Supporting Information

Efficient Synthesis of Chiral Vicinal Diamines with Four Contiguous Stereocenters *via* Sequential Dynamic Kinetic Resolution of 2,3-Diamino-1,4-diones

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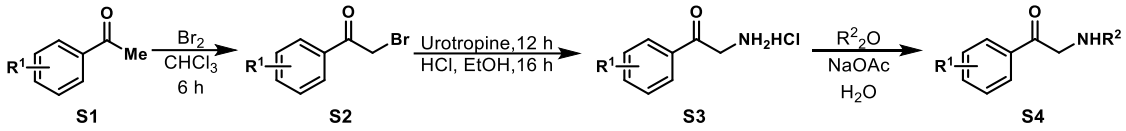
1. General Information

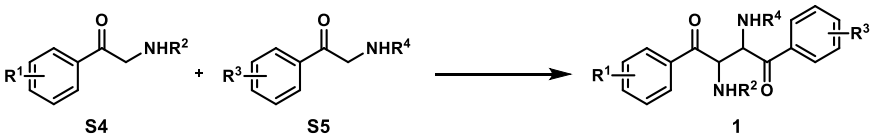
Unless otherwise mentioned, all experiments were carried out under an atmosphere of argon in a glovebox or using standard Schlenk techniques. Solvents were dried with standard procedures and degassed with N_2. Hydrogen gas (99.999%) was purchased from ZhongYiXing Co., Ltd. Anhydrous MeOH, EtOH, *i*-PrOH, DCM, 1,4-dioxane and *n*-hexane were purchased from J&K. $[Ir(COD)Cl]_2$ (Cat No.1091992, Leyan, Shanghai, China) were purchased from Leyan, Anhydrous THF, Et_2O were distilled from sodium benzophenoneketyl. Melting point (m.p.) was determined by RY-1 Melting Point Apparatus. ^1H and ^{13}C NMR spectra were recorded on Bruker AVANCE III 400 MHz (^1H: 400 MHz, ^{13}C: 101 MHz). Chemical shifts (δ) for ^1H and ^{13}C NMR spectra were given in ppm and were referenced to residual solvent or TMS peaks. HRMS were recorded on APEXII and ZAB-HS spectrometer. HPLC analyses were performed using an Agilent 1260 Series instrument. Column Chromatography was performed with silica gel Merck 60 (300 - 400 mesh).

2. Experimental Procedures for the Synthesis of Substrates

General procedure for the synthesis of N-acyl-2-aminoacetophenones[1]



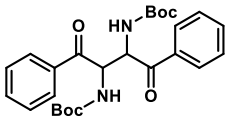
A solution of bromine (3.3 mmol) in chloroform (3 mL) was added dropwise to ketone **S1** (3 mmol) in chloroform (30 mL) at 0 ℃. The reaction mixture was stirred for an additional 6 h at room temperature. After the reaction completed, the reaction mixture was concentrated and purified by silica gel column chromatography (PE / EA = 50 : 1) to provide α-bromoketone **S2**. To a solution of α-bromoketone **S2** (3.0 mmol) in $CHCl_3$ was added urotropine (434 mg, 3.1 mmol, 1.03 equiv) all at once and the resulting mixture was stirred at room temperature overnight. After the reaction was complete, the mixture was filtered to give the precipitated quaternary ammonium salt and washed with small portions of ethanol. To the precipitate was added ethanol (10 mL) followed by concentrated hydrochloric acid (37%, 3.0 mL). The formed suspension was stirred at room temperature for 16 h and then filtered to remove a white precipitate. The filtrate was concentrated under reduced pressure to give a residue which was triturated with acetone followed by collection of the resulting solid by filtration to afford the desired primary amine as its HCl salt (**S3**). The HCl salt **S3** was suspended in anhydride $R^2{}_2O$ (5 mL) and anhydrous NaOAc (492 mg, 6.0 mmol, 2.0 equiv) was gradually added. After 30 min, water was added and the mixture was stirred vigorously for 1 h. The resulting solution was diluted with sat. aq $NaHCO_3$ and extracted with EtOAc (2×10 mL). The combined organic layers were washed with sat. aq $NaHCO_3$, sat. aq NaCl and dried over Na_2SO_4. Filtration and removal of the solvent under reduced pressure produced the crude product that could be purified if necessary by silica gel chromatography or recrystallization.



Add *t*-BuOK ((1 mmol, 1 eq.) to the DMSO solution of **S4** (1 mmol, 1 eq.) and **S5** (1 mmol, 1 eq.) within 30 minutes, then the reaction mixture was stirred for 2 h at room temperature under open air. After completion of the reaction (monitored by TLC), the mixture was diluted with water (10 mL) and extracted with DCM (3×10 mL). The combined organic extracts were washed with brine (2×7 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. Purification on silica gel (DCM: MeOH = 100 : 1 - 20 : 1) afforded the desired compound **1** as a white solid.

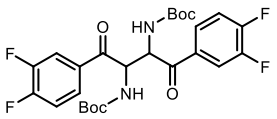
Characterization data of substrates

1a, 1b, 1c, 1f, 1i, 1k, 1m, 1n, 1u, 1v have been reported in previous articles.[1]



di-*tert*-butyl (1,4-dioxo-1,4-diphenylbutane-2,3-diyl)dicarbamate (1a)

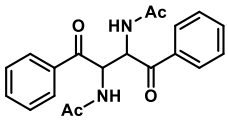
White solid, 304 mg, 65% yield. m.p. 148 - 150 ℃; as a mixture of diastereomers (*dl:meso* = 97:3). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.7 Hz, 4H), 7.66 – 7.58 (m, 2H), 7.53 – 7.46 (m, 4H), 5.66 (d, *J* = 8.5 Hz, 2H), 5.48 (d, *J* = 8.7 Hz, 2H), 1.41 (s, 18H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.5, 155.1, 135.0, 133.7, 128.8, 128.7, 80.3, 56.0, 28.2.



di-*tert*-butyl(1,4-bis(3,4-difluorophenyl)-1,4-dioxobutane-2,3-diyl)dicarbamate(1b)

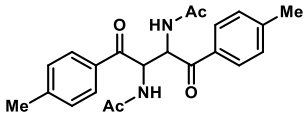
White solid, 324 mg 60% yield. m.p. 155 - 160 ℃; as a mixture of diastereomers (*dl:meso* > 20:1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.89 – 7.76 (m, 4H), 7.32 – 7.25 (m, 2H), 5.67 – 5.47 (m, 4H), 1.41 (s, 18H). **^{13}C NMR** (101

MHz, Chloroform-*d*) δ 194.3, 155.4, 154.1 (dd, *J* = 258.6, 13.1 Hz), 150.5 (dd, *J* = 251.4, 13.1 Hz), 131.8, 125.9 (d, *J* = 8.0 Hz), 118.3 (d, *J* = 18.2 Hz), 117.7 (d, *J* = 17.9 Hz), 80.8, 55.6, 28.2.



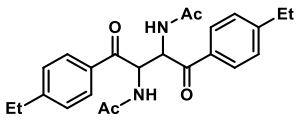
***N,N'*-(1,4-dioxo-1,4-diphenylbutane-2,3-diyl)diacetamide (1c)**

White solid, 246 mg, 70% yield. m.p. 214 - 216 ℃; as a mixture of diastereomers (*dl:meso* = 6:1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.52 (dd, *J* = 6.9, 2.8 Hz, 2H), 7.98 – 7.89 (m, 4H), 7.72 – 7.63 (m, 2H), 7.60 – 7.51 (m, 4H), 5.88 (dd, *J* = 7.0, 2.7 Hz, 2H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.8, 169.7, 135.1, 134.3, 129.4, 128.7, 53.1, 22.8.



***N,N'*-(1,4-dioxo-1,4-di-p-tolylbutane-2,3-diyl)diacetamide (1d)**

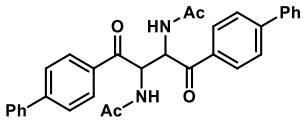
White solid, 247 mg, 65% yield. m.p. 198 - 203 ℃; as a mixture of diastereomers (*dl:meso* = 6:1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 7.9 Hz, 4H), 7.29 (d, *J* = 8.0 Hz, 4H), 6.55 (d, *J* = 7.9 Hz, 2H), 5.99 – 5.90 (m, 2H), 2.43 (s, 6H), 1.98 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.3, 169.7, 144.8, 132.6, 129.9, 128.8, 53.1, 22.8, 21.7. HRMS (ESI): calcd. for $[C_{22}H_{24}N_2NaO_4, M+Na]^+$: 403.1628, found: 403.1636.



***N,N'*-(1,4-bis(4-ethylphenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1e)**

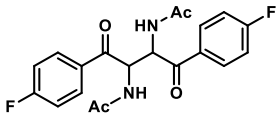
White solid, 245 mg, 60% yield. m.p. 200 - 205 ℃; as a mixture of diastereomers (*dl:meso* = 4:1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.55 – 8.44 (m, 2H), 7.91 – 7.84 (m, 4H), 7.44 – 7.36 (m, 4H), 5.91 – 5.83 (m, 2H), 2.71 – 2.65 (m, 4H), 1.80 (s, 6H), 1.24 – 1.19 (m, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.3, 169.6,

150.8, 132.8, 128.9, 128.8, 53.0, 40.0, 28.7, 22.8, 15.6. HRMS (ESI): calcd. for $[C_{24}H_{28}N_2NaO_4, M+Na]^+$: 431.1941, found: 431.1947.



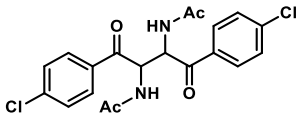
***N,N'*-(1,4-di([1,1'-biphenyl]-4-yl)-1,4-dioxobutane-2,3-diyl)diacetamide (1f)**

White solid, 302 mg, 60% yield. m.p. 261 - 262 ℃; as a mixture of diastereomers (*dl:meso* = 1.6:1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.56 (d, *J* = 9.7 Hz, 2H), 8.07 – 7.99 (m, 4H), 7.90 – 7.83 (m, 4H), 7.80 – 7.73 (m, 4H), 7.55 – 7.41 (m, 8H), 5.93 (dd, *J* = 7.1, 2.5 Hz, 2H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.3, 169.8, 145.6, 139.3, 133.9, 129.6, 129.4, 129.0, 127.5, 127.3, 53.2, 22.8.



***N,N'*-(1,4-bis(4-fluorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1g)**

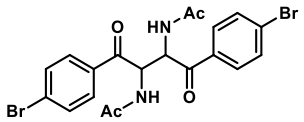
White solid, 217 mg, 56% yield. m.p. 208 - 215 ℃; as a mixture of diastereomers (*dl : meso*= 10 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.55 (dd, *J* = 6.9, 2.8 Hz, 2H), 8.05 – 7.97 (m, 4H), 7.43 – 7.36 (m, 4H), 5.82 (dd, *J* = 6.9, 2.7 Hz, 2H), 1.81 (s, 6H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 195.4, 169.8, 165.8 (d, *J* = 252.7 Hz), 131.8 (d, *J* = 2.6 Hz), 131.7 (d, *J* = 9.6 Hz), 116.5 (d, *J* = 22.1 Hz), 53.1, 22.8. HRMS (ESI): calcd. for $[C_{20}H_{18}F_2N_2NaO_4, M+Na]^+$: 411.1127, found: 411.1126.



