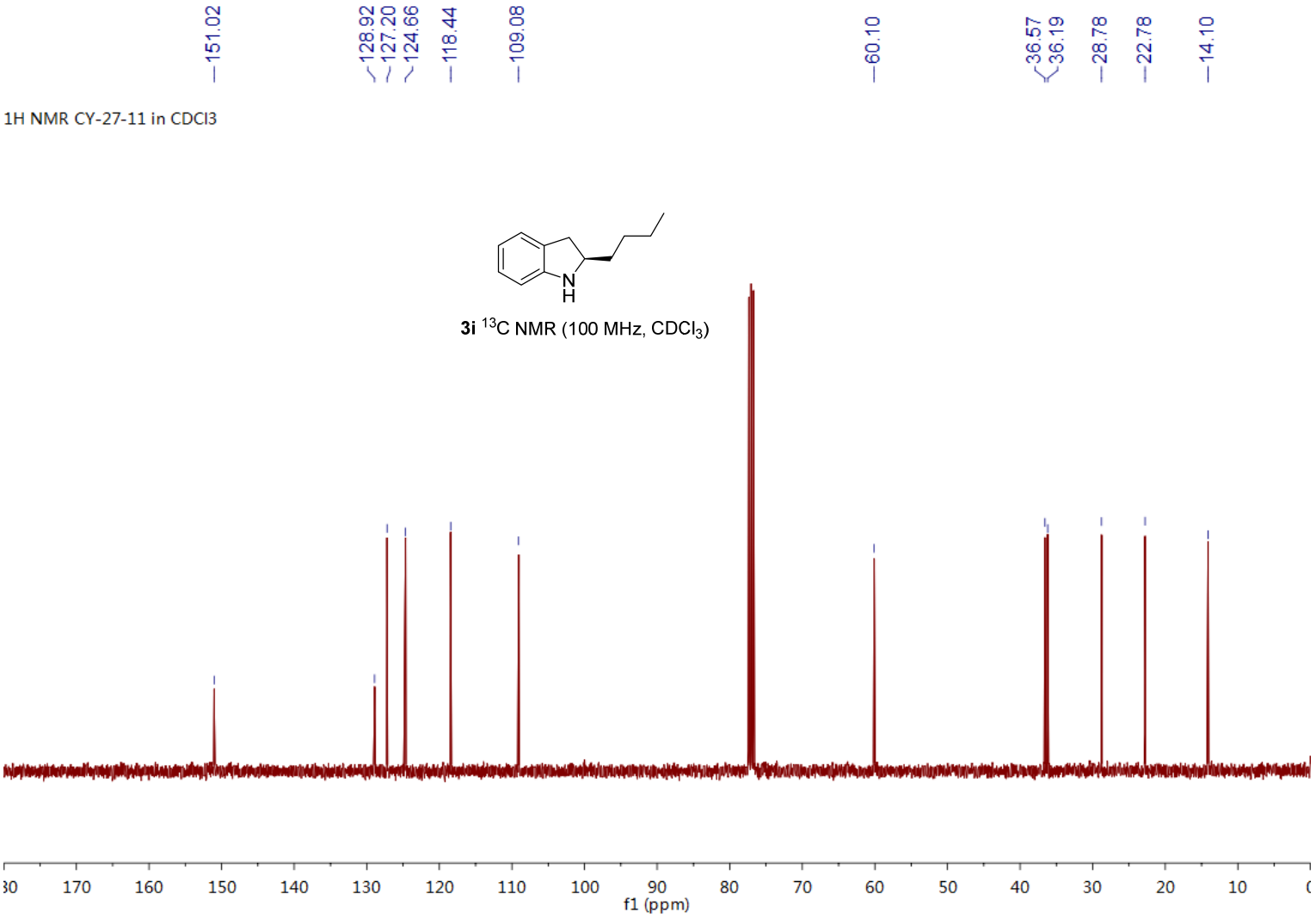


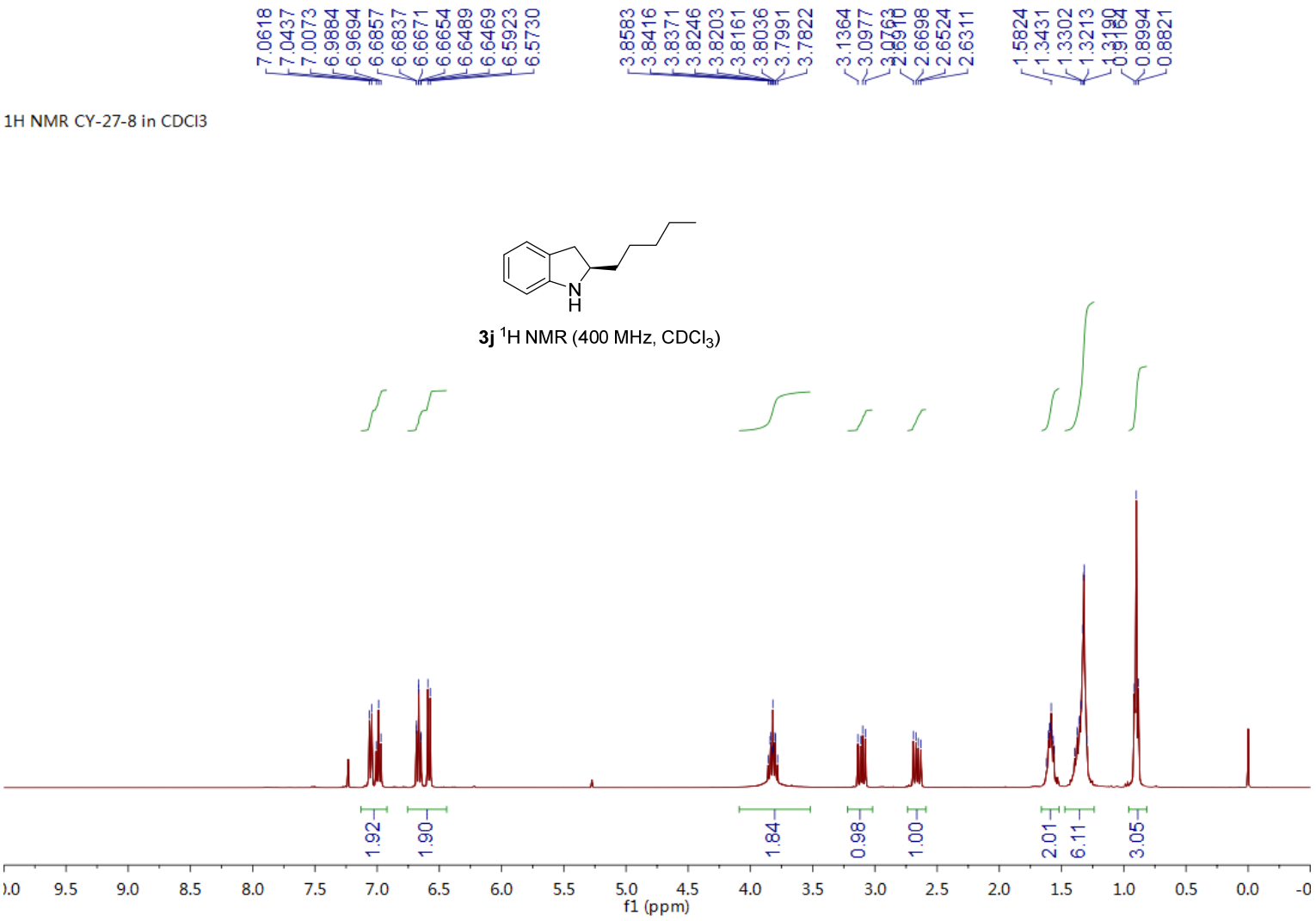
3h ^{13}C NMR (100 MHz, $CDCl_3$)

1H NMR CY-27-11 in CDCl3



3i ^{1}H NMR (400 MHz, $CDCl_3$)

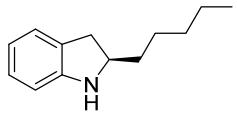

151.02
128.92
127.20
124.66
118.44
109.08
60.10
36.57
36.19
28.78
22.78
14.10
1H NMR CY-27-11 in CDCl3
N
H
3i ^{13}C NMR (100 MHz, $CDCl_3$)
f1 (ppm)



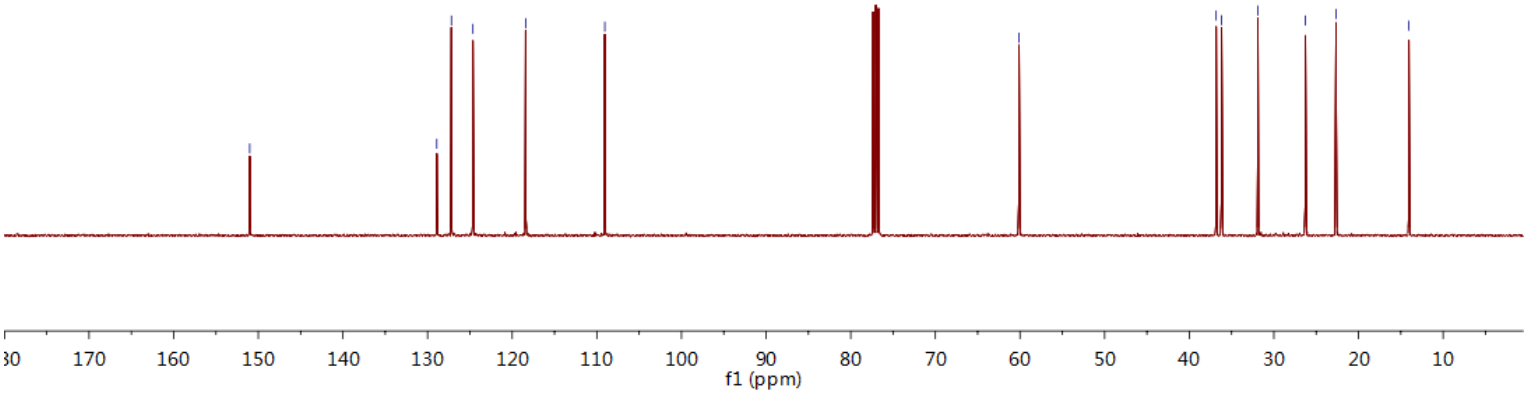
1H NMR CY-27-8 in CDCl3

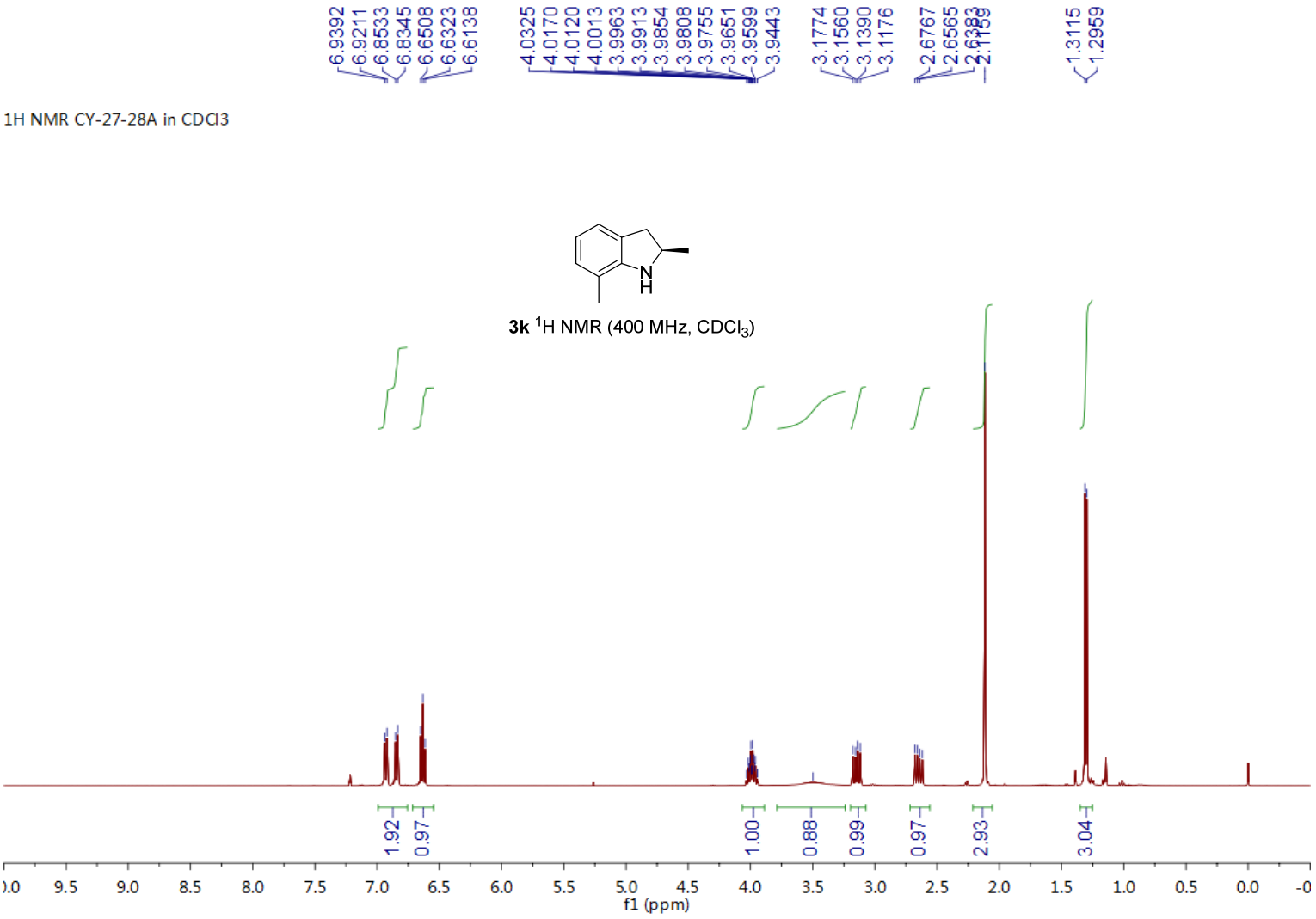
3j ^{1}H NMR (400 MHz, $CDCl_3$)

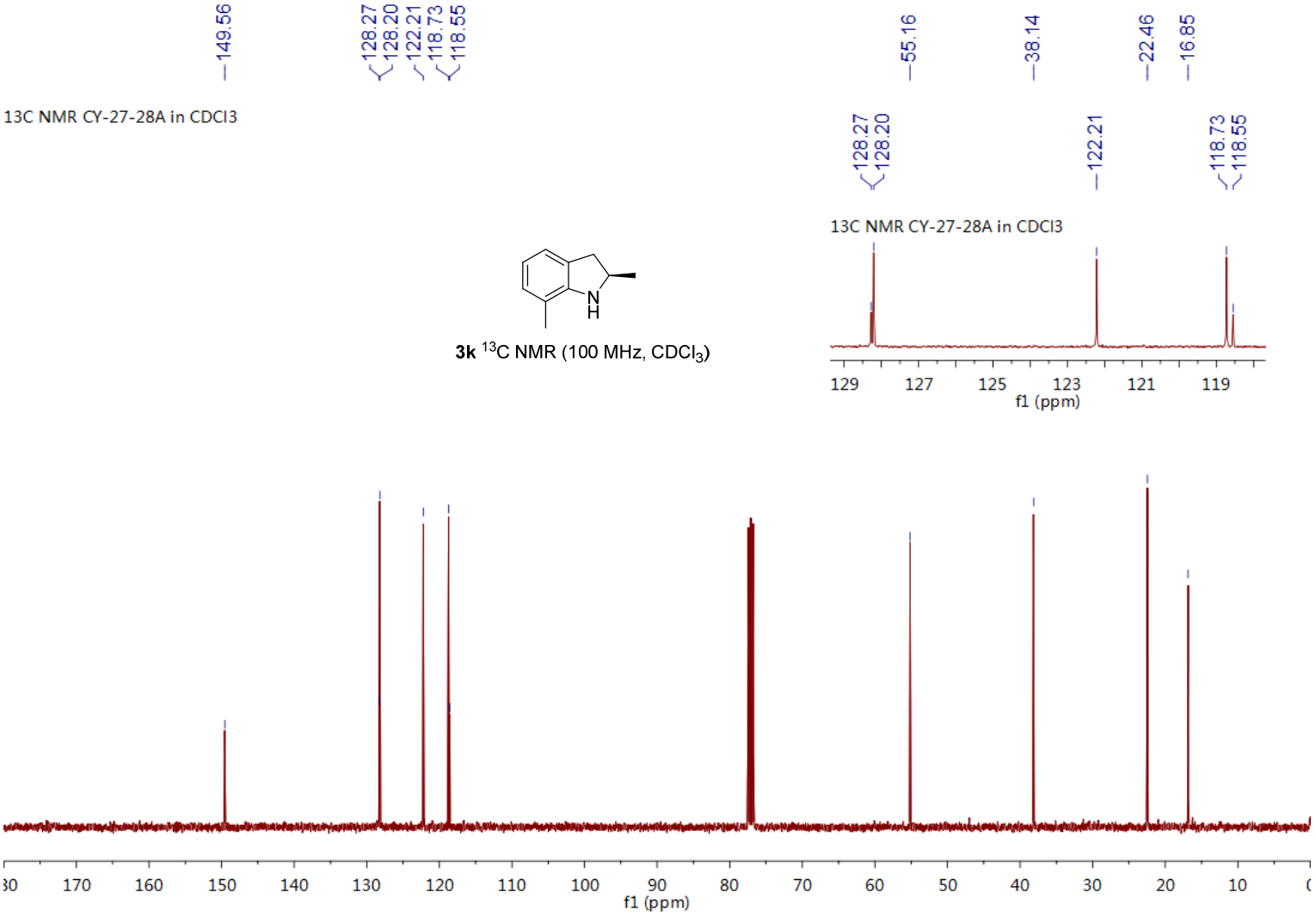
13C NMR CY-27-8 in CDCl3

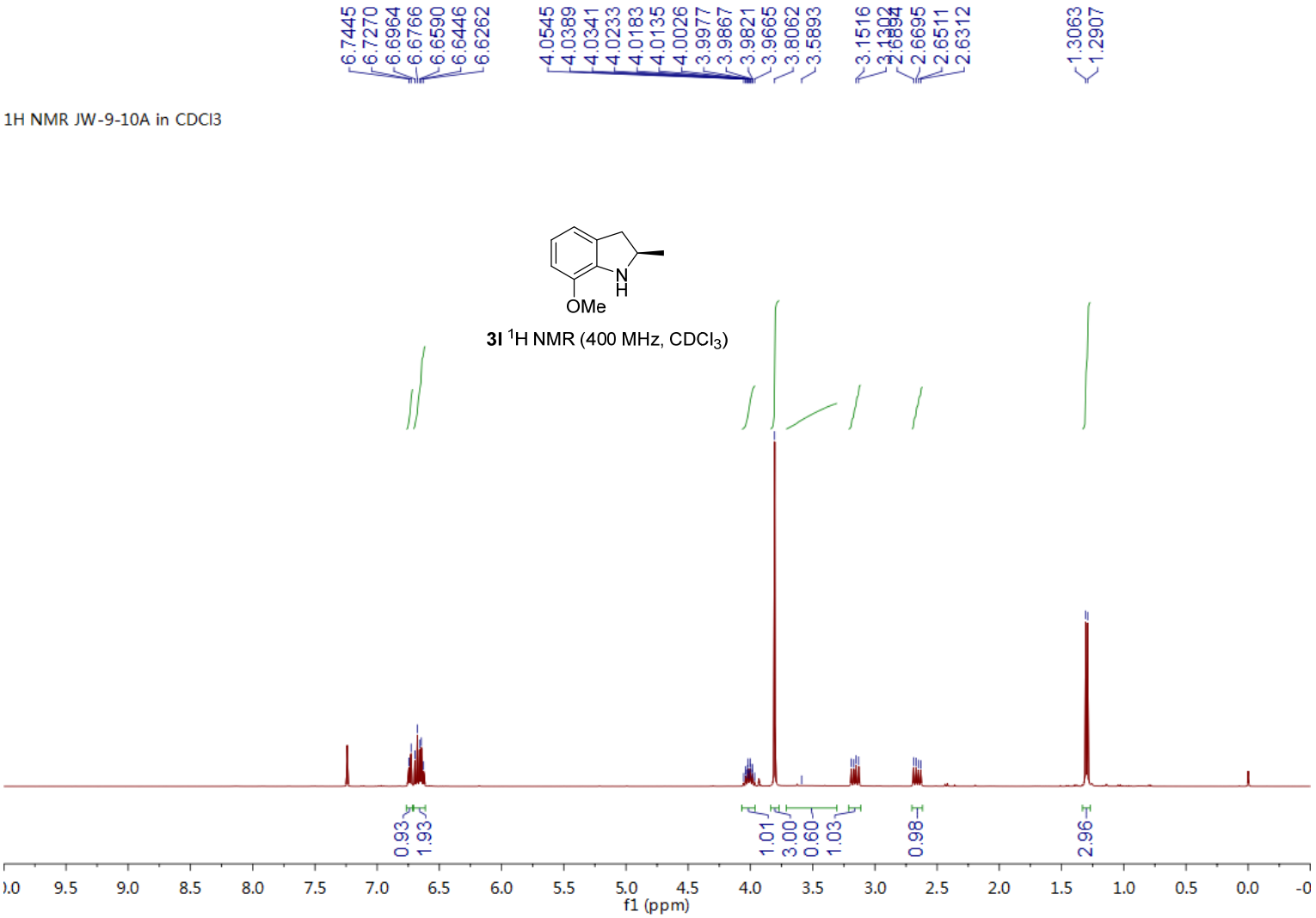


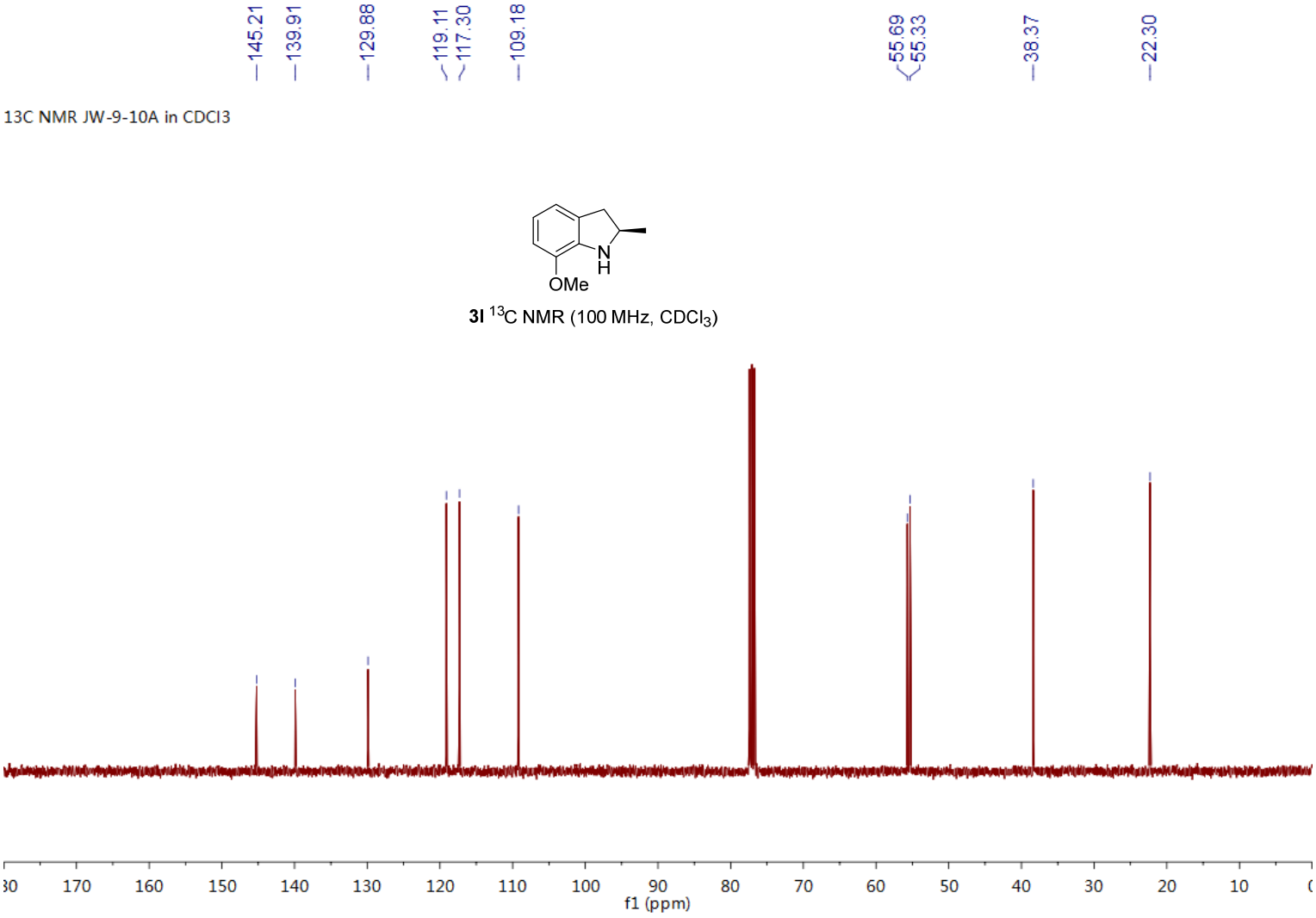
3j ^{13}C NMR (100 MHz, $CDCl_3$)



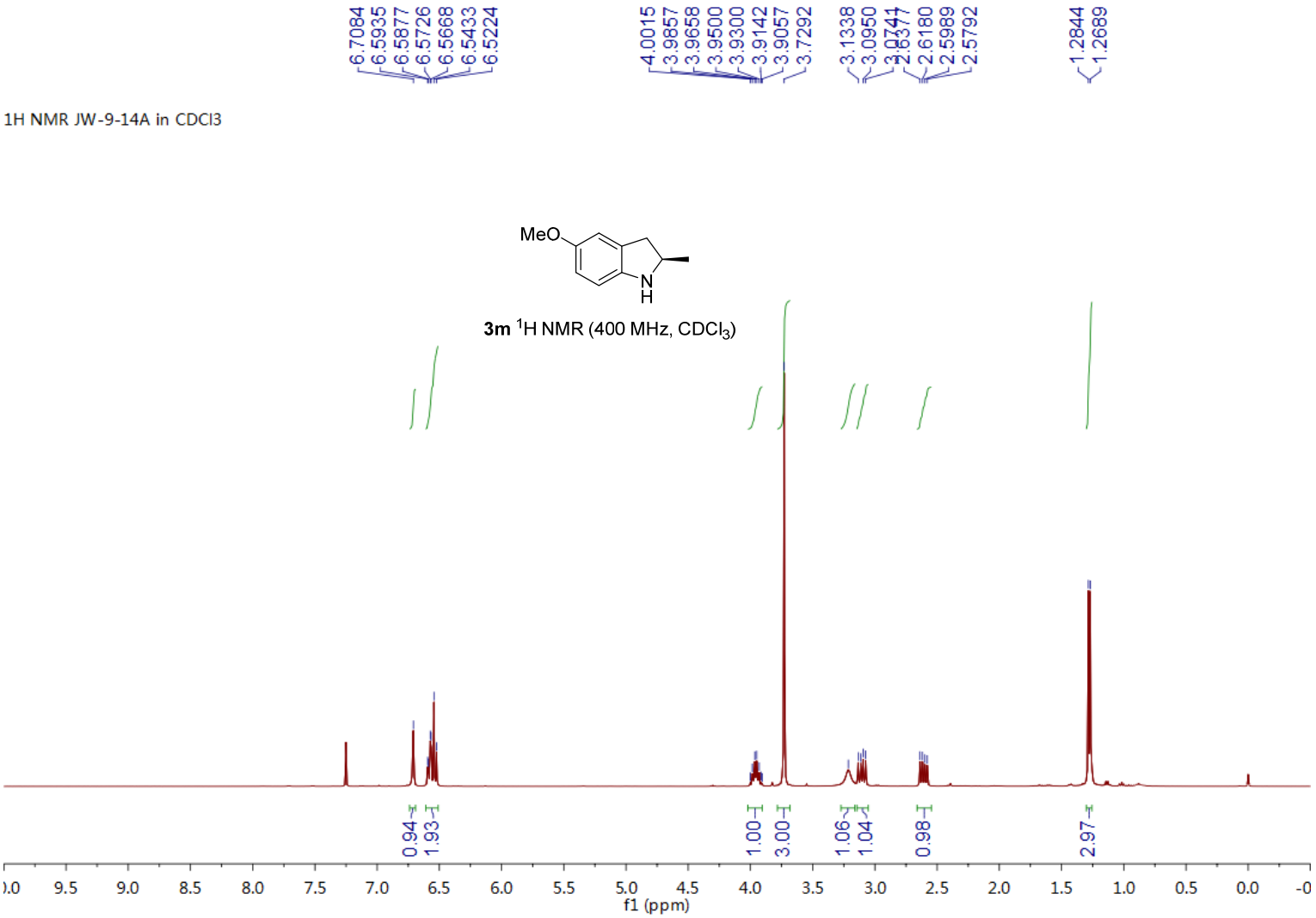

6.9392
6.9211
6.8533
6.8345
6.6508
6.6323
6.6138
4.0325
4.0170
4.0120
4.0013
3.9963
3.9913
3.9854
3.9808
3.9755
3.9651
3.9599
3.9443
3.1774
3.1560
3.1390
3.1176
2.6767
2.6565
2.1159
1.3115
1.2959
1H NMR CY-27-28A in CDCl3
N
H
3k 1H NMR (400 MHz, CDCl3)
1.92
0.97
1.00
0.88
0.99
0.97
2.93
3.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