***N,N'*-(1,4-bis(4-fluorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1h)**

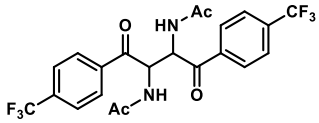
White solid, 253 mg, 60% yield. m.p. 246 - 250 ℃; as a mixture of diastereomers (*dl : meso*= 3 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.62 – 8.50 (m, 2H), 7.93 – 7.81 (m, 4H), 7.79 – 7.71 (m, 2H), 7.64 – 7.54 (m, 2H), 5.78 – 5.68 (m, 2H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 195.7, 169.9, 136.9, 134.3, 131.5, 128.3, 53.3, 22.7. HRMS (ESI): calcd. for $[C_{20}H_{18}Cl_2N_2NaO_4, M+Na]^+$: 443.0536,

found: 443.0534.



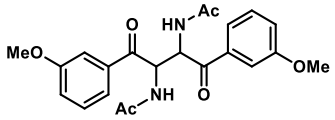
***N,N'*-(1,4-bis(4-bromophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1i)**

White solid, 322 mg, 60% yield. m.p. 262 - 263 ℃; as a mixture of diastereomers (*dl : meso* = 2 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.53 (dd, J = 14.5, 6.7 Hz, 2H), 7.90 – 7.73 (m, 8H), 5.77 (dd, J = 7.0, 2.5 Hz, 2H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.0, 169.9, 134.1, 132.5, 130.6, 128.5, 53.2, 22.7.



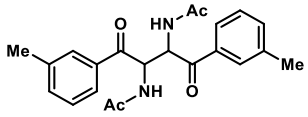
***N,N'*-(1,4-dioxo-1,4-bis(4-(trifluoromethyl)phenyl)butane-2,3-diyl)diacetamide (1j)**

White solid, 317 mg, 65% yield. m.p. 223 - 225 ℃; as a mixture of diastereomers (*dl : meso*= 5 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.61 (dd, J = 6.6, 2.6 Hz, 2H), 8.14 – 8.06 (m, 4H), 7.95 (d, J = 8.1 Hz, 4H), 5.81 (dd, J = 6.6, 2.6 Hz, 2H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 196.2, 170.0, 138.4, 133.4 (q, J = 31.9 Hz), 129.4, 126.4 (q, J = 4.0 Hz), 124.2 (q, J = 272.8 Hz), 53.6, 22.7. HRMS (ESI): calcd. for $[C_{22}H_{18}F_6N_2NaO_4, M+Na]^+$: 511.1063, found: 511.1067.



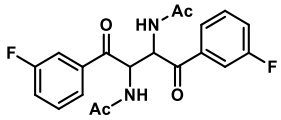
***N,N'*-(1,4-bis(3-methoxyphenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1k)**

White solid, 268 mg, 65% yield. m.p. 171 - 172 ℃; as a mixture of diastereomers (*dl : meso*= 2 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.57 – 7.12 (m, 8H), 6.75 (d, J = 7.9 Hz, 2H), 6.09 (d, J = 7.8 Hz, 2H), 3.77 (s, 6H), 2.09 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 197.0, 169.3, 159.7, 137.5, 130.3, 121.2, 119.9, 113.4, 55.8, 53.2, 22.6.



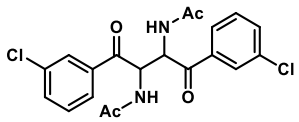
***N,N'*-(1,4-dioxo-1,4-di-m-tolylbutane-2,3-diyl)diacetamide (1l)**

White solid, 243 mg, 64% yield. m.p. 220 - 222 ℃; as a mixture of diastereomers (*dl : meso*= 5 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.52 – 8.39 (m, 2H), 7.80 – 7.67 (m, 4H), 7.53 – 7.37 (m, 4H), 5.89 – 5.77 (m, 2H), 2.37 (s, 6H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.8, 169.6, 138.7, 135.2, 134.8, 129.2, 129.1, 125.9, 53.3, 22.8, 21.4. HRMS (ESI): calcd. for $[C_{22}H_{24}N_2NaO_4, M+Na]^+$: 403.1634, found: 403.1628.



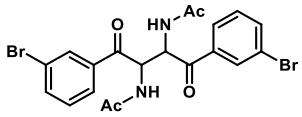
***N,N'*-(1,4-bis(3-fluorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1m)**

White solid, 237 mg, 61% yield. m.p. 219 - 221℃; as a mixture of diastereomers (*dl : meso*= 6 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.59 (dd, J = 6.8, 2.7 Hz, 2H), 7.78 (d, J = 7.7 Hz, 2H), 7.69 – 7.58 (m, 4H), 7.58 – 7.51 (m, 2H), 5.77 (dd, J = 6.8, 2.6 Hz, 2H), 1.80 (s, 6H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 195.7 (d, J = 2.1 Hz), 170.0, 162.7 (d, J = 245.5 Hz), 137.2 (d, J = 6.4 Hz), 131.7 (d, J = 7.9 Hz), 124.8, 121.3 (d, J = 21.4 Hz), 115.0 (d, J = 22.6 Hz), 53.3, 22.7.



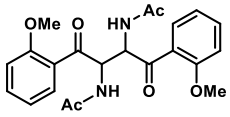
***N,N'*-(1,4-bis(3-chlorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1n)**

White solid, 240 mg, 57% yield. m.p. 198 - 199℃; as a mixture of diastereomers (*dl : meso*= 6 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.68 – 8.54 (m, 2H), 7.94 – 7.84 (m, 4H), 7.80 – 7.73 (m, 2H), 7.64 – 7.58 (m, 2H), 5.78 – 5.73 (m, 2H), 1.82 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 195.7, 169.9, 136.9, 134.3, 134.0, 131.4, 128.3, 127.2, 53.3, 22.7.



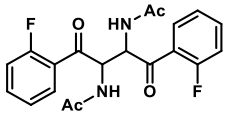
***N,N'*-(1,4-bis(3-bromophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1o)**

White solid, 316 mg, 62% yield. m.p. 190 - 195℃; as a mixture of diastereomers (*dl : meso*= 5 : 2). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.64 – 8.52 (m, 2H), 8.09 – 8.01 (m, 2H), 7.94 – 7.84 (m, 4H), 7.56 – 7.50 (m, 2H), 5.81 – 5.66 (m, 2H), 1.81 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 195.7, 169.9, 137.1, 136.9, 131.7, 131.2, 127.6, 122.7, 53.3, 22.7. HRMS (ESI): calcd. for $[C_{20}H_{18}Br_2N_2NaO_4, M+Na]^+$: 530.9525, found: 530.9525.



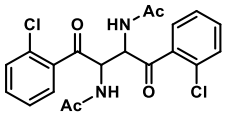
***N,N'*-(1,4-bis(2-methoxyphenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1p)**

White solid, 210 mg, 51% yield. m.p. 166 - 170℃; as a mixture of diastereomers (*dl : meso*= 3 : 2). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.83 (d, J = 30.7, 8.1 Hz, 2H), 7.57 – 7.44 (m, 2H), 7.44 – 7.29 (m, 2H), 7.16 – 6.92 (m, 4H), 5.80 (d, 2H), 3.61 (s, 6H), 1.81 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 198.5, 169.3, 157.9, 134.0, 130.5, 126.5, 121.0, 112.5, 58.2, 56.1, 23.0. HRMS (ESI): calcd. for $[C_{22}H_{24}N_2NaO_6, M+Na]^+$: 435.1526, found: 435.1524.



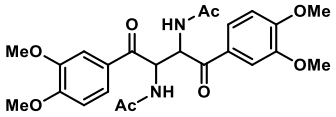
***N,N'*-(1,4-bis(2-fluorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1q)**

White solid, 252 mg, 65% yield. m.p. 160 - 165℃; as a mixture of diastereomers (*dl : meso*= 3 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.36 (d, J = 7.2 Hz, 2H), 7.84 – 7.54 (m, 4H), 7.47 – 7.16 (m, 4H), 5.54 (d, J = 8.5 Hz, 2H), 1.76 (s, 6H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 195.4, 170.0, 160.7 (d, J = 253.8 Hz), 135.3 (d, J = 21.6 Hz), 131.0, 125.2, 124.7 (d, J = 12.1 Hz), 117.1 (d, J = 23.3 Hz), 57.2, 22.6. HRMS (ESI): calcd. for $[C_{20}H_{18}F_2N_2NaO_4, M+Na]^+$: 411.1127, found: 411.1126.



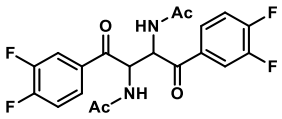
***N,N'*-(1,4-bis(2-chlorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide(1r)**

White solid, 265 mg, 63% yield. m.p. 135 - 140℃; as a mixture of diastereomers (*dl : meso* = 2 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.35 (dd, J = 7.0, 1.9 Hz, 2H), 7.86 – 7.29 (m, 8H), 5.62 (dd, J = 6.9, 1.9 Hz, 2H), 1.79 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 198.1, 170.0, 136.3, 133.2, 131.1, 130.9, 130.0, 127.7, 56.9, 40.0, 22.7. HRMS (ESI): calcd. for $[C_{20}H_{18}Cl_2N_2NaO_4, M+Na]^+$: 443.0536, found: 443.0542.



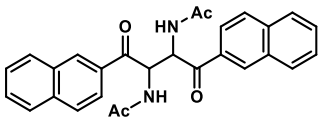
***N,N'*-(1,4-bis(3,4-dimethoxyphenyl)-1,4-dioxobutane-2,3-diyl)diacetamide(1s)**

White solid, 288 mg, 61% yield. m.p. 210 - 215℃; as a mixture of diastereomers (*dl : meso* = 10 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.56 (dd, J = 7.2, 2.8 Hz, 2H), 7.72 (dd, J = 8.5, 2.0 Hz, 2H), 7.56 (s, 2H), 7.16 (d, J = 8.6 Hz, 2H), 5.94 (dd, J = 7.2, 2.8 Hz, 2H), 3.90 (s, 6H), 3.83 (s, 6H), 1.87 (s, 6H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 195.2, 169.7, 154.0, 149.1, 127.8, 123.4, 111.6, 111.2, 56.3, 55.9, 52.8, 22.8. HRMS (ESI): calcd. for $[C_{24}H_{28}N_2NaO_8, M+Na]^+$: 495.1738, found: 495.1746.