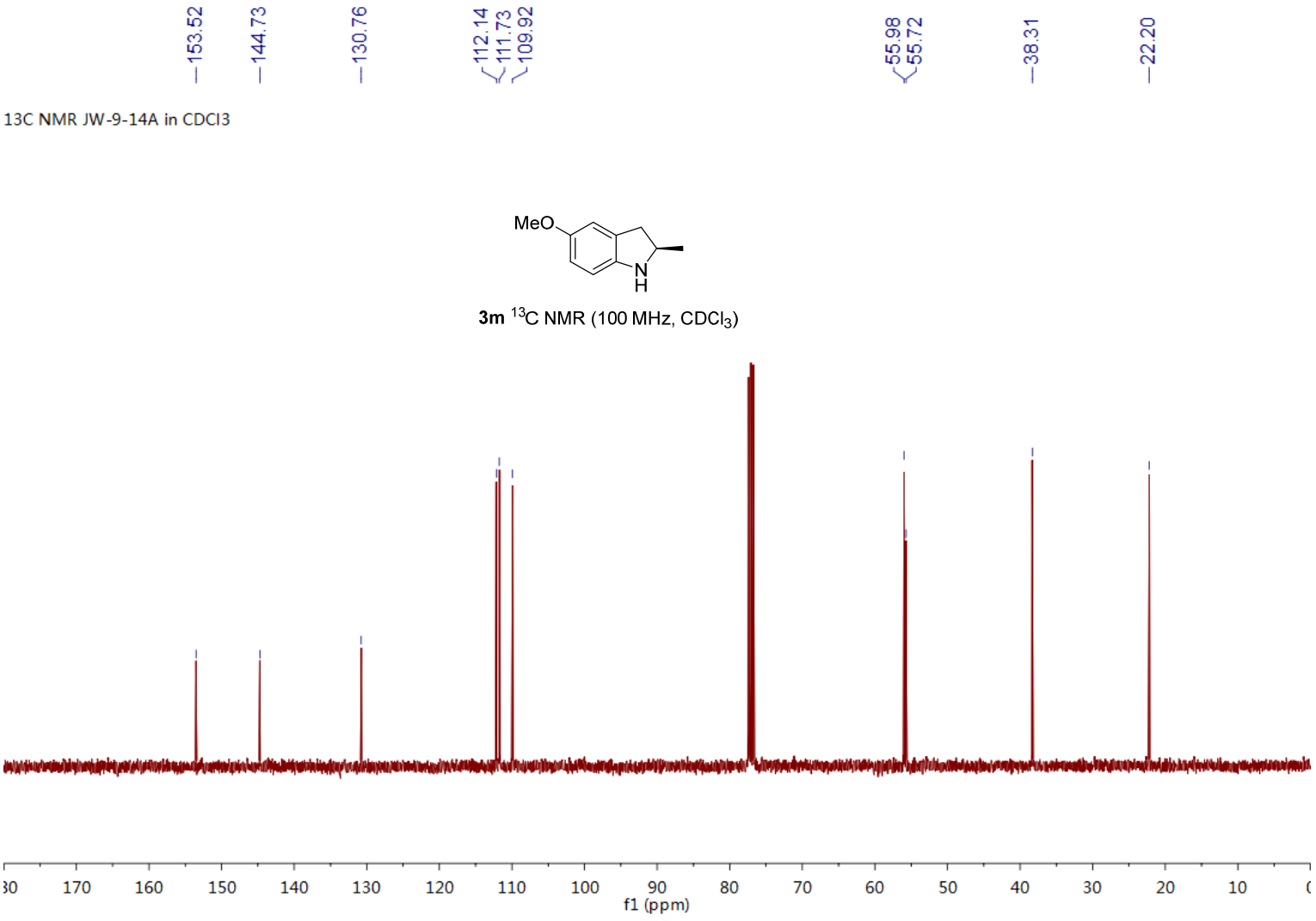

149.56
128.27
128.20
122.21
118.73
118.55
55.16
38.14
22.46
16.85
13C NMR CY-27-28A in CDCl3
N
H
3k 13C NMR (100 MHz, CDCl3)
13C NMR CY-27-28A in CDCl3
129
127
125
123
121
119
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


6.7445
6.7270
6.6964
6.6766
6.6590
6.6446
6.6262
4.0545
4.0389
4.0341
4.0233
4.0183
4.0135
4.0026
3.9977
3.9867
3.9821
3.9665
3.8062
3.5893
3.1516
2.6695
2.6511
2.6312
1.3063
1.2907
1H NMR JW-9-10A in CDCl3
N
H
OMe
3l 1H NMR (400 MHz, CDCl3)
0.93
1.93
1.01
3.00
0.60
1.03
0.98
2.96
f1 (ppm)



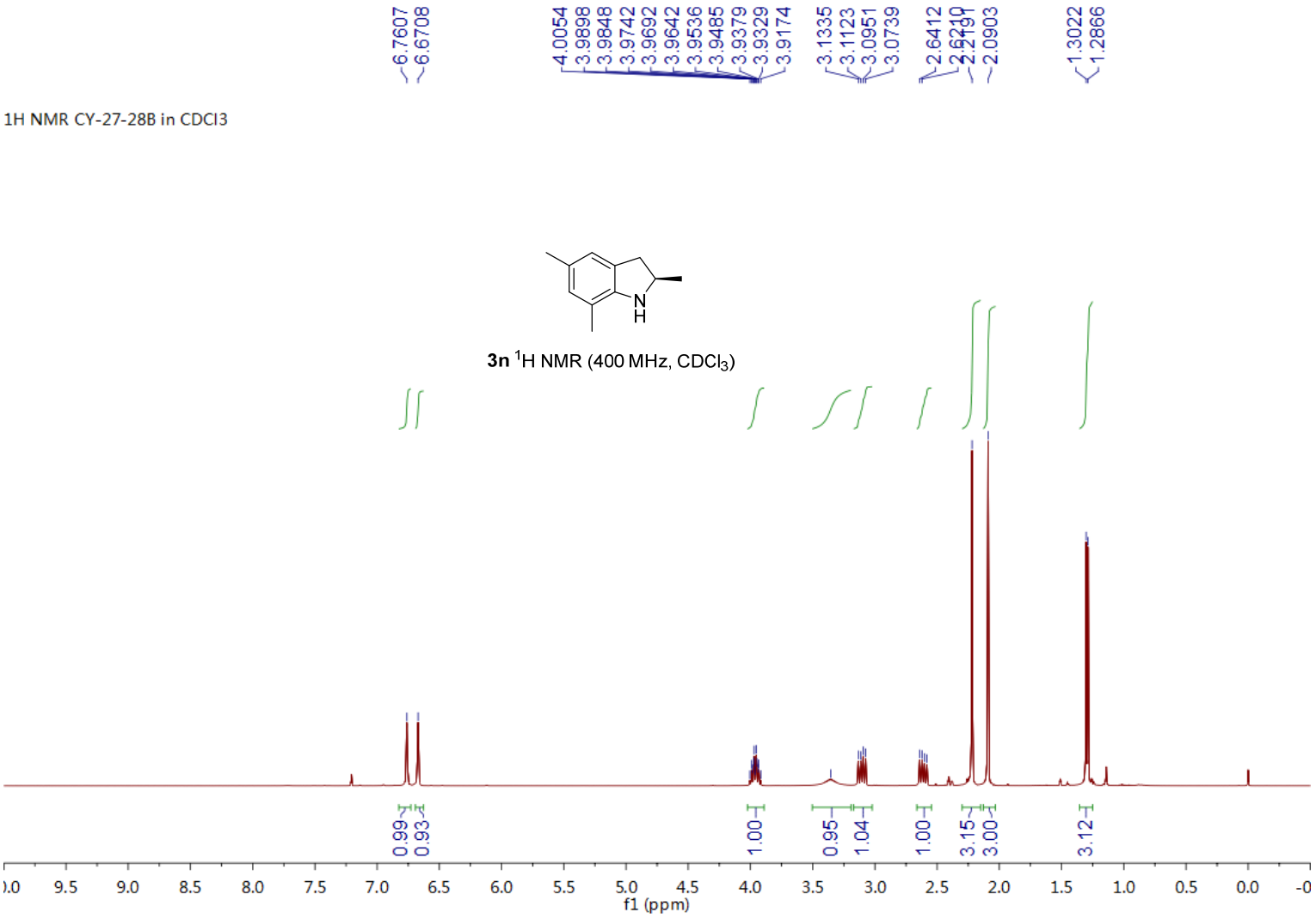
3l ^{13}C NMR (100 MHz, $CDCl_3$)


6.7084
6.5935
6.5877
6.5726
6.5668
6.5433
6.5224
4.0015
3.9857
3.9658
3.9500
3.9300
3.9142
3.9057
3.7292
3.1338
3.0950
2.6377
2.6180
2.5989
2.5792
1.2844
1.2689
1H NMR JW-9-14A in CDCl3
MeO
N
H
3m 1H NMR (400 MHz, CDCl3)
0.94
1.93
1.00
3.00
1.06
1.04
0.98
2.97
f1 (ppm)

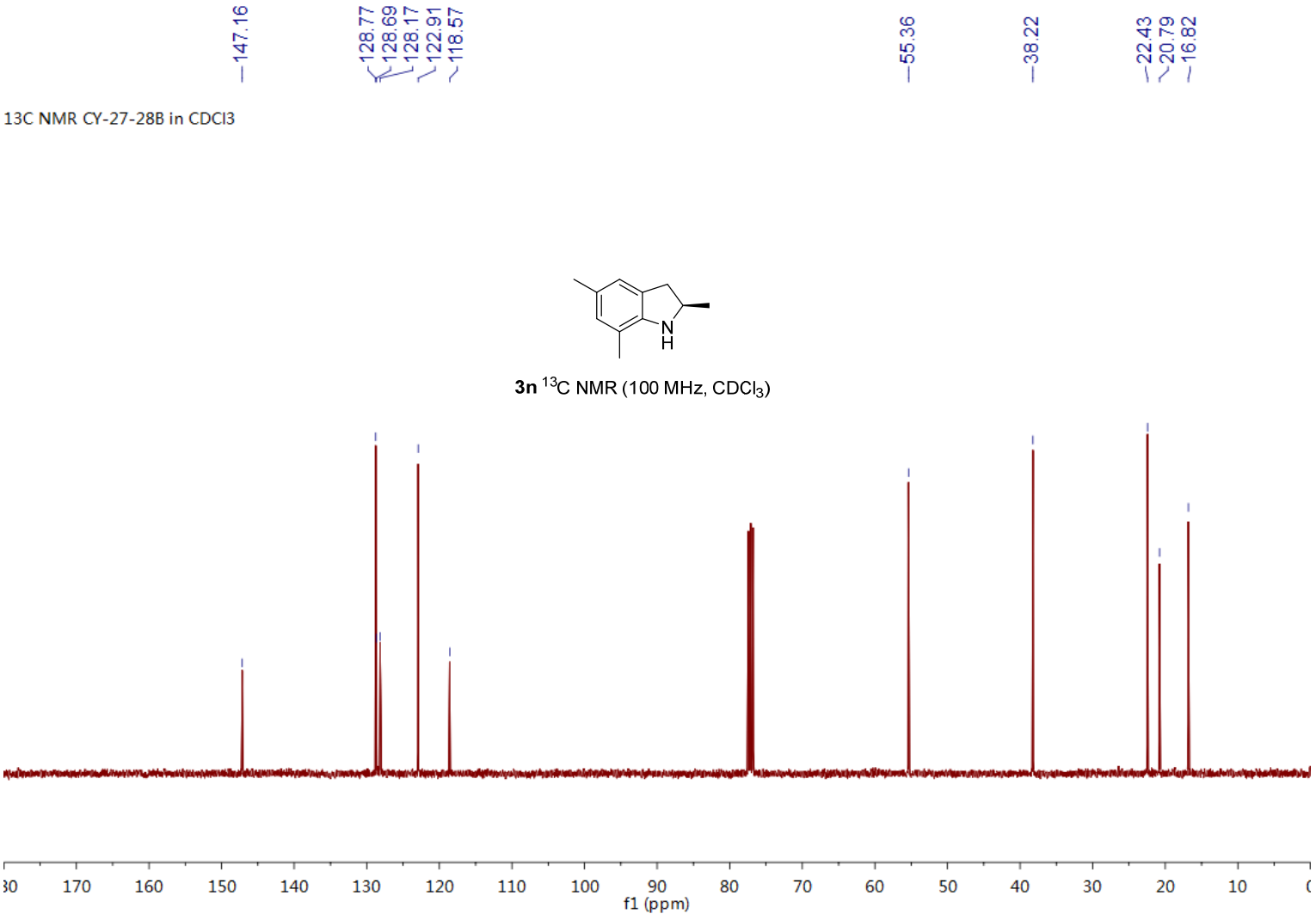


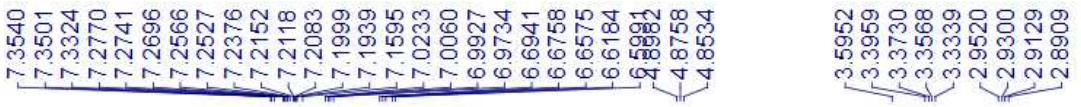
3m ^{13}C NMR (100 MHz, $CDCl_3$)

1H NMR CY-27-28B in CDCl3

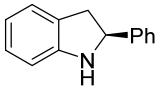


3n ^{1}H NMR (400 MHz, $CDCl_3$)

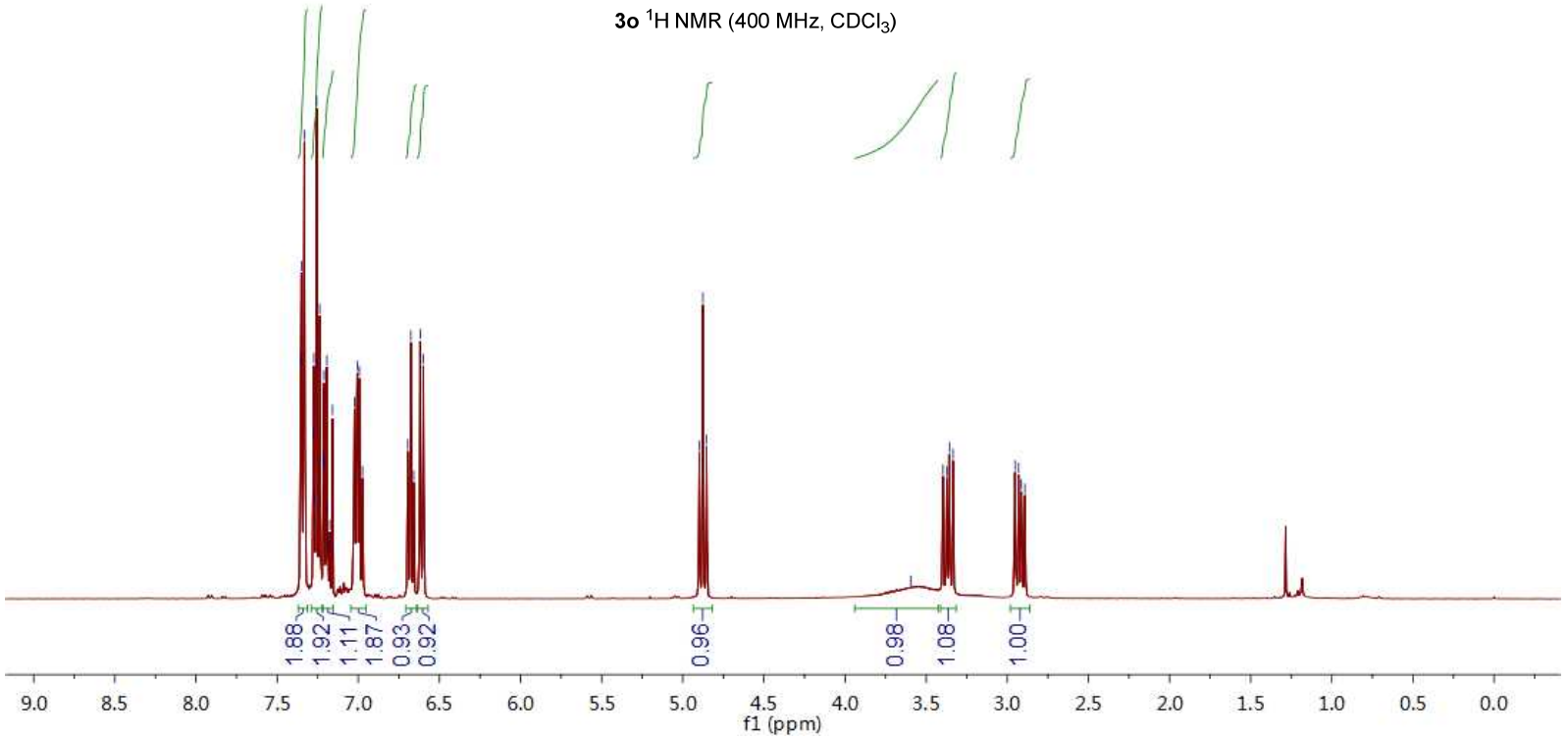

147.16
128.77
128.69
128.17
122.91
118.57
55.36
38.22
22.43
20.79
16.82
13C NMR CY-27-28B in CDCl3
N
H
3n 13C NMR (100 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



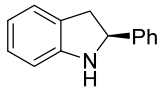
PROTON CDCI3 {D:\NMR400\02T4} nmr 21



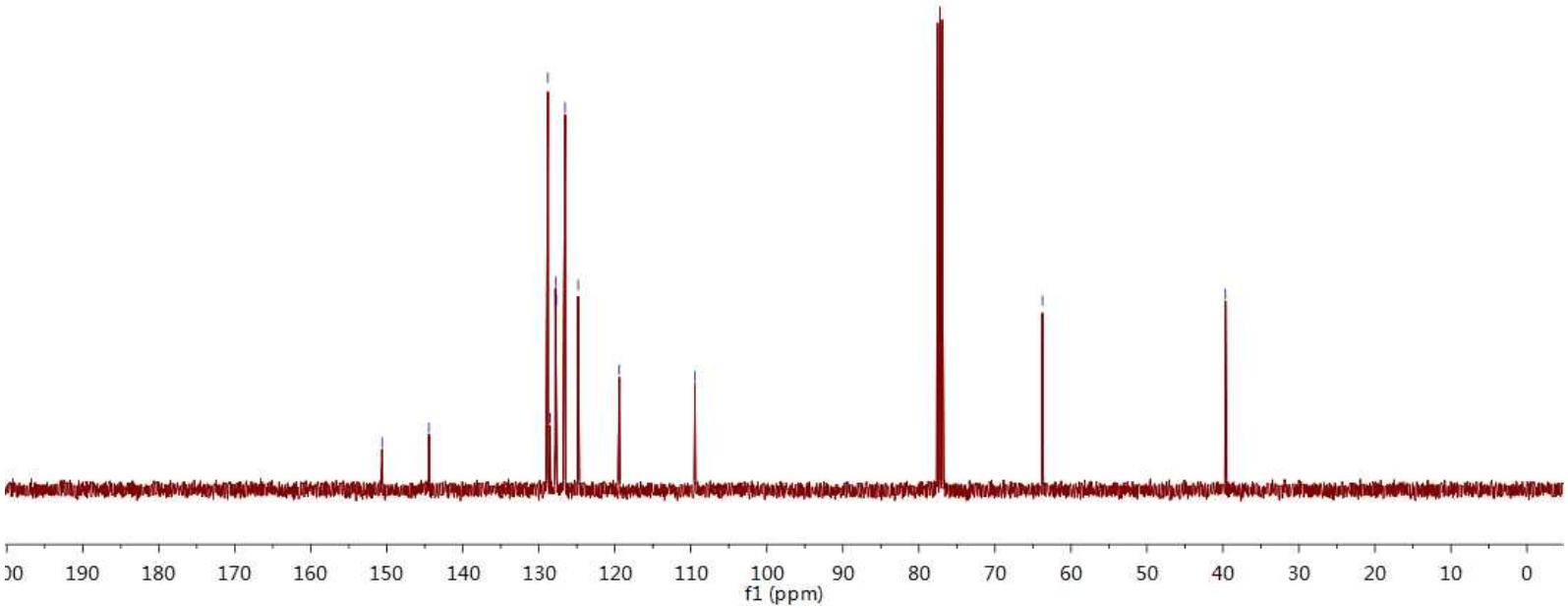
3o ^{1}H NMR (400 MHz, $CDCl_3$)

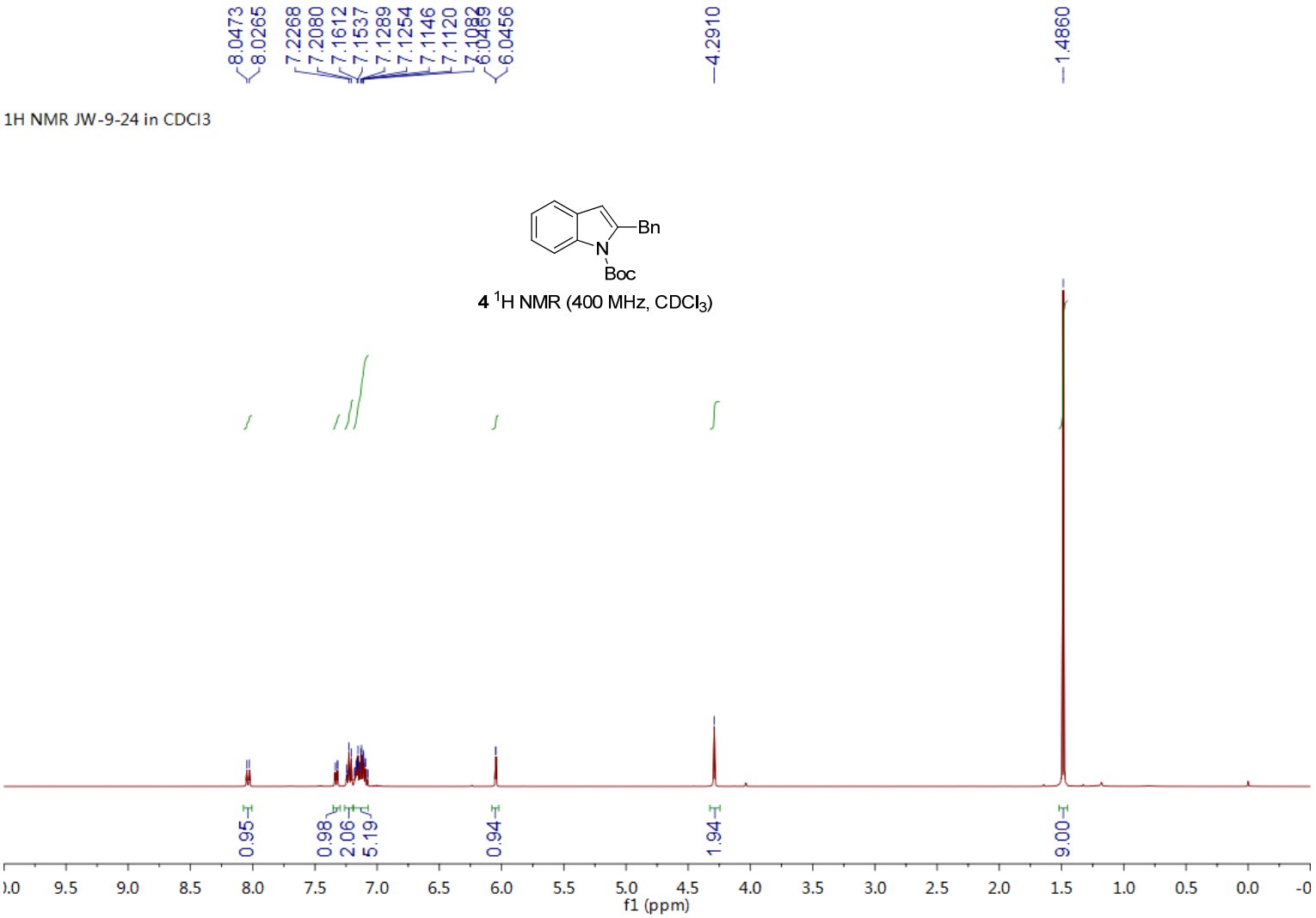


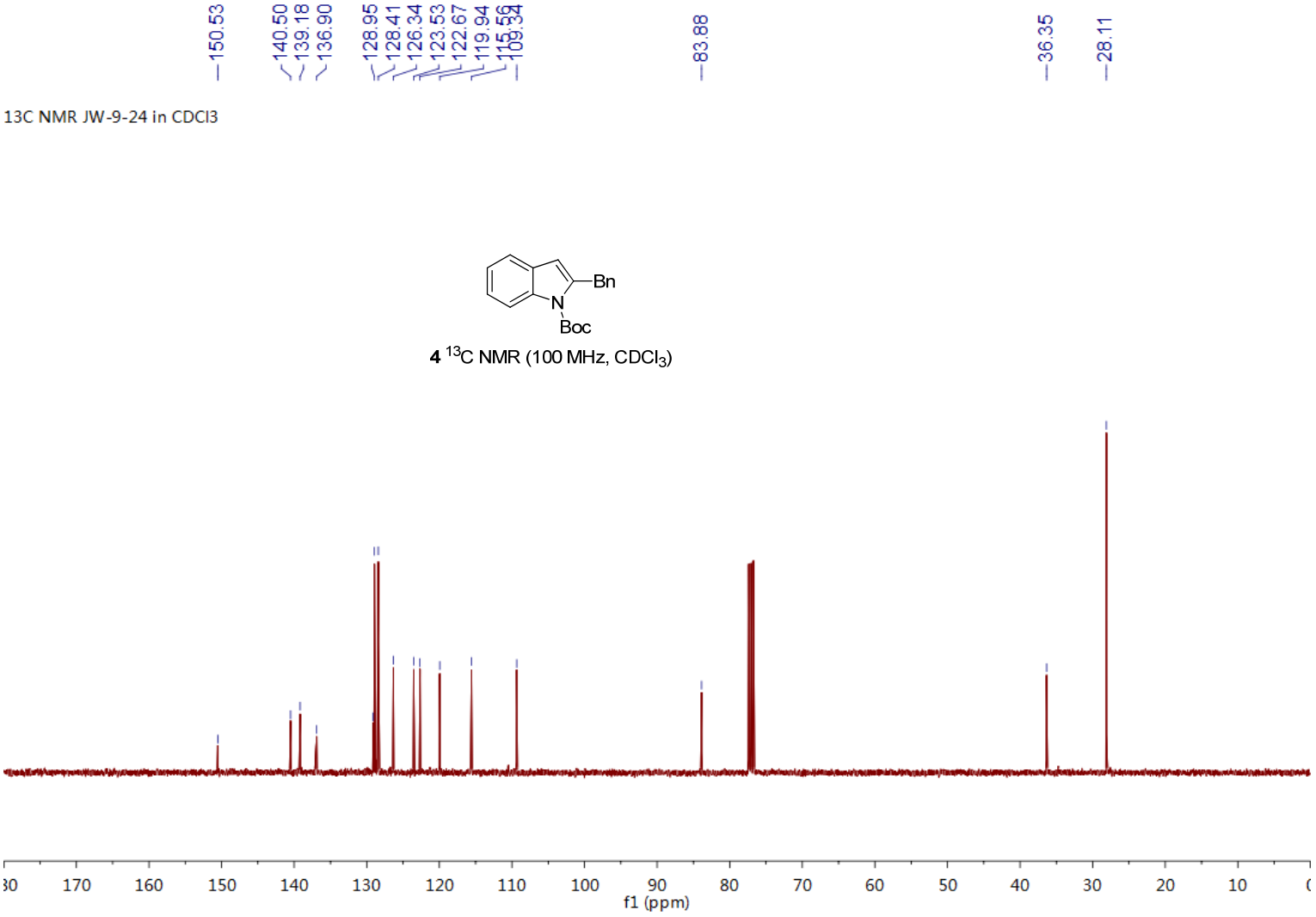
C13CPD CDCl3 {D:\NMR400\02T4} nmr 21



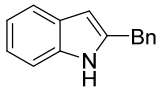
3o ^{13}C NMR (100 MHz, $CDCl_3$)