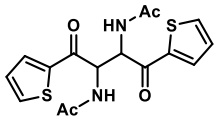
***N,N'*-(1,4-bis(3,4-difluorophenyl)-1,4-dioxobutane-2,3-diyl)diacetamide (1t)**

White solid, 254 mg, 60% yield. m.p. 216 - 220℃; as a mixture of diastereomers (*dl : meso* = 3 : 2). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.63 (dd, J = 6.8, 2.7 Hz, 2H), 7.98 – 7.88 (m, 2H), 7.87 – 7.80 (m, 2H), 7.72 – 7.61 (m, 2H), 5.80 – 5.68 (m, 2H), 1.82 (s, 6H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 194.5, 170.0, 153.4 (d, J = 268.0 Hz), 149.9 (d, J = 248.1 Hz), 132.4 (t, J = 16.0 Hz), 126.5 (q, J = 3.0 Hz), 118.8 (d, J = 17.8 Hz), 117.8 (d, J = 18.4 Hz), 53.1, 22.7. HRMS (ESI): calcd. for $[C_{20}H_{16}F_4N_2NaO_4, M+Na]^+$: 447.0938, found: 447.0946.



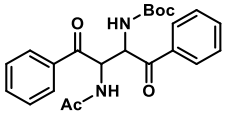
***N,N'*-(1,4-di(naphthalen-2-yl)-1,4-dioxobutane-2,3-diyl)diacetamide (1u)**

White solid, 300 mg, 66% yield. m.p. 222 - 224℃; as a mixture of diastereomers (*dl : meso* = 5 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.68 – 8.55 (m, 4H), 8.10 – 7.98 (m, 6H), 7.94 (dd, *J* = 8.6, 1.7 Hz, 2H), 7.75 – 7.61 (m, 4H), 6.06 (dd, *J* = 6.9, 2.5 Hz, 2H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 196.8, 169.8, 135.8, 132.5, 132.5, 130.5, 130.1, 129.5, 129.0, 128.2, 127.7, 124.3, 53.6, 22.8.



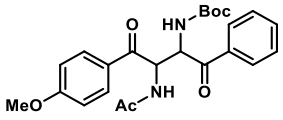
***N,N'*-(1,4-dioxo-1,4-di(thiophen-2-yl)butane-2,3-diyl)diacetamide (1v)**

White solid, 291 mg, 80% yield. m.p. 237 - 239 ℃; as a mixture of diastereomers (*dl : meso* = 5 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.62 (dd, *J* = 6.9, 2.7 Hz, 2H), 8.07 (dd, *J* = 4.9, 1.1 Hz, 2H), 7.96 (dd, *J* = 3.8, 1.2 Hz, 2H), 7.28 (dd, *J* = 4.9, 3.8 Hz, 2H), 5.72 (dd, *J* = 6.9, 2.7 Hz, 2H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 189.6, 169.8, 142.0, 136.4, 134.0, 129.4, 53.8, 22.8.



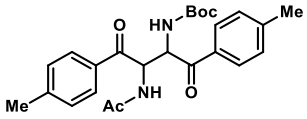
***tert*-butyl (3-acetamido-1,4-dioxo-1,4-diphenylbutan-2-yl)carbamate (1w)**

White solid, 143 mg, 35% yield. m.p. 205 - 210 ℃; as a mixture of diastereomers (dr = 7 : 3), major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.01 – 7.31 (m, 10H), 6.56 (m, 1H), 6.03 (m, 1H), 5.87 – 5.60 (m, 1H), 5.43 (m, 1H), 2.00 (s, 3H), 1.45 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.4, 196.3, 169.8, 155.2, 134.9, 134.1, 134.0, 133.9, 128.9, 128.9, 128.8, 128.7, 80.5, 55.9, 54.7, 28.3, 23.1. dr = 2 : 1. HRMS (ESI): calcd. for $[C_{23}H_{26}N_2NaO_5, M+Na]^+$: 433.1734, found: 433.1736.



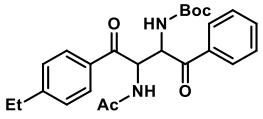
***tert*-butyl(3-acetamido-4-(4-methoxyphenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1x)**

White solid, 132 mg, 30% yield. m.p.175 - 180℃; as a mixture of diastereomers (dr = 10 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 – 7.91 (m, 4H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.44 (d, *J* = 9.1 Hz, 1H), 5.93 (q, *J* = 9.1, 5.6 Hz, 1H), 5.69 (q, *J* = 9.5, 5.6 Hz, 1H), 5.41 (d, *J* = 9.5 Hz, 1H), 3.89 (s, 3H), 1.99 (s, 3H), 1.42 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.5, 194.7, 169.8, 164.3, 155.3, 135.0, 133.8, 131.3, 128.9, 128.8, 127.6, 114.1, 80.4, 56.0, 55.6, 54.3, 28.2, 23.1. HRMS (ESI): calcd. for $[C_{24}H_{28}N_2NaO_6, M+Na]^+$: 463.1839, found: 463.1845.



***tert*-butyl (3-acetamido-1,4-dioxo-1,4-di-p-tolylbutan-2-yl)carbamate (1y)**

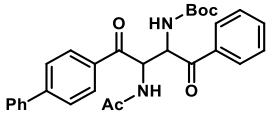
White solid, 132 mg, 30% yield. m.p.160 - 170 ℃; as a mixture of diastereomers (dr = 3 : 2). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.92 – 7.77 (m, 3H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.35 – 7.28 (m, 2H), 7.24 – 7.13 (m, 2H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.09 – 5.89 (m, 1H), 5.81 – 5.39 (m, 2H), 2.49 – 2.37 (m, 6H), 2.02 (s, 3H), 1.43 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 195.7, 170.3, 155.7, 145.1, 144.8, 132.4, 132.3, 129.5, 129.4, 129.0, 128.8, 80.3, 56.7, 55.5, 28.2, 23.2, 21.8. HRMS (ESI): calcd. for $[C_{25}H_{30}N_2NaO_5, M+Na]^+$: 461.2047, found:. Exact Mass: 461.2050



***tert*-butyl(3-acetamido-4-(4-ethylphenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1z)**

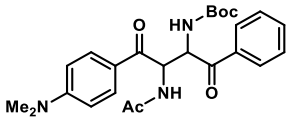
White solid, 140 mg, 32% yield. m.p. 180 - 185℃; as a mixture of diastereomers (dr = 4 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.96 – 7.91 (m, 3H),

7.71 – 7.56 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.40 (d, *J* = 9.0 Hz, 1H), 5.95 (dd, *J* = 9.1, 5.1 Hz, 1H), 5.67 (dd, *J* = 9.3, 5.0 Hz, 1H), 5.41 (d, *J* = 9.3 Hz, 1H), 2.74 (q, *J* = 7.6 Hz, 2H), 1.99 (s, 3H), 1.42 (s, 9H), 1.28 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.4, 169.8, 155.2, 151.1, 133.8, 129.1, 129.0, 128.9, 128.8, 128.7, 128.4, 80.4, 56.0, 54.6, 29.1, 28.2, 23.1, 15.0. HRMS(ESI): calcd. for $[C_{25}H_{30}N_2NaO_5, M+Na]^+$: 461.2047, found: 461.2051.



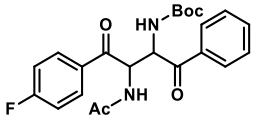
***tert*-butyl(4-([1,1'-biphenyl]-4-yl)-3-acetamido-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1aa)**

White solid, 160 mg, 33% yield. m.p. 157 - 160℃; as a mixture of diastereomers (dr = 2 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.76 – 8.38 (m, 1H), 8.15 – 7.27 (m, 15H), 6.14 – 5.74 (m, 1H), 5.58 (m, 2.1 Hz, 1H), 1.83 (s, 3H), 1.37 (s, 9H). **^{13}C NMR** (101 MHz, DMSO) δ 196.8, 196.3, 169.6, 155.8, 145.5, 139.3, 135.2, 134.1, 134.0, 129.6, 129.4, 129.3, 129.0, 128.6, 127.5, 79.2, 55.2, 52.9, 28.5, 22.8. HRMS(ESI): calcd. for $[C_{29}H_{30}N_2NaO_5, M+Na]^+$: 509.2047, found: 509.2052.



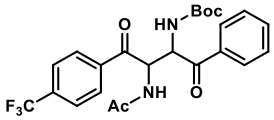
***tert*-butyl(3-acetamido-4-(4-(dimethylamino)phenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1ab)**

White solid, 136 mg, 30% yield. m.p. 210 - 215℃; as a mixture of diastereomers (dr = 9 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.19 – 8.08 (m, 1H), 8.04 – 7.87 (m, 3H), 7.73 – 7.41 (m, 4H), 7.05 (dd, *J* = 8.4, 2.5 Hz, 1H), 6.49 (d, *J* = 8.5 Hz, 1H), 5.91 – 5.81 (m, 1H), 5.74 – 5.63 (m, 1H), 5.47 (d, *J* = 9.4 Hz, 1H), 2.96 (s, 6H), 2.00 (s, 3H), 1.42 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.3, 193.5, 169.8, 156.5, 155.3, 135.2, 133.8, 129.3, 128.9, 128.8, 119.4, 116.2, 80.5, 56.1, 54.3, 43.4, 28.2, 23.0. HRMS (ESI): calcd. for $[C_{25}H_{31}N_3NaO_5, M+Na]^+$: 476.2156, found: 476.2162.



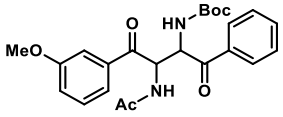
***tert*-butyl(3-acetamido-4-(4-fluorophenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1ac)**

White solid, 135 mg, 32% yield. m.p. 215 - 220℃; as a mixture of diastereomers (dr = 9 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.07 – 7.89 (m, 4H), 7.56 – 7.44 (m, 3H), 7.18 (t, *J* = 8.6 Hz, 2H), 6.45 (d, *J* = 9.2 Hz, 1H), 5.94 (dd, *J* = 9.4, 5.8 Hz, 1H), 5.69 (dd, *J* = 9.5, 5.9 Hz, 1H), 5.42 (d, *J* = 9.5 Hz, 1H), 2.00 (s, 3H), 1.42 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 196.3, 194.5, 169.9, 166.3 (d, *J* = 256.4 Hz), 155.3, 134.7, 134.0, 131.6 (d, *J* = 9.5 Hz), 128.8 (d, *J* = 4.0 Hz), 116.0 (d, *J* = 22.0 Hz), 80.6, 55.7, 54.5, 28.2, 23.1. HRMS (ESI): calcd. for $[C_{23}H_{25}FN_2NaO_5, M+Na]^+$: 451.1641, found: 451.1648.



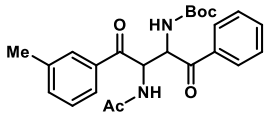
***tert*-butyl (3-acetamido-1,4-dioxo-1-phenyl-4-(4-(trifluoromethyl)phenyl)butan-2-yl)carbamate(1ad)**

White solid, 180 mg, 38% yield. m.p. 208 - 212℃; as a mixture of diastereomers (dr = 7 : 2). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.58 (d, *J* = 9.6 Hz, 1H), 8.12 (dd, *J* = 17.2, 8.1 Hz, 2H), 8.01 – 7.86 (m, 4H), 7.71 – 7.51 (m, 4H), 5.83 – 5.74 (m, 1H), 5.60 – 5.49 (m, 1H), 1.80 (s, 3H), 1.35 (s, 9H). **^{13}C NMR (**101 MHz, DMSO-d_6) δ 196.9, 196.2, 169.8, 155.8, 138.6, 135.1, 134.2, 133.7 (q, *J* = 23.1 Hz), 129.4, 129.3, 128.7, 126.5(q, *J* = 3.1 Hz), 124.2 (q, *J* = 272.8 Hz), 79.3, 55.2, 53.3, 28.5, 22.8. HRMS (ESI): calcd. for $[C_{23}H_{25}F_3N_2NaO_5, M+Na]^+$: 501.1608, found: 501.1616.