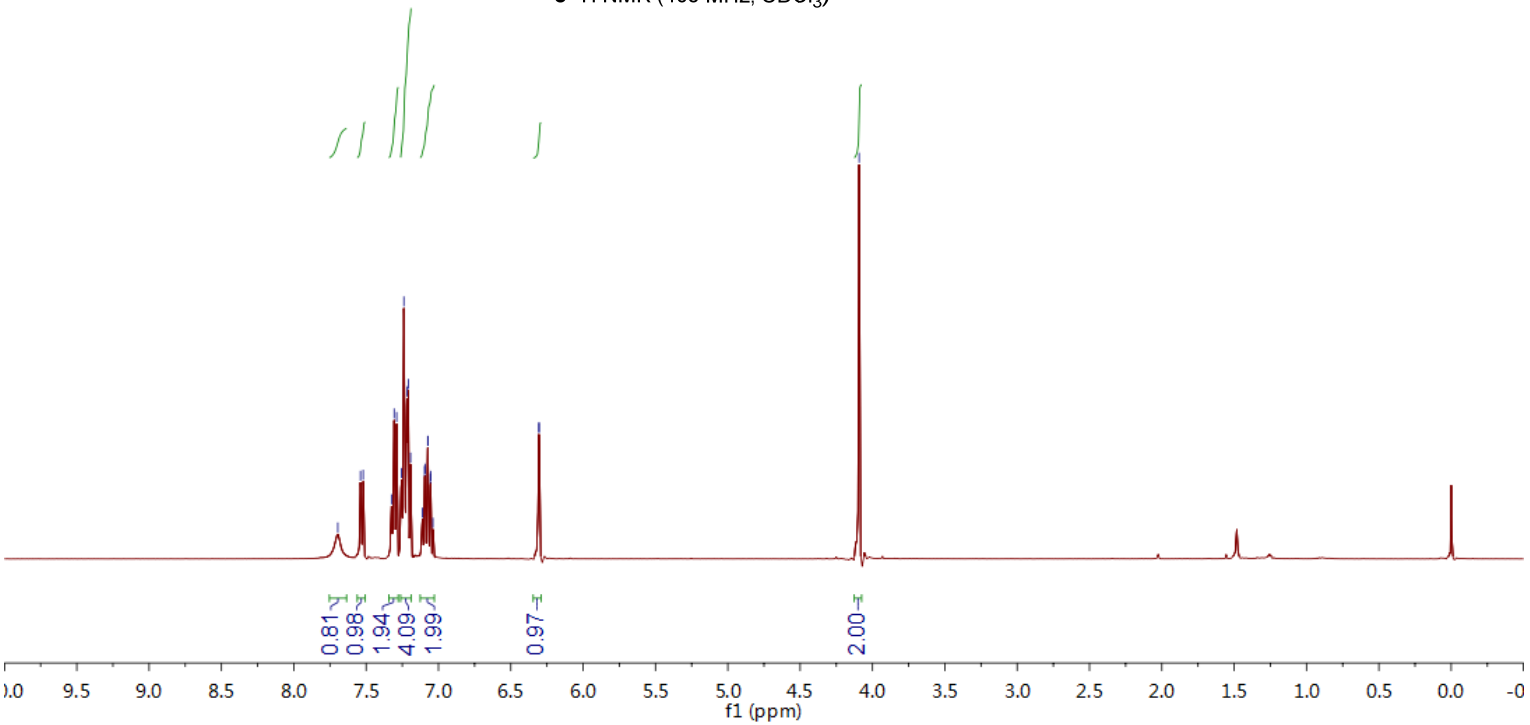

8.0473
8.0265
7.2268
7.2080
7.1612
7.1537
7.1289
7.1254
7.1146
7.1120
7.1082
6.0469
6.0456
4.2910
1.4860
1H NMR JW-9-24 in CDCl3
Bn
N
Boc
4 1H NMR (400 MHz, CDCl3)
0.95
0.98
2.06
5.19
0.94
1.94
9.00
f1 (ppm)

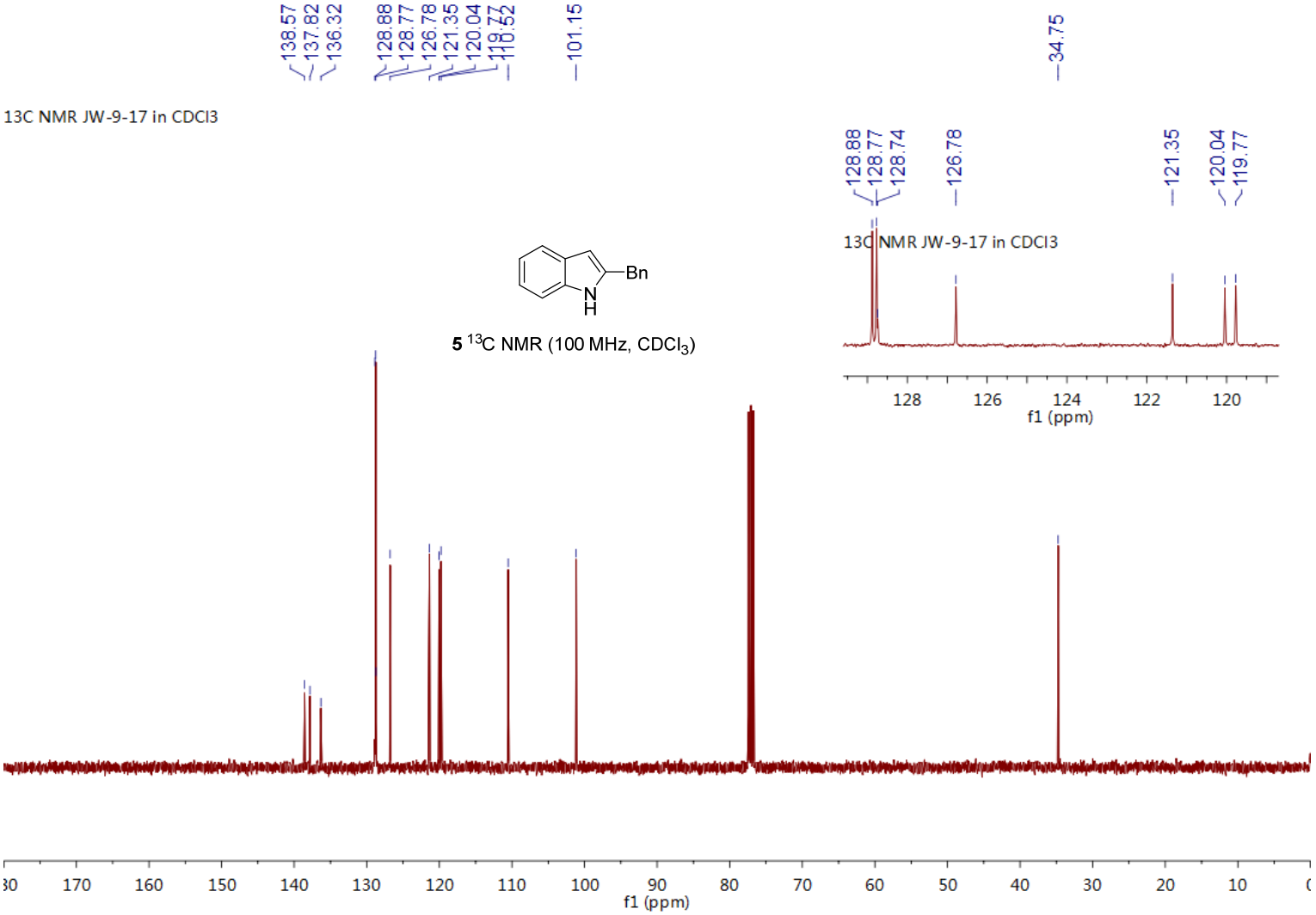

150.53
140.50
139.18
136.90
128.95
128.41
126.34
123.53
122.67
119.94
115.56
109.34
83.88
36.35
28.11
13C NMR JW-9-24 in CDCl3
Bn
N
Boc
4 13C NMR (100 MHz, CDCl3)
f1 (ppm)

1H NMR JW-9-17 in CDCl3



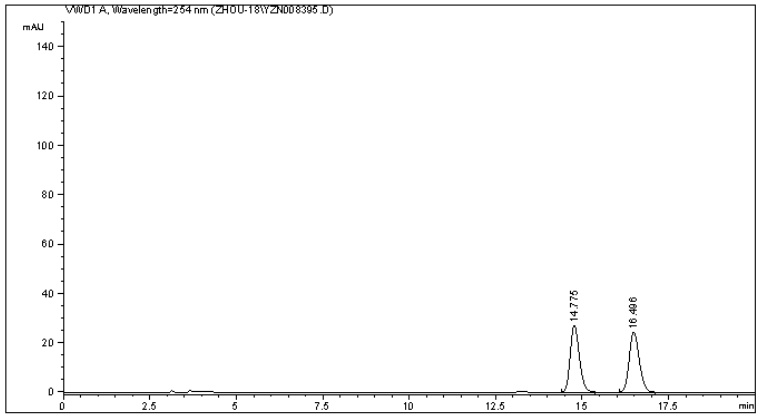
5 ^{1}H NMR (400 MHz, $CDCl_3$)



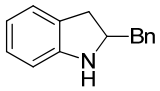

138.57
137.82
136.32
128.88
128.77
126.78
121.35
120.04
119.77
118.52
101.15
34.75
13C NMR JW-9-17 in CDCl3
Bn
N
H
5 13C NMR (100 MHz, CDCl3)
128.88
128.77
128.74
126.78
121.35
120.04
119.77
13C NMR JW-9-17 in CDCl3
128
126
124
122
120
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008395.D
Sample Name: CY-26-101+-

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/6/2018 3:07:39 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/6/2018 3:04:56 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/6/2018 3:36:04 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0mL/min, 30 oC, 254 nm
```



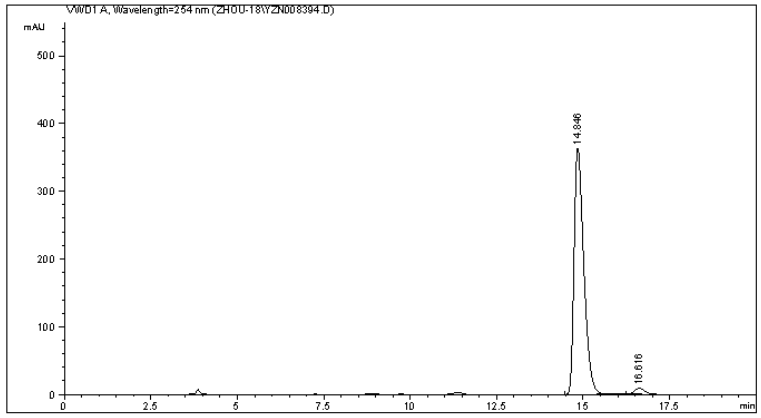

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.775 BB    0.2846  500.49707   27.19790  50.3035
   2  16.496 BB    0.3147  494.45779   24.43822  49.6965

Totals :                   994.95486   51.63612

                          *** End of Report ***
```



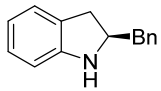
(±)-**3a**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008394.D
Sample Name: CY-26-101

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/6/2018 2:44:56 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/6/2018 2:21:23 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/6/2018 3:34:45 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0mL/min, 30 oC, 254 nm
```



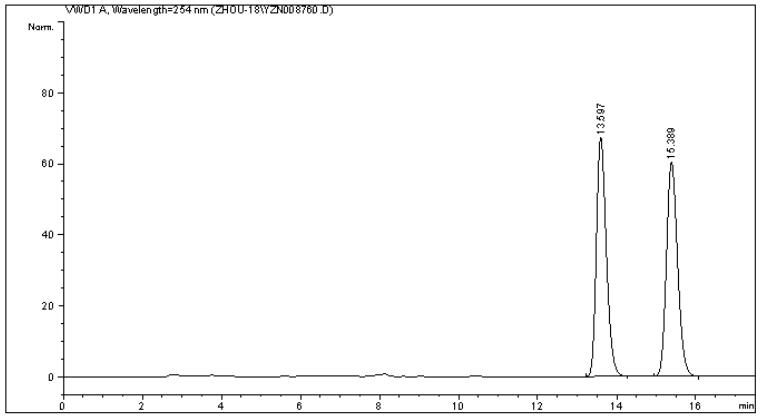

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.846 BB    0.2992 7110.40674  364.07016  97.4869
   2  16.616 BB    0.3163  183.29590    8.99711   2.5131

Totals :                  7293.70264  373.06727

                          *** End of Report ***
```