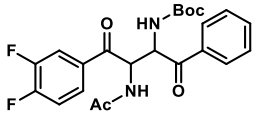
***tert*-butyl(3-acetamido-4-(3-methoxyphenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate(1ae)**

White solid, 145 mg, 33% yield. m.p. 186 - 190℃; as a mixture of diastereomers (dr = 5 : 1). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.50 (d, J = 9.8 Hz, 1H), 7.95 – 7.88 (m, 2H), 7.70 – 7.63 (m, 1H), 7.60 – 7.44 (m, 6H), 7.28 – 7.15 (m, 1H), 5.79 (t, J = 9.5 Hz, 1H), 5.55 (t, J = 9.5 Hz, 1H), 3.80 (d, J = 5.4 Hz, 3H), 1.81 (s, 3H), 1.36 (s, 9H). **^{13}C NMR** (101 MHz, DMSO) δ 196.8, 196.4, 169.6, 159.9, 155.8, 136.5, 135.2, 134.1, 130.5, 129.3, 128.6, 121.0, 120.2, 113.3, 79.2, 55.8, 55.1, 53.0, 28.5, 22.8. HRMS (ESI): calcd. for [$C_{24}H_{28}N_2NaO_6$, M+Na]$^+$: 463.1840, found: 463.1847.



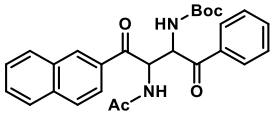
***tert*-butyl (3-acetamido-1,4-dioxo-1-phenyl-4-(m-tolyl)butan-2-yl)carbamate (1af)**

White solid, 130 mg, 31% yield. m.p. 188 - 192℃; as a mixture of diastereomers (dr = 3 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.99 – 7.88 (m, 2H), 7.82 – 7.33 (m, 7H), 6.43 (d, J = 8.6 Hz, 1H), 5.93 (dd, J = 8.9, 4.6 Hz, 1H), 5.66 (dd, J = 9.3, 4.9 Hz, 1H), 5.45 (d, J = 9.2 Hz, 1H), 2.44 (s, 3H), 2.00 (s, 3H), 1.42 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.4, 196.0, 169.8, 155.2, 138.6, 134.9, 133.9, 129.3, 128.9, 128.8, 128.7, 126.2, 125.9, 80.5, 56.1, 54.8, 28.3, 23.1, 21.4. HRMS (ESI): calcd. for [$C_{24}H_{28}N_2NaO_5$, M+Na]$^+$: 447.1891, found: 447.1895.



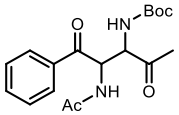
***tert*-butyl(3-acetamido-4-(3,4-difluorophenyl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate (1ag)**

White solid, 140 mg, 31% yield. m.p. 215 - 220℃; as a mixture of diastereomers (dr = 5 : 2). major diastereomer : **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.59 (d, J = 9.6 Hz, 1H), 8.00 – 7.82 (m, 4H), 7.71 – 7.49 (m, 5H), 5.79 – 5.64 (m, 1H), 5.59 – 5.38 (m, 1H), 1.81 (s, 3H), 1.35 (s, 9H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 196.8, 194.6, 169.8, 155.8, 135.1, 134.2, 132.8, 129.3, 128.8, 128.7, 126.5, 118.8 (d, J = 17.9 Hz), 117.8 (d, J = 18.3 Hz), 79.3, 55.1, 52.9, 28.5, 22.8. HRMS (ESI): calcd. for [$C_{23}H_{24}F_2N_2NaO_5$, M+Na]$^+$: 469.1545, found: 469.1553.



***tert*-butyl(3-acetamido-4-(naphthalen-2-yl)-1,4-dioxo-1-phenylbutan-2-yl)carbamate(1ah)**

White solid, 150 mg, 32% yield. m.p. 217 - 220℃; as a mixture of diastereomers (dr = 9 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.52 (s, 1H), 8.02 – 7.80 (m, 6H), 7.71 – 7.48 (m, 5H), 6.56 (d, *J* = 8.8 Hz, 1H), 6.18 – 6.05 (m, 1H), 5.79 – 5.68 (m, 1H), 5.54 (d, *J* = 9.0 Hz, 1H), 2.03 (s, 3H), 1.43 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.3, 196.1, 169.9, 155.2, 136.0, 134.9, 133.9, 132.4, 132.1, 130.6, 129.6, 128.9, 128.8, 127.9, 127.0, 124.5, 123.7, 80.5, 56.2, 54.8, 28.1, 23.1. HRMS (ESI): calcd. for $[C_{27}H_{28}N_2NaO_5, M+Na]^+$: 483.1891, found: 483.1898.

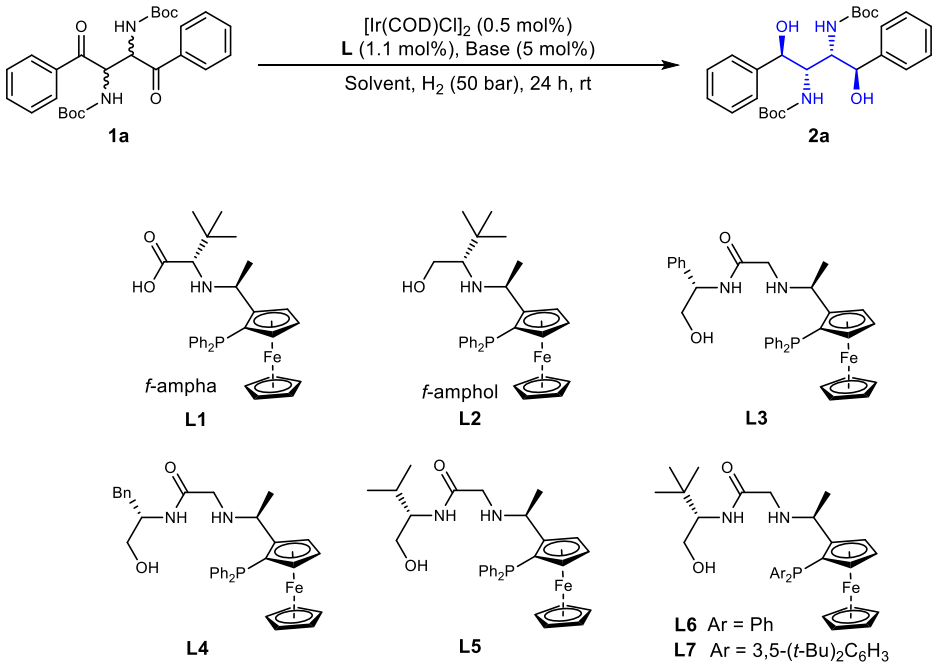


tert-butyl (2-acetamido-1,4-dioxo-1-phenylpentan-3-yl)carbamate(1ai)

White solid, 105 mg, 30% yield. m.p. 150 - 160℃; as a mixture of diastereomers (dr = 20 : 1). major diastereomer : **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 7.6 Hz, 2H), 7.72 – 7.60 (m, 1H), 7.57 – 7.48 (m, 2H), 6.74 (d, *J* = 8.5 Hz, 1H), 6.05 – 5.94 (m, 1H), 5.37 (d, *J* = 9.0 Hz, 1H), 4.80 – 4.69 (m, 1H), 2.45 (s, 3H), 2.01 (s, 3H), 1.40 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.8, 196.0, 170.0, 155.1, 134.3, 134.0, 128.9, 128.6, 80.4, 60.7, 54.9, 28.1, 27.5, 23.0. HRMS (ESI): calcd. for $[C_{18}H_{24}N_2NaO_5, M+Na]^+$: 371.1577, found: 371.1580.

3. Reaction Condition Optimization

Supplementary Table 1. Optimization of ligands[a]

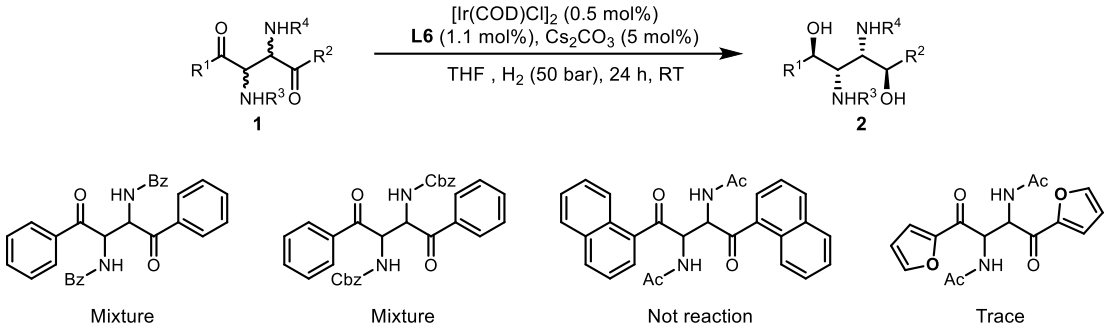


Entry	Ligand	Solvent	Base	Yield (%)[b]	Ee (%)[c]
1	**L1**	*i*-PrOH	Cs_2CO_3	-[d]	-
2	**L1**	*i*-PrOH	KO*t*-Bu	-[d]	-
3	**L1**	*i*-PrOH	K_2CO_3	-[d]	-
4	**L1**	THF	Cs_2CO_3	-[d]	-
5	**L2**	*i*-PrOH	Cs_2CO_3	-[d]	-
6	**L2**	*i*-PrOH	KO*t*-Bu	-[d]	-
7	**L3**	THF	Cs_2CO_3	92	95
8	**L4**	THF	Cs_2CO_3	90	93
9	**L5**	THF	Cs_2CO_3	97	99
10	**L6**	THF	Cs_2CO_3	99	>99
11	**L7**	THF	Cs_2CO_3	92	>99
12	**L6**	EtOAc	Cs_2CO_3	79	98
13	**L6**	*n*-hexane	Cs_2CO_3	79	97
14	**L6**	*i*-PrOH	Cs_2CO_3	35	98
15	**L6**	MeOH	Cs_2CO_3	trace	-
16	**L6**	1,4-dioxane	Cs_2CO_3	trace	-
17	**L6**	THF	LiO*t*-Bu	50	95
18	**L6**	THF	LiOH	58	94

19	**L6**	THF	NaOMe	96	98
20	**L6**	THF	Na_2CO_3	trace	-
21	**L6**	THF	K_2CO_3	trace	-
22	**L6**	THF	KOH	94	96

[a] Unless otherwise mentioned, all reactions were performed on a 0.05 mmol scale at room temperature in 1 mL THF with 0.5 mol% $[Ir(COD)Cl]_2$, 1.1 mol% ligand, 50 bar H_2, and a reaction time of 24 hours. [b] Determined by ^1H NMR spectroscopy. [c] Determined by HPLC analysis using a chiral stationary phase. [d] **1a** was partly consumed, but the mixture is too complicated to afford pure 2a.