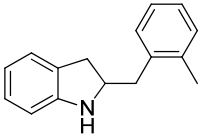
(+)-**3a**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008760.D
Sample Name: jw-9-11A(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/3/2018 4:27:27 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/3/2018 4:25:15 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:11:59 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.597 BB    0.2699 1177.79517   67.23775  49.9936
   2  15.389 BB    0.3021 1178.09595   60.31648  50.0064

Totals :                  2355.89111  127.55423

                          *** End of Report ***
```

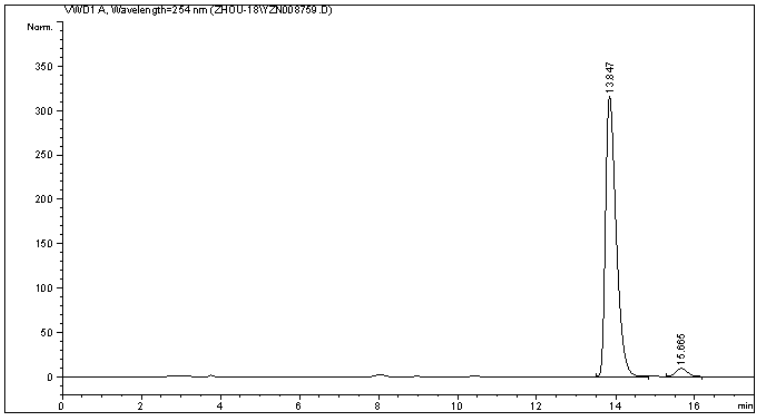


(±)-**3b**

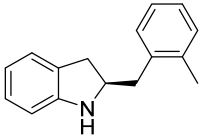
Instrument 1 6/10/2018 4:12:01 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008759.D
Sample Name: jw-9-11A

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/3/2018 4:06:08 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/3/2018 4:02:59 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:11:10 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.847 BB    0.2861 5887.67676  315.56094  96.9825
   2  15.665 BB    0.3069  183.19060    9.24295   3.0175

Totals :                  6070.86736  324.80389

                          *** End of Report ***
```

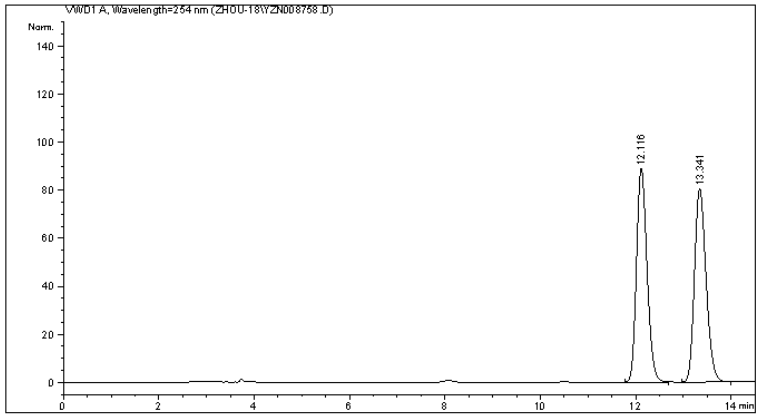


(+)-**3b**

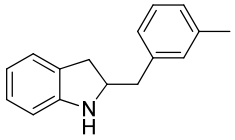
Instrument 1 6/10/2018 4:11:17 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008758.D
Sample Name: jw-9-11B(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/3/2018 3:45:42 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/3/2018 3:44:03 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:13:53 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



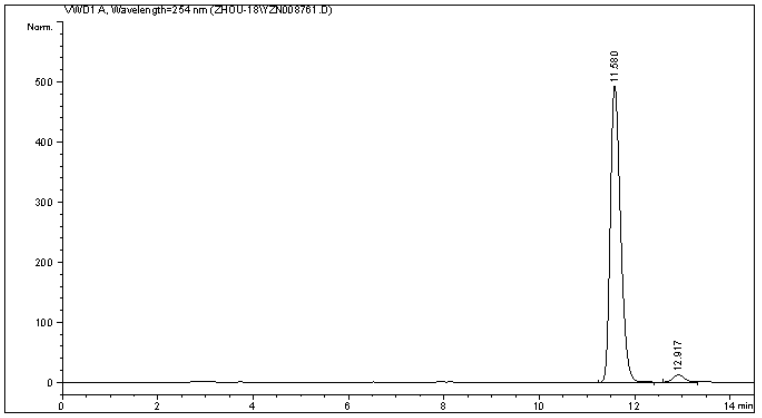

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.116 BB    0.2374 1365.78735   88.94563  50.0283
   2  13.341 BB    0.2632 1364.24390   80.52128  49.9717

Totals :                  2730.03125  169.46690

                          *** End of Report ***
```



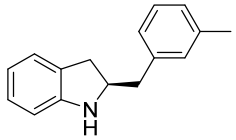
(±)-**3c**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008761.D
Sample Name: jw-9-11B

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/3/2018 4:48:00 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/3/2018 4:45:48 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:13:01 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



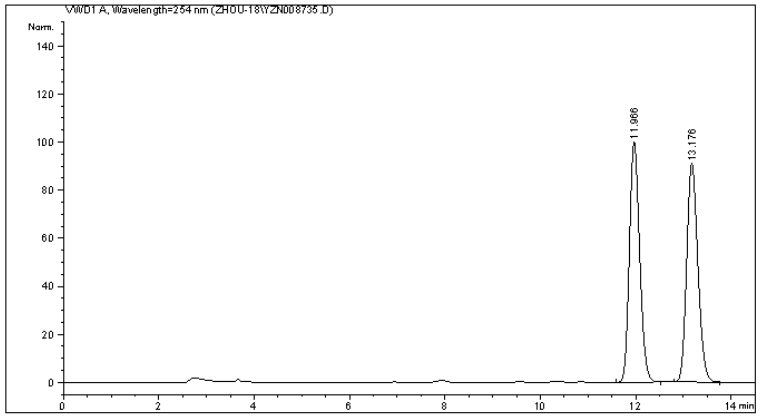

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.580 BB    0.2292 7352.47070  493.38843  97.3795
   2  12.917 BV    0.2506  197.85622   12.18607   2.6205

Totals :                  7550.32692  505.57450

                          *** End of Report ***
```



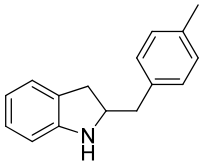
(+)-**3c**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008735.D
Sample Name: JW-9-10B(+-)

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 6/1/2018 8:17:02 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 6/1/2018 8:14:26 PM
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 6/10/2018 4:07:40 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



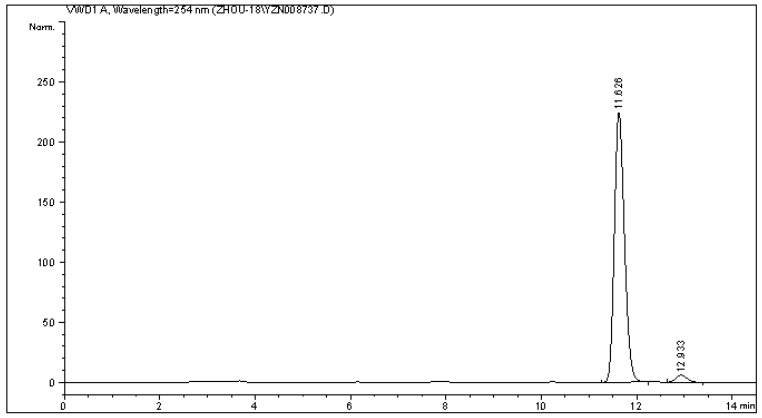

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.966 BB    0.2309 1489.76465   99.82134  50.0914
       2  13.176 BB    0.2532 1484.32629   90.84733  49.9086

    Totals :                  2974.09094  190.66867

    ======================================================================
                              *** End of Report ***
```



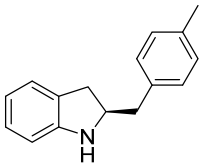
(±)-**3d**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008737.D
Sample Name: JW-9-10B

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 6/1/2018 8:55:17 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
    Last changed    : 6/1/2018 8:53:33 PM by
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 6/10/2018 4:05:11 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



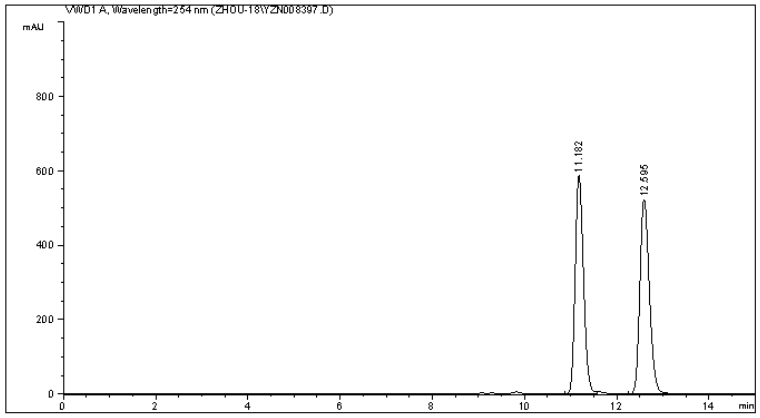

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.626 BB    0.2278 3290.12695  224.42123  97.2877
       2  12.933 BB    0.2442   91.72623    5.84698   2.7123

    Totals :                  3381.85318  230.26822

    ======================================================================
                              *** End of Report ***
```



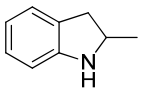
(+)-**3d**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008397.D
Sample Name: CY-27-6+-

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/6/2018 3:59:20 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/6/2018 3:54:53 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 10:47:28 AM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 97/3, 0.8 mL/min, 30 oC, 254 nm
```



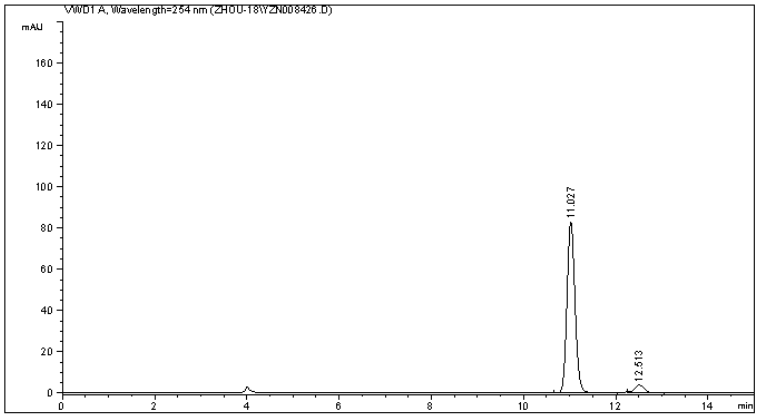

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.182 BV    0.1944 7358.73682  586.76031  49.6127
   2  12.595 BB    0.2221 7473.63721  522.89587  50.3873

Totals :                  1.48324e4  1109.65619

                          *** End of Report ***
```



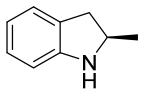
(±)-**3e**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008426.D
Sample Name: CY-27-6

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/8/2018 11:05:05 AM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 10:45:15 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 11:26:22 AM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 97/3, 0.8 mL/min, 30 oC, 254 nm
```



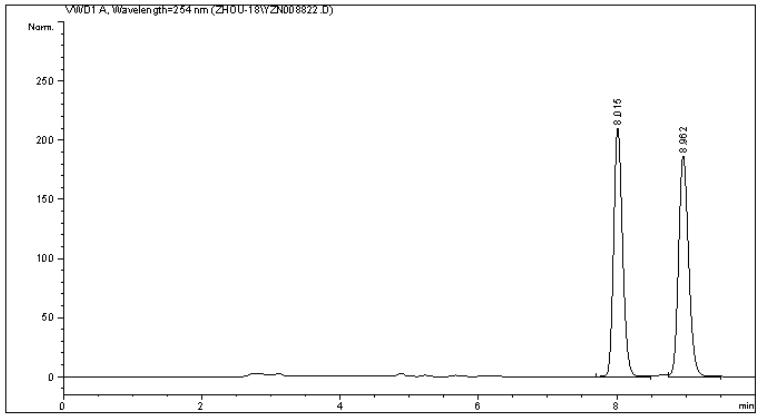

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.027 MM R  0.2022 1007.92560   83.06752  94.8035
   2  12.513 MM R  0.2392   55.24738    3.84912   5.1965

Totals :                  1063.17298   86.91664

                          *** End of Report ***
```



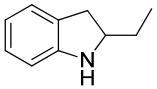
(+)-**3e**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008822.D
Sample Name: JW-9-14B(+-)

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 6/6/2018 5:43:46 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
    Last changed    : 6/6/2018 5:40:47 PM by
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 6/10/2018 4:16:13 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



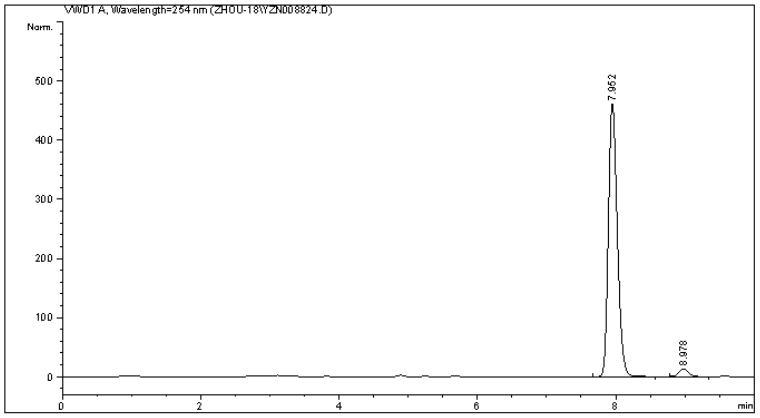

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   8.015 BB    0.1390 1876.17773  210.42880  49.9859
       2   8.962 VB    0.1552 1877.23914  186.71622  50.0141

    Totals :                  3753.41687  397.14502

    ======================================================================
                              *** End of Report ***
```



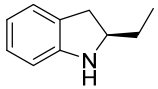
(±)-3f

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008824.D
Sample Name: JW-9-15B

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 6/6/2018 6:09:12 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
    Last changed    : 6/6/2018 6:06:59 PM by
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 6/10/2018 4:15:24 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



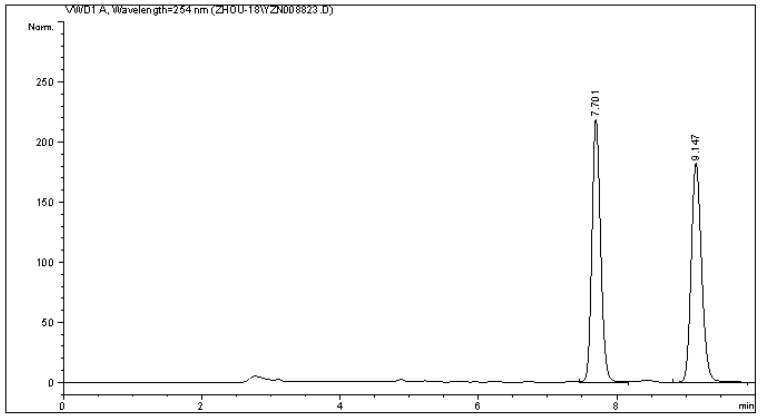

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   7.952 BB    0.1381 4146.00977  462.17194  96.8127
       2   8.978 BB    0.1569  136.49561   13.37699   3.1873

    Totals :                  4282.50537  475.54893

    ======================================================================
                              *** End of Report ***
```



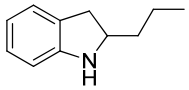
(+)-3f

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008823.D
Sample Name: JW-9-15C(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/6/2018 5:55:57 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/6/2018 5:54:24 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:17:59 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



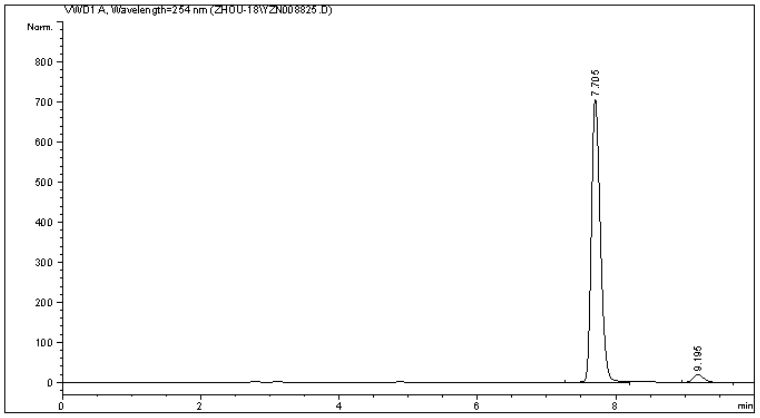

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.701 VV    0.1352 1909.96460  219.01628  50.0737
   2   9.147 VB    0.1613 1904.33984  182.16226  49.9263

Totals :                  3814.30444  401.17854

                          *** End of Report ***
```



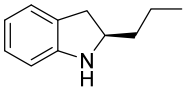
(±)-**3g**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008825.D
Sample Name: JW-9-15C

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/6/2018 6:21:08 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/6/2018 6:19:14 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:17:10 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



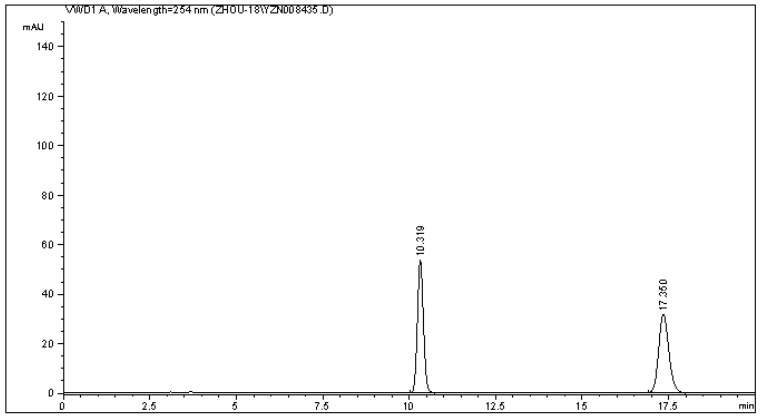

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.705 BV    0.1387 6382.35449  707.82910  96.8564
   2   9.195 BB    0.1646  207.14548   19.50937   3.1436

Totals :                  6589.49997  727.33847

                          *** End of Report ***
```



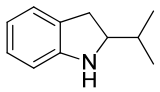
(+)-**3g**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008435.D
Sample Name: CY-27-13+-

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/9/2018 5:51:56 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/9/2018 5:49:52 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/9/2018 6:12:16 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.319 BB    0.1875  647.21832   53.59042  50.0688
   2  17.350 BB    0.3145  645.44043   31.73360  49.9312

Totals :                  1292.65875   85.32402
```

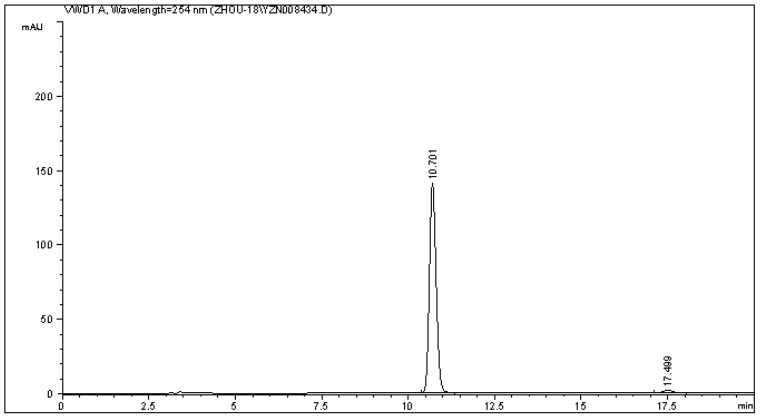


(±)-**3h**

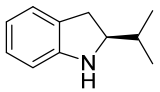
```
                          *** End of Report ***
```


Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008434.D
Sample Name: CY-27-13

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/9/2018 5:29:43 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/9/2018 5:09:32 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/9/2018 5:52:17 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.701 BB    0.2109 1916.72522  141.05399  98.1376
   2  17.499 BB    0.3154   36.37471    1.73852   1.8624

Totals :                  1953.09993  142.79251
```

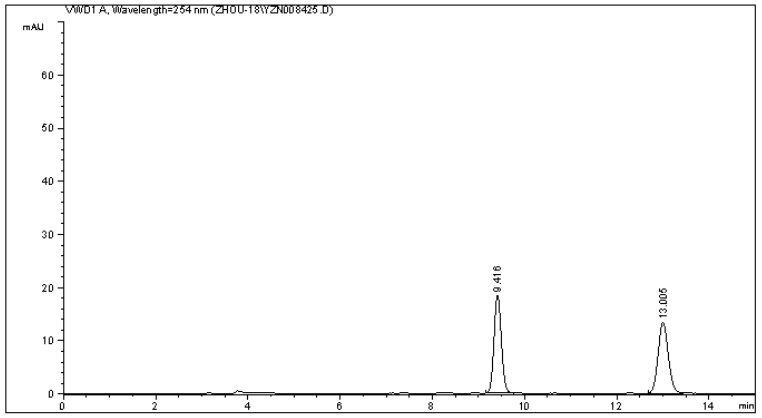


(+)-**3h**

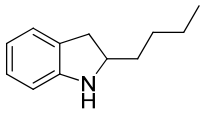
```
                          *** End of Report ***
```

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008425.D
Sample Name: CY-27-11+-

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 5/8/2018 10:17:36 AM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/8/2018 10:15:48 AM
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/8/2018 10:48:54 AM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```



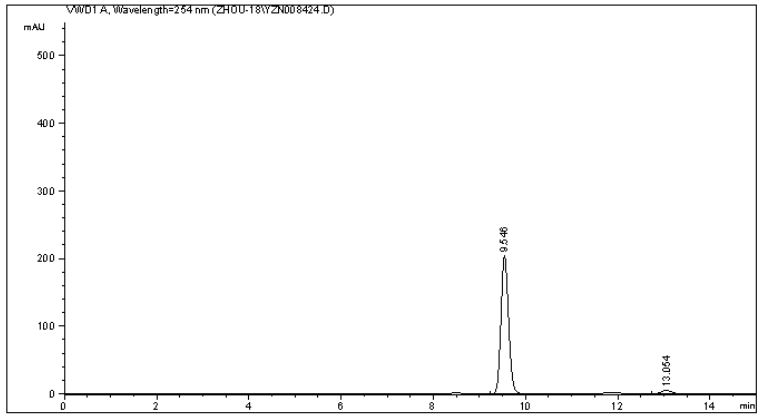

```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.416 BB    0.1714  204.64835   18.48359  50.1908
       2  13.005 BB    0.2356  203.09268   13.35832  49.8092

    Totals :                  407.74103   31.84191

    ======================================================================
                               *** End of Report ***
```



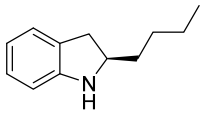
(±)-**3i**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008424.D
Sample Name: CY-27-11

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 5/8/2018 9:54:25 AM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/8/2018 9:51:06 AM
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/8/2018 9:05:17 AM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```



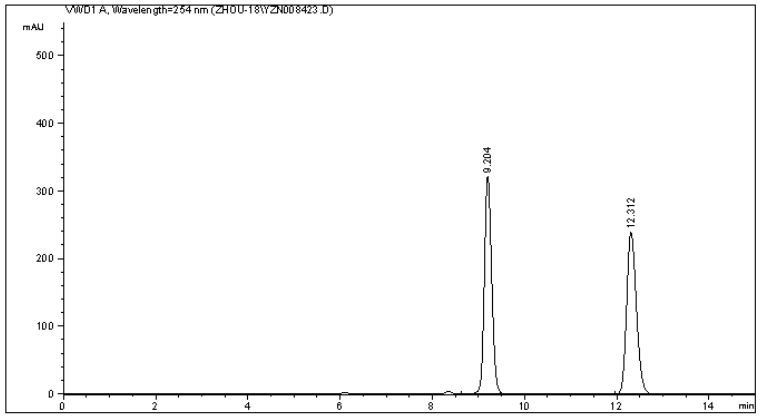

```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.546 VB    0.1779 2323.77856  204.19144  96.8279
       2  13.054 VB    0.2345   76.12829    5.03897   3.1721

    Totals :                 2399.90685  209.23041

    ======================================================================
                               *** End of Report ***
```



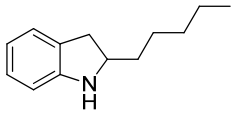
(+)-**3i**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008423.D
Sample Name: CY-27-8+-

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/8/2018 9:25:57 AM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 9:24:35 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 9:05:17 AM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.204 VV    0.1704 3538.93457  322.22589  50.2287
   2  12.312 BB    0.2276 3506.70923  239.50800  49.7713

Totals :                  7045.64380  561.73389
```

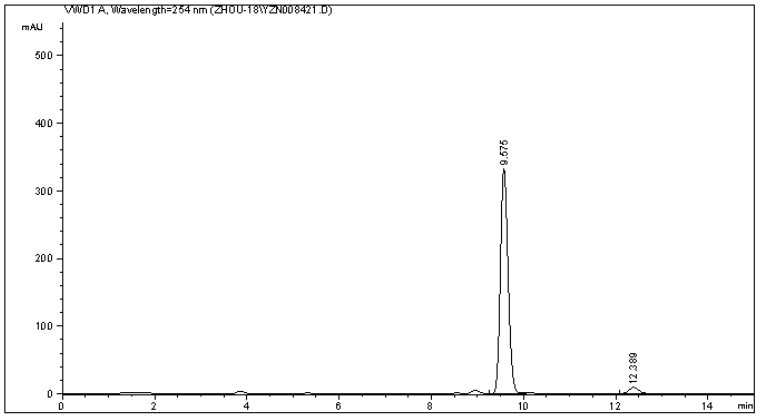


(±)-**3j**

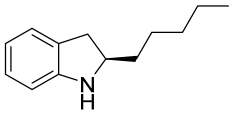
```
                          *** End of Report ***
```


Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008421.D
Sample Name: CY-27-8

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/8/2018 8:45:32 AM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 8:41:47 AM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/8/2018 9:05:17 AM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.575 VB    0.1800 3849.57104  332.95560  96.7103
   2  12.389 BB    0.2282  130.94591    8.91416   3.2897

Totals :                  3980.51695  341.86976
```

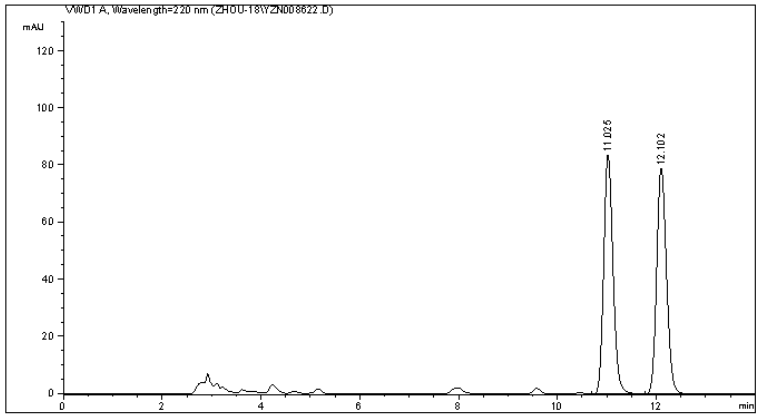


(+)-**3j**

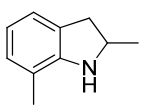
```
                          *** End of Report ***
```

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008622.D
Sample Name: CY-27-28A+/-

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 5/22/2018 1:26:59 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/22/2018 1:23:45 PM
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/22/2018 2:05:08 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.025 VB    0.2022 1095.52393   83.69418  50.2155
       2  12.102 BB    0.2145 1086.12134   78.89933  49.7845