Supplementary Table 2. Unsuitable substrate scope for the dual dynamic kinetic resolution

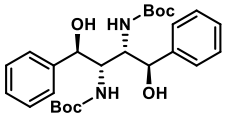


The above-mentioned substrate did not undergo smooth transformation, revealing that the amine protecting group, steric hindrance, and coordination ability of the substrate significantly influence this reaction.

4. Experimental Procedures for Asymmetric Hydrogenation

General procedure of asymmetric hydrogenation of 2,3-diamino-1,4-diones (S/C = 100): To a 4.0 mL vial was added the catalyst precursor $[Ir(COD)Cl]_2$ (3.3 mg, 5×10^{-3} mmol, 1.0 eq.), (S_C, S_C, R_{FC})-**L6** (6.3 mg, 1.1×10^{-2}mmol, 2.2 eq.) and anhydrous *i*-PrOH (1.0 mL) in the argon-filled glovebox. The mixture was stirred for 1.0 h at 25 ℃. The resulting orange solution (50 μL) was transferred by syringe into a vial (5.0 mL) charged with substrate (0.05 mmol), Cs_2CO_3 (0.8 mg, 0.0025 mmol) and anhydrous THF (1.0 mL). The vial was transferred to an autoclave, which was then charged with of H_2 (50 bar) and stirred at room temperature for 24 h. The hydrogen gas was released slowly in a well-ventilated hood and the solution was concentrated and purified by flash chromatography on silica gel (CH_2Cl_2/MeOH, 10:1) to afford the product.

The racemic samples for the standard of chiral HPLC spectra were prepared using the mixture of 0.25 mol% (R_C, R_C, S_{FC})-**L6** and 0.25 mol% (S_C, S_C, R_{FC})-**L6** as ligands. It should be noted that due to weighing errors, the ratio of (R_C, R_C, S_{FC})-**L6** to (S_C, S_C, R_{FC})-**L6** is not exactly 1:1. As a result, the peak area ratio of the enantiomers in the racemate's HPLC spectrum does not always precisely equal 50:50



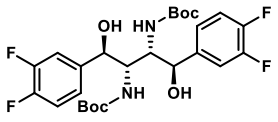
2a

***di-tert*-butyl((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-diphenylbutane-2,3-diyl)dicarbamate (2a)**

White solid, 23.1 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.24 – 7.05 (m, 10H), 5.20 – 5.09 (m, 2H), 4.85 – 4.75 (m, 2H), 4.55 – 4.45 (m, 2H), 4.09 – 4.00 (m, 2H), 1.43 (s, 18H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.4, 140.0, 128.6, 127.4, 126.2, 81.0, 75.5, 53.7, 28.4. HRMS (ESI): calcd. for $[C_{26}H_{36}N_2NaO_6, M+Na]^+$: 495.2465, found: 495.2468.

Optical Rotation: 2a $[\alpha]^{25}_D$ = -33.6 (c = 0.5, MeOH), 99.9% ee and *ent*-**2a** $[\alpha]^{25}_D$ = +33.6 (c = 0.5, MeOH), 99.9% ee, The absolute configuration of **2a** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 85:15,

flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 4.9 min, second peak: t_R = 6.6 min).

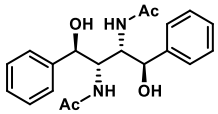


2b

di-*tert*-butyl ((*1R,2S,3S,4R*)-1,4-bis(3,4-difluorophenyl)-1,4-dihydroxybutane-2,3-diyl)dicarbamate (2b)

White solid, 26.4 mg, 97% yield. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.13 – 6.89 (m, 6H), 5.15 (d, *J* = 8.9 Hz, 2H), 4.94 (d, *J* = 9.2 Hz, 2H), 4.57 – 4.42 (m, 2H), 3.98 – 3.86 (m, 2H), 1.44 (s, 18H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.2, 150.2 (dd, *J* = 248.9, 13.0 Hz)., 149.7 (dd, *J* = 249.1, 13.0 Hz), 137.3, 122.2, 117.1 (d, *J* = 16.9 Hz), 115.4 (d, *J* = 17.4 Hz), 81.5, 74.1, 53.8, 28.2. HRMS (ESI): calcd. for [$C_{26}H_{33}F_4N_2O_6$, M+H]$^+$: 545.2269, found: 545.2270.

Optical Rotation: 2b $[\alpha]^{25}_D$ = -17.3 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2b** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 85:15, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 4.6 min, second peak: t_R = 5.8 min).



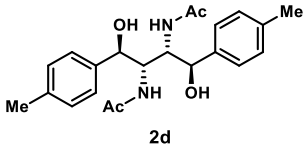
2c

***N,N*'-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-diphenylbutane-2,3-diyl)diacetamide (2c)**

White solid, 17.4 mg, 98% yield. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.74 (d, *J* = 9.1 Hz, 2H), 7.38 – 7.33 (m, 4H), 7.31 – 7.25 (m, 4H), 7.24 – 7.19 (m, 2H), 5.56 – 5.46 (m, 2H), 4.39 – 4.22 (m, 4H), 1.74 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 171.0, 143.1, 128.1, 127.5, 127.4, 71.9, 55.3, 22.9. HRMS (ESI): calcd. for [$C_{20}H_{25}N_2O_4$, M+H]$^+$: 357.1809, found: 357.1814.

Optical Rotation: 2c $[\alpha]^{25}_D$ = - 17.0 (c = 0.5, MeOH), 99.9% ee. The absolute config-

uration of **2c** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 27.1 min, second peak: t_R = 30.1 min).

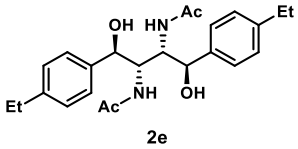


2d

***N,N'*-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-di-p-tolylbutane-2,3-diyl)diacetamide (2d)**

White solid, 18.8 mg, 98% yield. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.96 – 7.84 (m, 2H), 7.35 – 7.27 (m, 4H), 7.21 – 7.13 (m, 4H), 5.63 – 5.47 (m, 2H), 4.43 – 4.26 (m, 4H), 2.35 (s, 6H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 171.5, 140.1, 136.6, 128.9, 127.5, 71.8, 55.5, 23.0, 21.4. HRMS (ESI): calcd. for $[C_{22}H_{29}N_2O_4, M+H]^+$: 385.2121, found: 385.2126.

Optical Rotation: 2d $[\alpha]^{25}_D$ = -12.2 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2d** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 92:08, flow rate = 0.5 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 68.0 min, second peak: t_R = 74.0 min).



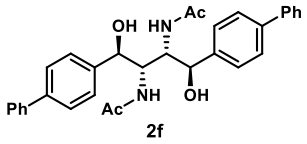
2e

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(4-ethylphenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2e)**

White solid, 19.7 mg, 96% yield and *ent*-**2e** White solid, 40.2 mg, 98% yield. m.p. 200-205 ℃. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.94 – 6.88 (m, 4H), 6.86 – 6.77 (m, 6H), 5.43 – 5.34 (m, 2H), 4.56 – 4.48 (m, 2H), 4.14 – 4.07 (m, 2H), 2.61 (q, *J* = 7.6 Hz, 4H), 2.10 (s, 6H), 1.24 (t, *J* = 7.6 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 142.8, 136.3, 127.9, 125.4, 75.3, 51.8, 28.5, 23.2, 15.5. HRMS (ESI): calcd. for $[C_{24}H_{33}N_2O_4, M+H]^+$: 413.2434, found: 413.2434.

Optical Rotation: 2e $[\alpha]^{25}_D$ = -16.5 (c = 0.5, MeOH), 97% ee and *ent*-**2e** $[\alpha]^{25}_D$ = +13.2

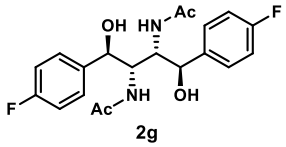
(c = 0.5, MeOH), 99.9% ee, The absolute configuration of **2e** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 15.7 min, second peak: t_R = 21.0 min).



2f

***N,N*'-((*1R,2S,3S,4R*)-1,4-di([1,1'-biphenyl]-4-yl)-1,4-dihydroxybutane-2,3-diyl)di-acetamide (2f)**

White solid, 24.8 mg, 98% yield. m.p. 210-215 ℃. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.38 – 7.32 (m, 10H), 7.26 – 7.18 (m, 4H), 7.01 – 6.92 (m, 4H), 6.82 – 6.72 (m, 2H), 5.64 – 5.41 (m, 2H), 4.68 – 4.57 (m, 2H), 4.23 – 4.13 (m, 2H), 2.17 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.3, 140.4, 139.7, 138.1, 128.8, 127.3, 126.9, 125.7, 75.5, 51.7, 23.3. HRMS (ESI): calcd. for $[C_{32}H_{33}N_2O_4, M+H]^+$: 509.2434, found: 509.2439. **Optical Rotation: 2f** $[\alpha]^{25}_D$ = -17.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2f** $[\alpha]^{25}_D$ = +17.2 (c = 0.5, MeOH), 99.9% ee, The absolute configuration of **2f** was assigned by analogy. (HPLC condition: Daicel Chiralcel IA-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 0.5 ml/min, T = 25 ℃, wavelength = 254 nm, first peak: t_R = 33.0 min, second peak: t_R = 39.0 min).



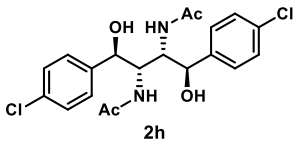
2g

***N,N*'-((*1R,2S,3S,4R*)-1,4-bis(4-fluorophenyl)-1,4-dihydroxybutane-2,3-diyl)diac-etamide (2g)**

White solid, 19.0 mg, 97% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.06 – 6.99 (m, 2H), 6.91 – 6.84 (m, 4H), 6.84 – 6.76 (m, 4H), 5.71 – 5.56 (m, 2H), 4.62 – 4.47 (m, 2H), 4.09 – 3.95 (m, 2H), 2.08 (s, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.6,

161.8 (d, J = 246.2 Hz), 135.0 (d, J = 3.1 Hz), 127.1 (d, J = 8.2 Hz), 115.3 (d, J = 21.5 Hz), 74.6, 51.7, 22.9. HRMS (ESI): calcd. for $[C_{20}H_{23}F_2N_2O_4, M+H]^+$: 393.1620, found: 393.1622.

Optical Rotation: 2g $[\alpha]^{25}_D$ = -17.2 (c = 0.5, MeOH), 99% ee. The absolute configuration of **2g** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 215 nm, first peak: t_R = 16.8 min, second peak: t_R = 20.6 min).

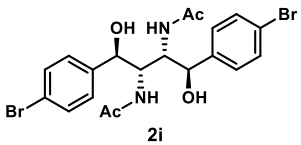


2h

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(4-chlorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2h)**

White solid, 20.4 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.10 (d, J = 8.4 Hz, 4H), 6.83 (d, J = 8.4 Hz, 4H), 6.66 (d, J = 8.3 Hz, 2H), 5.49 (d, J = 12.1 Hz, 2H), 4.57 – 4.50 (m, 2H), 3.99 (dd, J = 8.1, 3.2 Hz, 2H), 2.14 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 170.3, 141.9, 131.4, 128.8, 127.5, 71.0, 54.4, 39.5, 22.4. HRMS (ESI): calcd. for $[C_{20}H_{23}Cl_2N_2O_4, M+H]^+$: 425.1029, found: 425.1029.