    Totals :                  2181.64526  162.59351

    ======================================================================
                              *** End of Report ***
```

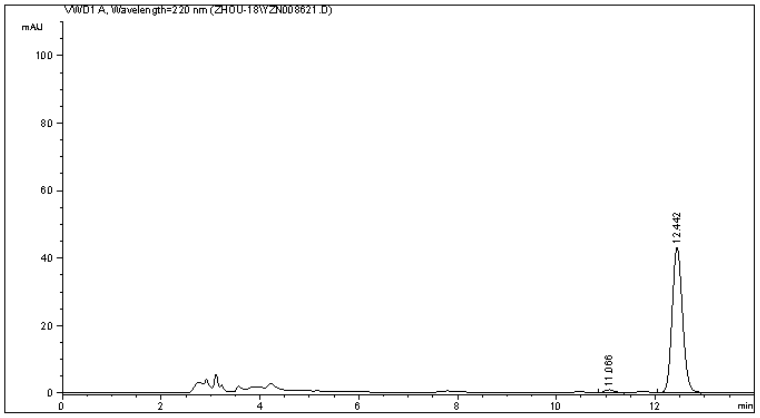


(±)-**3k**

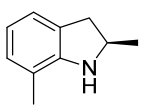
Instrument 1 5/22/2018 2:05:13 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008621.D
Sample Name: CY-27-28A

```
    ======================================================================
    Acq. Operator   :
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 5/22/2018 1:07:09 PM
    Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/22/2018 12:44:05 PM
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
    Last changed    : 5/22/2018 2:07:03 PM
                      (modified after loading)
    Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.066 MM R  0.2283   12.11573 8.84545e-1   1.9035
       2  12.442 VB    0.2257  624.36621   43.11478  98.0965

    Totals :                  636.48194   43.99933

    ======================================================================
                              *** End of Report ***
```

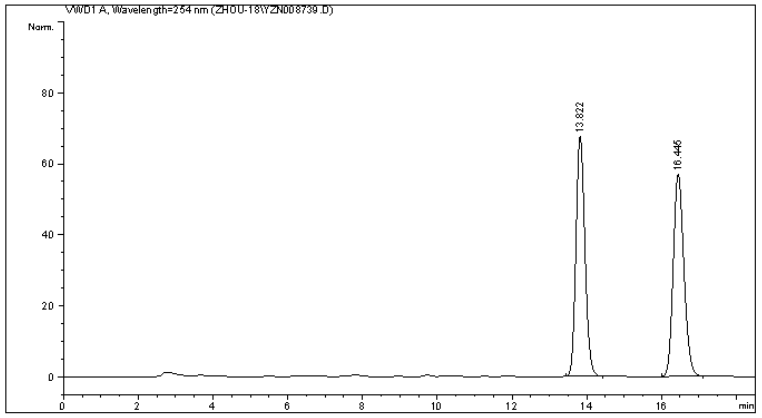


(+)-**3k**

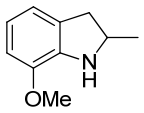
Instrument 1 5/22/2018 2:07:07 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008739.D
Sample Name: JW-9-10A(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/1/2018 9:32:39 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/1/2018 9:31:17 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:09:58 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



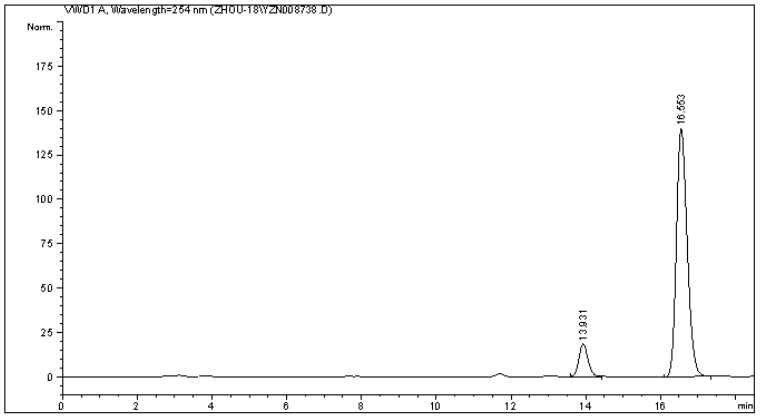

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.822 BB    0.2571 1127.22070   67.63029  49.9487
   2  16.445 BB    0.3069 1129.53516   56.99560  50.0513

Totals :                  2256.75586  124.62589

                          *** End of Report ***
```



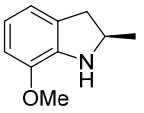
(±)-**3l**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008738.D
Sample Name: JW-9-10A

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/1/2018 9:12:26 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/1/2018 9:10:30 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:09:03 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =99/1, 1.0 mL/min, 30 oC, 254 nm
```



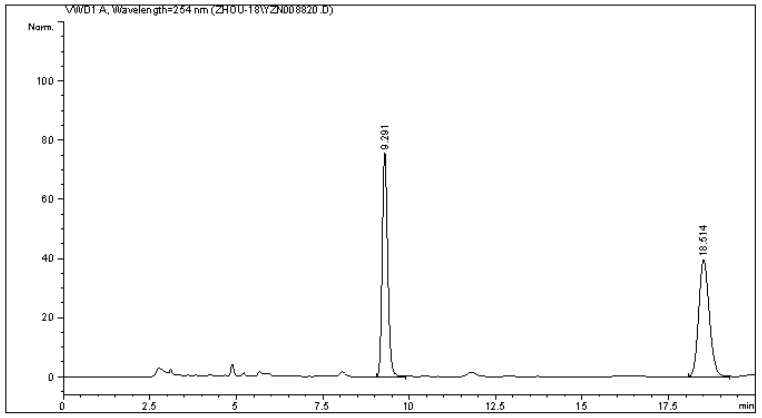

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.931 BB    0.2565  305.32391   18.37258   9.8454
   2  16.553 BB    0.3091 2795.86841  139.75655  90.1546

Totals :                  3101.19232  158.12912

                          *** End of Report ***
```



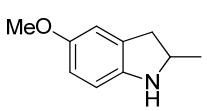
(+)-**3l**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008820.D
Sample Name: JW-9-14A(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/6/2018 4:55:11 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/6/2018 4:44:51 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:20:09 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



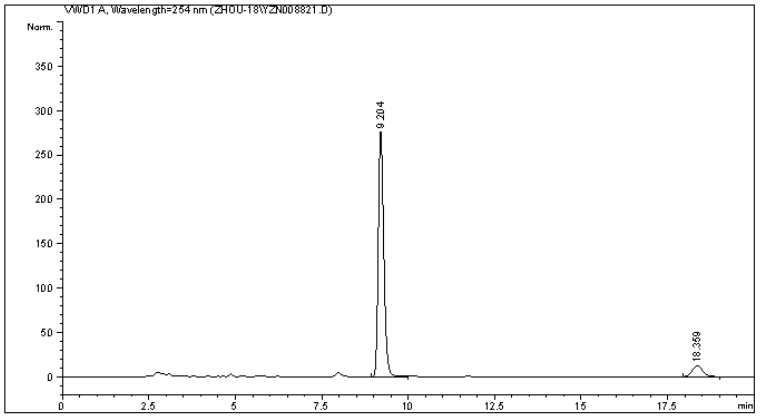

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.291 BB    0.1691  833.34967   75.74040  49.7126
   2  18.514 BB    0.3308  842.98486   39.46210  50.2874

Totals :                  1676.33453  115.20250

                          *** End of Report ***
```



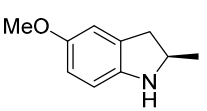
(±)-**3m**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008821.D
Sample Name: JW-9-14A

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/6/2018 5:20:37 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF LC.M
Last changed    : 6/6/2018 5:16:14 PM by
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 6/10/2018 4:19:10 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH =95/5, 1.0 mL/min, 30 oC, 254 nm
```



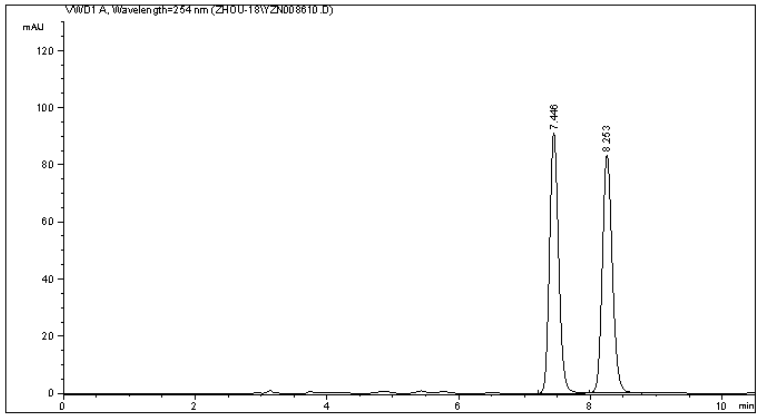

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.204 BB    0.1696 3054.58105  276.70883  92.0863
   2  18.359 BB    0.3305  262.50253   12.30077   7.9137

Totals :                  3317.08359  289.00960

                          *** End of Report ***
```



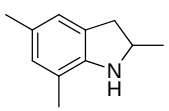
(+)-**3m**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008610.D
Sample Name: CY-27-28B(+-)

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/21/2018 8:56:36 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/21/2018 8:55:06 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/22/2018 12:40:02 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```



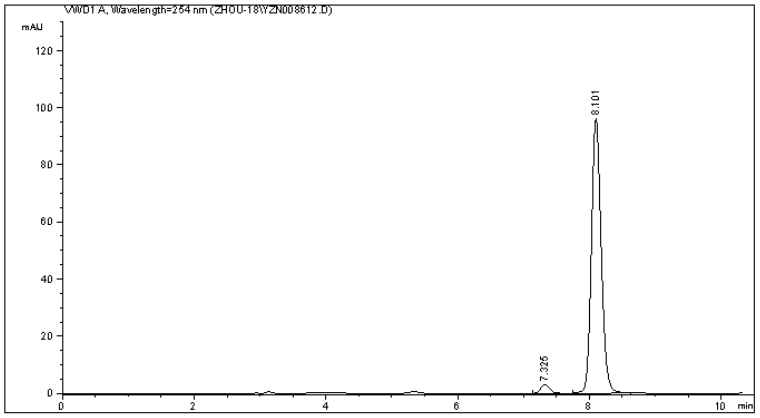

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.446 BB    0.1477  870.81885   91.25665  49.8177
   2   8.253 BB    0.1633  877.19373   83.50922  50.1823

Totals :                  1748.01257  174.76587

                          *** End of Report ***
```



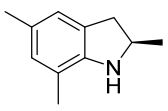
(±)-**3n**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN008612.D
Sample Name: CY-27-28B

```
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/21/2018 9:26:31 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/21/2018 9:24:44 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed    : 5/22/2018 12:40:02 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm
```



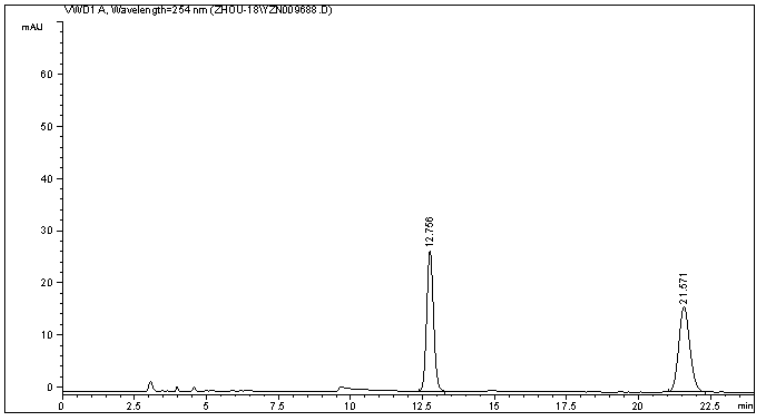

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.325 BB    0.1453   28.57020    3.06216   2.7987
   2   8.101 BB    0.1598  992.28149   96.09990  97.2013

Totals :                  1020.85170   99.16206

                          *** End of Report ***
```



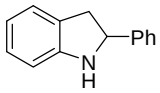
(+)-**3n**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN009688.D
Sample Name: CY-28-33+-

```
==========================================================================
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/2/2018 5:24:52 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed    : 8/2/2018 5:21:43 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed    : 8/2/2018 7:31:16 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm
```



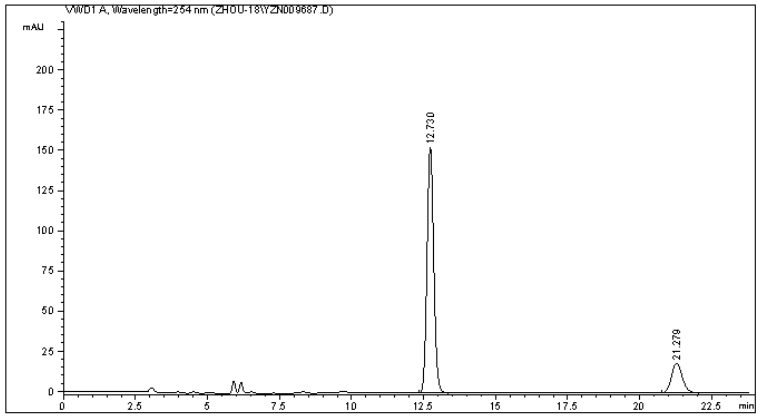

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.756 BB    0.2556  444.30624   26.86493  50.4494
   2  21.571 BB    0.4147  436.39029   16.27583  49.5506

Totals :                   880.69653   43.14075

==========================================================================
                        *** End of Report ***
```



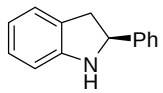
(±)-**3o**

Data File C:\CHEM32\1\DATA\ZHOU-18\YZN009687.D
Sample Name: CY-28-33

```
==========================================================================
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/2/2018 4:57:44 PM
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed    : 8/2/2018 4:40:44 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC11.M
Last changed    : 8/2/2018 7:33:26 PM
                  (modified after loading)
Sample Info     : OD-H, Hexane/i-PrOH = 90/10, 01.0 mL/min, 30 oC, 254 nm
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.730 BB    0.2518 2481.02319  153.02696  83.7969
   2  21.279 BB    0.4059  479.73358   18.41287  16.2031

Totals :                  2960.75677  171.43983

==========================================================================
                        *** End of Report ***
```



(-)-**3o**