Optical Rotation: 2h $[\alpha]^{25}_D$ = -24.0 (c = 0.5, MeOH). The absolute configuration of **2h** was assigned by X-ray. 99.9% ee (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 210 nm, first peak: t_R = 16.7 min, second peak: t_R = 19.0 min).



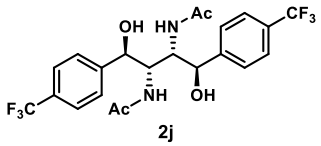
2i

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(4-bromophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2i)**

White solid, 25.2 mg, 98% yield and *ent*-**2i** White solid, 23.8 mg, 97% yield.**^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.79 – 7.65 (m, 2H), 7.54 – 7.40 (m, 4H), 7.38 – 7.23 (m, 4H),

5.62 – 5.46 (m, 2H), 4.36 – 4.20 (m, 4H), 1.75 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 138.2, 131.5, 127.0, 121.3, 51.6, 23.1. HRMS (ESI): calcd. for [$C_{20}H_{23}Br_2N_2O_4$, M+H]$^+$: 513.0019, found: 513.0024.

Optical Rotation: 2i $[\alpha]^{25}_D$ = -14.8 (c = 0.5, MeOH), 98% ee and *ent*-**2i** $[\alpha]^{25}_D$ = +13.1 (c = 0.5, MeOH), 99.9% ee, The absolute configuration of **2i** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 18.9 min, second peak: t_R = 20.4 min)

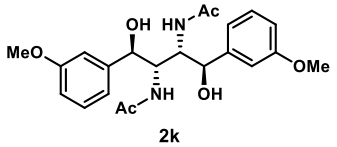


2j

***N,N*'-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-bis(4-(trifluoromethyl)phenyl)butane-2,3-diyl)diacetamide (2j)**

White solid, 23.8 mg, 97% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.1 Hz, 4H), 6.99 (d, *J* = 7.8 Hz, 6H), 5.79 (d, *J* = 12.3 Hz, 2H), 4.60 (dd, *J* = 12.3, 3.0 Hz, 2H), 4.02 (dd, *J* = 8.2, 3.0 Hz, 2H), 2.14 (s, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.7, 143.1, 129.6 (q, *J* = 32.8 Hz), 125.7, 125.5 (q, *J* = 3.0 Hz), 123.9 (q, *J* = 272.0 Hz), 75.0, 51.6, 29.7, 23.1. HRMS (ESI): calcd. for [$C_{22}H_{23}F_6N_2O_4$, M+H]$^+$: 493.1557, found: 493.1562.

Optical Rotation: 2j $[\alpha]^{25}_D$ = -10.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2j** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 13.5 min, second peak: t_R = 18.0 min)

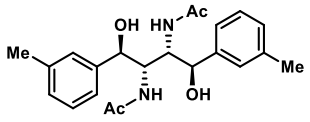


2k

***N,N*'-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-bis(3-methoxyphenyl)butane-2,3-diyl)diacetamide (2k)**

White solid, 20.4 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.09 – 7.00 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.71 – 6.54 (m, 4H), 6.43 – 6.33 (m, 2H), 5.61 – 5.38 (m, 2H), 4.59 – 4.46 (m, 2H), 4.18 – 4.04 (m, 2H), 3.66 (s, 6H), 2.10 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 159.4, 140.7, 129.3, 117.6, 113.5, 110.4, 75.4, 54.9, 52.0, 23.2. HRMS (ESI): calcd. for $[C_{22}H_{29}N_2O_6, M+H]^+$: 417.2020, found: 417.2027.

Optical Rotation: 2k $[\alpha]^{25}_D$ = -45.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2k** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 32.5 min, second peak: t_R = 55.5 min)

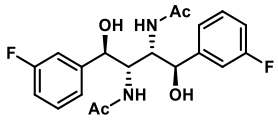


2l

***N,N*'-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-di-m-tolylbutane-2,3-diyl)diacetamide(2l)**

White solid, 18.8 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.04 – 6.99 (m, 2H), 6.97 – 6.93 (m, 2H), 6.83 – 6.78 (m, 2H), 6.69 – 6.58 (m, 4H), 5.49 – 5.18 (m, 2H), 4.56 – 4.48 (m, 2H), 4.12 – 4.08 (m, 2H), 2.17 (s, 6H), 2.13 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.2, 138.8, 137.8, 128.2, 128.0, 126.2, 122.4, 51.9, 23.3, 21.5. HRMS (ESI): calcd. for $[C_{22}H_{29}N_2O_4, M+H]^+$: 385.2122, found: 385.2127.

Optical Rotation: 2l $[\alpha]^{25}_D$ = -13.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2l** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 12.0 min, second peak: t_R = 20.4 min)



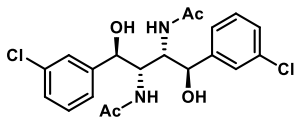
2m

***N,N*'-((*1R,2S,3S,4R*)-1,4-bis(3-fluorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2m)**

White solid, 19.4 mg, 99% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.14 – 7.08 (m,

2H), 6.97 (d, *J* = 8.3 Hz, 2H), 6.88 (dd, *J* = 8.4, 2.5 Hz, 2H), 6.79 (d, *J* = 7.7 Hz, 2H), 6.62 – 6.54 (m, 2H), 5.68 (d, *J* = 12.1 Hz, 2H), 4.54 (dd, *J* = 11.6, 3.7 Hz, 2H), 4.05 (dd, *J* = 8.2, 3.5 Hz, 2H), 2.10 (s, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.6, 162.6 (d, *J* = 246.7 Hz), 141.8 (d, *J* = 6.6 Hz), 123.0 (d, *J* = 8.2 Hz), 121.0 (d, *J* = 2.8 Hz), 114.5 (d, *J* = 21.2 Hz), 112.7 (d, *J* = 22.4 Hz), 74.7 (d, *J* = 1.8 Hz), 51.9, 23.0. HRMS (ESI): calcd. for $[C_{20}H_{22}N_2F_2NaO_4, M+Na]^+$: 415.1440, found: 415.1440.

Optical Rotation: 2m $[\alpha]^{25}_D$ = -95.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2m** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 21.9 min, second peak: t_R = 29.9 min)

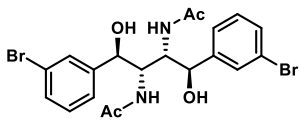


2n

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(3-chlorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2n)**

White solid, 20.4 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.19 – 7.15 (m, 2H), 7.10 – 7.05 (m, 2H), 6.91 – 6.85 (m, 4H), 6.78 (d, *J* = 8.3 Hz, 2H), 5.60 (d, *J* = 11.8 Hz, 2H), 4.57 – 4.47 (m, 2H), 4.08 – 4.00 (m, 2H), 2.11 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.5, 141.0, 134.4, 129.6, 128.0, 125.8, 123.4, 74.8, 51.8, 23.1. HRMS (ESI): calcd. for $[C_{20}H_{22}N_2Cl_2NaO_4, M+Na]^+$: 447.0848, found: 447.0833.

Optical Rotation: 2n $[\alpha]^{25}_D$ = -16.8 (c = 0.5, MeOH), 96% ee. The absolute configuration of **2n** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 16.9 min, second peak: t_R = 21.8 min)

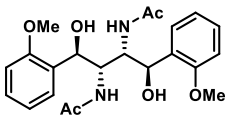


2o

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(3-bromophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2o)**

White solid, 24.4 mg, 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.38 – 7.31 (m, 2H), 7.11 – 6.88 (m, 8H), 5.79 – 5.55 (m, 2H), 4.61 – 4.46 (m, 2H), 4.14 – 3.98 (m, 2H), 2.10 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.6, 141.4, 131.0, 130.0, 128.8, 124.0, 122.7, 74.7, 52.0, 23.1. HRMS (ESI): calcd. for $[C_{20}H_{23}N_2Br_2NaO_4, M+H]^+$: 513.0019, found: 513.0012.

Optical Rotation: 2o $[\alpha]^{25}_D$ = -3.2 (c = 0.5, MeOH), 99.9% ee. and *ent*-**2o** $[\alpha]^{25}_D$ = +3.1 (c = 0.5, MeOH), 99.9% ee, The absolute configuration of **2o** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 35.3 min, second peak: t_R = 44.6 min)

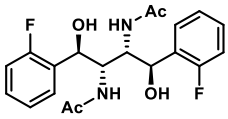


2p

***N,N'*-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-bis(2-methoxyphenyl)butane-2,3-diyl)diacetamide (2p)**

White solid, 20.0 mg, 99% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 7.6 Hz, 2H), 7.18 – 7.06 (m, 2H), 6.99 – 6.77 (m, 4H), 6.34 (d, *J* = 8.2 Hz, 2H), 5.71 (d, *J* = 11.8 Hz, 2H), 4.73 (dd, *J* = 11.9, 3.6 Hz, 2H), 4.26 (dd, *J* = 8.5, 3.6 Hz, 2H), 3.40 (s, 6H), 2.09 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 154.8, 128.0, 127.5, 126.8, 120.1, 109.5, 70.8, 54.6, 50.7, 23.1. HRMS (ESI): calcd. for $[C_{22}H_{28}N_2NaO_6, M+Na]^+$: 439.1839, found: 439.1842.

Optical Rotation: 2p $[\alpha]^{25}_D$ = -8.5 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2p** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 30.8 min, second peak: t_R = 39.7 min).

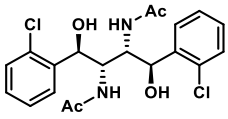


2q

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(2-fluorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2q)**

White solid, 19.2 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.64 – 7.54 (m, 1H), 7.22 – 7.14 (m, 2H), 6.63 – 6.50 (m, 2H), 5.84 – 5.72 (m, 1H), 4.84 – 4.74 (m, 1H), 4.25 – 4.18 (m, 1H), 2.10 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.6, 158.7 (d, *J* = 244.6 Hz), 129.1 (d, *J* = 8.3 Hz), 127.6 (d, *J* = 3.9 Hz), 126.6 (d, *J* = 13.4 Hz), 124.6 (d, *J* = 3.4 Hz), 114.9 (d, *J* = 21.8 Hz), 69.0 (d, *J* = 2.2 Hz), 52.1, 23.0. HRMS (ESI): calcd. for [$C_{20}H_{22}F_2N_2NaO_4$, M+Na]$^+$: 415.1440, found: 415.1442.

Optical Rotation: 2q $[\alpha]^{25}_D$ = -14.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2q** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R =24.2min, second peak: t_R = 58.1 min).



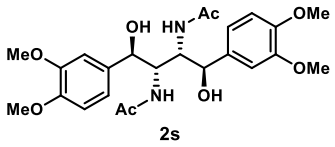
2r

***N,N'*-((*1R,2S,3S,4R*)-1,4-bis(2-chlorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2r)**

White solid, 20.1 mg, 99% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.63 (d, *J* = 7.8 Hz, 2H), 7.29 (m, 2H), 7.13 – 7.01 (m, 4H), 6.80 (d, *J* = 8.0 Hz, 2H), 6.07 (d, *J* = 11.6 Hz, 2H), 4.82 (dd, *J* = 11.7, 3.2 Hz, 2H), 4.35 (dd, *J* = 8.2, 3.3 Hz, 2H), 2.11 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 136.2, 130.9, 129.2, 128.5, 127.9, 127.4, 72.6, 49.8, 22.8. HRMS (ESI): calcd. For [$C_{20}H_{22}Cl_2N_2NaO_4$, M+Na]$^+$: 447.0848, found: 447.0846.

Optical Rotation: 2r $[\alpha]^{25}_D$ = -23.2 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2r** was assigned by analogy. (HPLC condition: Daicel Chiralcel OD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 0.5 ml/min, T = 25 ℃, wavelength = 220

nm, first peak: t_R = 12.6 min, second peak: t_R = 27.1 min).

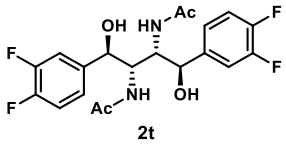


2s

***N,N*'-((*1R,2S,3S,4R*)-1,4-bis(3,4-dimethoxyphenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2s)**

White solid, 23.1 mg, 97% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.75 (d, *J* = 8.4 Hz, 2H), 6.59 (d, *J* = 8.1 Hz, 2H), 6.52 – 6.40 (m, 4H), 5.39 (d, *J* = 12.0 Hz, 2H), 4.57 – 4.48 (m, 2H), 4.08 (dd, *J* = 8.5, 3.3 Hz, 2H), 3.89 (s, 6H), 3.72 (s, 6H), 2.13 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 148.8, 147.8, 131.6, 117.5, 110.6, 108.2, 75.2, 55.7, 55.5, 51.9, 23.2. HRMS (ESI): calcd. for $[C_{24}H_{32}N_2NaO_8, M+Na]^+$: 499.2050, found: 499.2047.

Optical Rotation: 2s $[\alpha]^{25}_D$ = -67.2 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2s** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 32.2 min, second peak: t_R = 53.8 min).



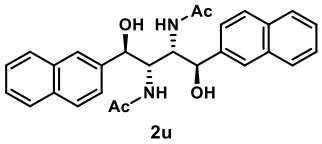
2t

***N,N*'-((*1R,2S,3S,4R*)-1,4-bis(3,4-difluorophenyl)-1,4-dihydroxybutane-2,3-diyl)diacetamide (2t)**

White solid, 20.9 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.99 (d, *J* = 8.8 Hz, 2H), 6.80 – 6.52 (m, 6H), 5.54 (d, *J* = 11.4 Hz, 2H), 4.54 (s, 2H), 4.04 – 3.92 (m, 2H), 2.14 (s, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.7, 149.2 (dd, *J* = 248.0, 12.6 Hz), 148.6 (dd, *J* = 248.3, 12.7 Hz), 135.4 (d, *J* = 4.3 Hz), 120.5 (q, *J* = 3.0 Hz), 116.2 (d, *J* = 17.4 Hz), 113.8 (d, *J* = 18.0 Hz), 73.2, 50.9, 22.0. HRMS (ESI): calcd. for $[C_{20}H_{20}F_4N_2NaO_4, M+Na]^+$: 451.1251, found: 451.1245.

Optical Rotation: 2t $[\alpha]^{25}_D$ = -10.4 (c = 0.5, MeOH), 99.9% ee and *ent*-**2t** $[\alpha]^{25}_D$ =

+10.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2t** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 12.3 min, second peak: t_R = 13.6 min).

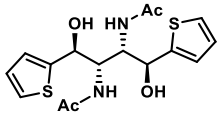


2u

***N,N*'-((*1R,2S,3S,4R*)-1,4-dihydroxy-1,4-di(naphthalen-2-yl)butane-2,3-diyl)diacetamide (2u)**

White solid, 21.9 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 – 7.55 (m, 2H), 7.50 – 7.42 (m, 6H), 7.42 – 7.35 (m, 2H), 7.05 – 6.96 (m, 2H), 6.88 – 6.78 (m, 2H), 6.59 – 6.51 (m, 2H), 5.83 – 5.70 (m, 2H), 4.74 – 4.62 (m, 2H), 4.33 – 4.21 (m, 2H), 2.16 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.5, 136.3, 132.9, 132.4, 128.1, 128.0, 127.5, 126.0, 125.7, 124.3, 122.7, 75.6, 51.6, 23.1. HRMS (ESI): calcd. for $[C_{28}H_{28}N_2NaO_4, M+Na]^+$: 479.1941, found: 479.1949.

Optical Rotation: 2u $[\alpha]^{25}_D$ = -76.5 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2u** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 33.9 min, second peak: t_R = 42.3 min).



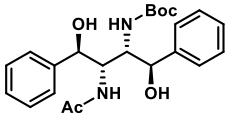
2v

***N,N*'-((*1S,2S,3S,4S*)-1,4-dihydroxy-1,4-di(thiophen-2-yl)butane-2,3-diyl)diacetamide (2v)**

White solid, 18.0 mg, 98% yield. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.15 – 7.97 (m, 2H), 7.42 – 7.33 (m, 2H), 7.04 – 6.89 (m, 4H), 6.07 – 5.78 (m, 2H), 4.63 – 4.50 (m, 2H), 4.33 – 4.22 (m, 2H), 1.83 (s, 6H). **^{13}C NMR** (101 MHz, DMSO) δ 171.6, 146.8, 126.6, 125.0, 124.7, 68.1, 56.0, 23.0. HRMS (ESI): calcd. for $[C_{16}H_{20}N_2S_2NaO_4$,

M+Na]$^+$: 391.0757, found: 391.0762.

Optical Rotation: 2v [α]$^{25}_D$ = -14.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2v** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 92:08, flow rate = 0.5 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 122.7 min, second peak: t_R = 134.7 min).

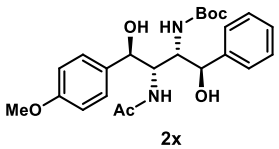


2w

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-1,4-diphenylbutan-2-yl)carbamate (2w)**

White solid, 19.6 mg, 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.19 – 7.11 (m, 6H), 7.04 – 6.95 (m, 4H), 6.39 (d, *J* = 8.6 Hz, 1H), 5.42 (d, *J* = 8.7 Hz, 1H), 5.09 (dd, *J* = 20.2, 10.6 Hz, 2H), 4.59 – 4.48 (m, 2H), 4.18 (dd, *J* = 8.7, 4.6 Hz, 1H), 3.97 (dd, *J* = 8.9, 4.6 Hz, 1H), 2.09 (s, 3H), 1.46 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.4, 139.6, 139.4, 128.6, 128.5, 127.2, 127.1, 125.8, 125.7, 81.1, 75.4, 68.0, 53.1, 52.2, 28.4, 25.6. HRMS (ESI): calcd. for [$C_{23}H_{30}N_2NaO_5$, M+Na]$^+$: 437.2046, found: 437.2045.

Optical Rotation: 2w [α]$^{25}_D$ = -16.0 (c = 0.5, MeOH), 99.9% ee and *ent*-**2w** [α]$^{25}_D$ = +16.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2w** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 13.8 min, second peak: t_R = 18.7 min).



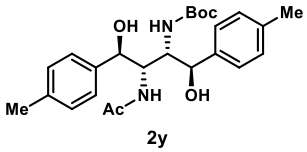
2x

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-4-(4-methoxyphenyl)-1-phenylbutan-2-yl)carbamate (2x)**

White solid, 21.3 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.16 (d, *J* = 6.5

Hz, 3H), 7.07 – 7.01 (m, 2H), 6.90 (d, *J* = 8.3 Hz, 2H), 6.68 (d, *J* = 8.3 Hz, 2H), 6.34 (d, *J* = 8.6 Hz, 1H), 5.38 (d, *J* = 8.8 Hz, 1H), 5.12 (d, *J* = 10.6 Hz, 1H), 4.95 (d, *J* = 10.6 Hz, 1H), 4.56 – 4.48 (m, 2H), 4.14 (dd, *J* = 8.9, 4.6 Hz, 1H), 3.99 (dd, *J* = 8.8, 4.6 Hz, 1H), 3.80 (s, 3H), 2.09 (s, 3H), 1.46 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 158.6, 157.3, 139.7, 131.5, 128.4, 127.1, 126.8, 125.8, 113.9, 81.1, 75.4, 75.0, 55.2, 53.0, 52.2, 28.3, 23.3. HRMS (ESI): calcd. for $[C_{24}H_{32}N_2NaO_6, M+Na]^+$: 467.2153, found: 467.2156.

Optical Rotation: 2x $[\alpha]^{25}_D$ = -33.6 (c = 0.5, MeOH), 99.9% ee and *ent*-**2x** $[\alpha]^{25}_D$ = +33.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2x** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 17.7 min, second peak: t_R = 24.4 min).

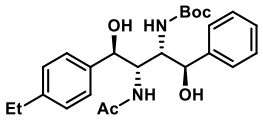


2y

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-1,4-di-p-tolylbutan-2-yl)carbamate (2y)**

White solid, 21.0 mg, 95% yield.^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.98 – 6.79 (m, 8H), 6.38 (d, *J* = 8.5 Hz, 1H), 5.39 (d, *J* = 8.7 Hz, 1H), 5.10 – 4.93 (m, 2H), 4.58 – 4.46 (m, 2H), 4.23 – 4.09 (m, 1H), 3.98 – 3.86 (m, 1H), 2.32 (s, 3H), 2.32 (s, 3H), 2.09 (s, 3H), 1.47 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 157.3, 136.6, 136.5, 136.3, 129.0, 128.9, 125.6, 125.5, 81.0, 75.4, 53.0, 52.0, 29.7, 28.3, 23.3, 21.1, 21.0. HRMS (ESI): calcd. for $[C_{25}H_{34}N_2NaO_5, M+Na]^+$: 465.2360, found: 465.2356.

Optical Rotation: 2y $[\alpha]^{25}_D$ = -29.7(c = 0.5, MeOH), 99.9% ee and *ent*-**2y** $[\alpha]^{25}_D$ = +29.7 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2y** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 10.8 min, second peak: t_R = 15.9 min).

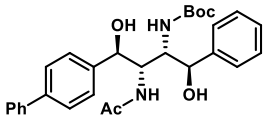


2z

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-4-(4-ethylphenyl)-1,4-dihydroxy-1-phenyl-butan-2-yl)carbamate (2z)**

White solid, 21.9 mg, 99% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.20 – 6.86 (m, 9H), 6.40 (d, *J* = 8.6 Hz, 1H), 5.43 (d, *J* = 8.8 Hz, 1H), 5.14 (d, *J* = 10.7 Hz, 1H), 4.98 (d, *J* = 10.9 Hz, 1H), 4.61 – 4.48 (m, 2H), 4.21 – 4.14 (m, 1H), 4.02 – 3.93 (m, 1H), 2.61 (q, *J* = 7.6 Hz, 2H), 2.09 (s, 3H), 1.46 (s, 9H), 1.23 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.4, 143.1, 139.7, 136.5, 128.4, 128.1, 127.1, 125.9, 125.6, 81.1, 75.5, 75.3, 53.0, 52.1, 28.5, 28.4, 23.3, 15.6. HRMS (ESI): calcd. for $[C_{27}H_{28}N_2NaO_5, M+Na]^+$: 465.2360, found: 465.2360.

Optical Rotation: 2z $[\alpha]^{25}_D$ = -13.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2z** $[\alpha]^{25}_D$ = +13.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2z** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 210 nm, first peak: t_R = 13.9 min, second peak: t_R = 16.6 min).

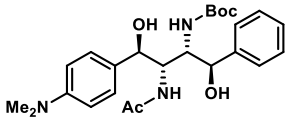


2aa

***tert*-butyl((*1R,2S,3S,4R*)-4-([1,1'-biphenyl]-4-yl)-3-acetamido-1,4-dihydroxy-1-phenylbutan-2-yl)carbamate(2aa)**

White solid, 23.3 mg, 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.40 – 7.34 (m, 3H), 7.18 – 7.00 (m, 7H), 6.53 (d, *J* = 8.6 Hz, 1H), 5.56 (d, *J* = 8.7 Hz, 1H), 5.24 – 5.11 (m, 2H), 4.63 – 4.53 (m, 2H), 4.21 (dd, *J* = 8.9, 4.4 Hz, 1H), 4.03 (d, *J* = 5.3 Hz, 1H), 2.10 (s, 3H), 1.47 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.4, 140.7, 139.8, 139.6, 138.4, 128.8, 128.3, 127.3, 127.1, 127.0, 126.9, 126.0, 125.8, 81.1, 75.4, 75.2, 53.4, 52.2, 28.3, 23.2. HRMS (ESI): calcd. for $[C_{29}H_{34}N_2NaO_5, M+Na]^+$:513.2350, found: 513.2354.

Optical Rotation: 2aa $[\alpha]^{25}_D$ = -64.0 (c = 0.5, MeOH), 99.9% ee and *ent*-**2aa** $[\alpha]^{25}_D$ = +63.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2aa** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 19.3 min, second peak: t_R = 23.0 min).

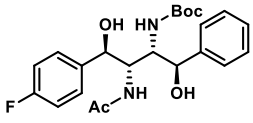


2ab

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-4-(4-(dimethylamino)phenyl)-1,4-dihydroxy-1-phenylbutan-2-yl)carbamate (2ab)**

White solid, 21.9 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.38 – 6.85 (m, 9H), 6.41 (d, *J* = 8.6 Hz, 1H), 5.46 (d, *J* = 8.6 Hz, 1H), 5.12 (dd, *J* = 10.5, 7.6 Hz, 2H), 4.56 (dd, *J* = 10.4, 4.7 Hz, 1H), 4.46 (dd, *J* = 10.6, 4.5 Hz, 1H), 4.18 – 4.10 (m, 1H), 3.98 (dd, *J* = 8.9, 4.8 Hz, 1H), 2.79 (s, 6H), 2.09 (s, 3H), 1.45 (s, 9H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.5, 150.8, 139.4, 135.3, 131.5, 128.3, 127.5, 125.7, 125.3, 120.3, 119.0, 81.2, 75.2, 74.5, 53.3, 52.1, 44.2, 28.3, 23.2. HRMS (ESI): calcd. for $[C_{25}H_{35}N_3NaO_5, M+Na]^+$: 480.2469, found: 480.2475.

Optical Rotation: 2ab $[\alpha]^{25}_D$ = -13.8 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ab** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 210 nm, first peak: t_R = 11.1 min, second peak: t_R = 16.6 min)



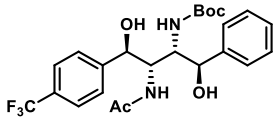
2ac

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-4-(4-fluorophenyl)-1,4-dihydroxy-1-phenylbutan-2-yl)carbamate (2ac)**

White solid, 20.5 mg, 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.22 – 7.14 (m, 3H), 7.06 – 7.01 (m, 2H), 6.96 – 6.90 (m, 2H), 6.81 (t, *J* = 8.5 Hz, 2H), 6.44 (d, *J* = 8.6 Hz, 1H), 5.48 (d, *J* = 8.7 Hz, 1H), 5.12 (d, *J* = 10.5 Hz, 2H), 4.56 – 4.49 (m, 2H), 4.16

(dd, *J* = 8.8, 4.5 Hz, 1H), 3.98 – 3.87 (m, 1H), 2.09 (s, 3H), 1.45 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.1, 161.8 (d, *J* = 245.5 Hz), 157.5, 139.7, 135.2, 128.4, 127.3, 127.2, 125.8, 115.3 (d, *J* = 21.6 Hz), 81.2, 75.3, 74.8, 53.4, 52.1, 28.3, 23.2. HRMS (ESI): calcd. for $[C_{23}H_{29}FN_2NaO_5, M+Na]^+$: 455.1952, found: 455.1941.

Optical Rotation: 2ac $[\alpha]^{25}_D$ = -5.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2ac** $[\alpha]^{25}_D$ = +5.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ac** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 10.4 min, second peak: t_R = 13.7 min).

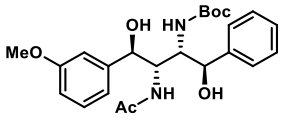


2ad

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-1-phenyl-4-(4-(trifluoromethyl)phenyl)butan-2-yl)carbamate (2ad)**

White solid, 23.4 mg, 97% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.20 – 7.07 (m, 3H), 6.99 (dd, *J* = 26.0, 7.8 Hz, 4H), 6.48 (d, *J* = 8.6 Hz, 1H), 5.56 (d, *J* = 8.6 Hz, 1H), 5.35 (d, *J* = 11.7 Hz, 1H), 5.08 (d, *J* = 11.5 Hz, 1H), 4.66 – 4.52 (m, 2H), 4.19 (dd, *J* = 8.8, 3.6 Hz, 1H), 3.77 (dd, *J* = 8.5, 3.7 Hz, 1H), 2.15 (s, 3H), 1.49 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.1, 157.6, 143.4, 139.3, 128.5, 127.2, 125.6 (q, *J* = 23.6 Hz), 125.4 (q, *J* = 3.9 Hz), 121.4 (d, *J* = 272.5 Hz), 81.5, 75.7, 75.3, 52.9, 51.4, 28.3, 23.3. HRMS (ESI): calcd. for $[C_{27}H_{28}N_2NaO_5, M+Na]^+$: 505.1921, found: 505.1927.

Optical Rotation: 2ad $[\alpha]^{25}_D$ = -24.8 (c = 0.5, MeOH), 99% ee and *ent*-**2ad** $[\alpha]^{25}_D$ = +24.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ad** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 9.4 min, second peak: t_R = 10.3 min).

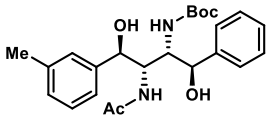


2ae

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-4-(3-methoxyphenyl)-1-phenylbutan-2-yl)carbamate (2ae)**

White solid, 21.3 mg, 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.17 – 7.08 (m, 4H), 7.01 – 6.93 (m, 2H), 6.78 – 6.65 (m, 2H), 6.45 – 6.35 (m, 2H), 5.44 (d, *J* = 8.7 Hz, 1H), 5.19 – 5.04 (m, 2H), 4.59 – 4.47 (m, 2H), 4.13 – 3.97 (m, 2H), 3.66 (s, 3H), 2.10 (s, 3H), 1.47 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 159.6, 157.4, 141.0, 139.5, 129.6, 128.3, 127.0, 125.6, 117.9, 113.6, 110.7, 81.2, 75.6, 75.4, 68.0, 54.9, 52.9, 52.2, 28.4, 23.3. HRMS (ESI): calcd. for $[C_{27}H_{28}N_2NaO_5, M+Na]^+$: 467.2152, found: 467.2158.

Optical Rotation: 2ae $[\alpha]^{25}_D$ = -8.8 (c = 0.5, MeOH), 99.9% ee and *ent*-**2ae** $[\alpha]^{25}_D$ = +8.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ae** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 16.6 min, second peak: t_R = 22.8 min).

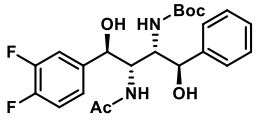


2af

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-1-phenyl-4-(m-tolyl)butan-2-yl)carbamate (2af)**

White solid, 20.3 mg, 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.16 – 7.08 (m, 4H), 6.96 (dd, *J* = 23.0, 7.5 Hz, 4H), 6.68 (s, 1H), 6.39 (d, *J* = 8.6 Hz, 1H), 5.44 (d, *J* = 8.7 Hz, 1H), 5.09 (dd, *J* = 19.5, 10.8 Hz, 2H), 4.58 – 4.46 (m, 2H), 4.15 – 3.98 (m, 2H), 2.19 (s, 3H), 2.09 (s, 3H), 1.46 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.4, 139.5, 139.4, 138.0, 128.3, 128.2, 127.0, 126.6, 125.7, 122.7, 81.0, 75.4, 75.2, 53.4, 52.5, 28.3, 23.2, 21.5. HRMS (ESI): calcd. for $[C_{24}H_{32}N_2NaO_5, M+Na]^+$: 451.2203, found: 451.2210.

Optical Rotation: 2af $[\alpha]^{25}_D$ = -11.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2af** $[\alpha]^{25}_D$ = +11.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2af** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 11.2 min, second peak: t_R = 14.8 min).

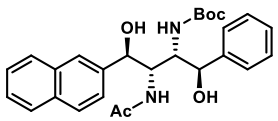


2ag

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-4-(3,4-difluorophenyl)-1,4-dihydroxy-1-phenylbutan-2-yl)carbamate (2ag)**

White solid, 22.0 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.25 – 7.15 (m, 3H), 7.03 (d, *J* = 6.7 Hz, 2H), 6.98 – 6.87 (m, 1H), 6.75 – 6.60 (m, 2H), 6.42 (d, *J* = 8.7 Hz, 1H), 5.52 (d, *J* = 8.5 Hz, 1H), 5.29 (d, *J* = 11.3 Hz, 1H), 5.09 (d, *J* = 11.2 Hz, 1H), 4.64 – 4.47 (m, 2H), 4.12 (dd, *J* = 9.1, 3.9 Hz, 1H), 3.81 (dd, *J* = 8.7, 4.1 Hz, 1H), 2.13 (s, 3H), 1.47 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.0, 157.5, 150.0 (dd, *J* = 248.7, 12.7 Hz). 149.5 (dd, *J* = 247.6, 12.7 Hz), 139.4, 136.6, 128.3, 127.5, 125.6, 121.4 (d, *J* = 6.0 Hz), 117.1 (d, *J* = 17.3 Hz), 114.9 (d, *J* = 17.8 Hz), 81.5, 75.5, 74.6, 53.1, 51.5, 28.3, 23.3. HRMS (ESI): calcd. for $[C_{23}H_{28}F_2N_2NaO_5, M+Na]^+$: 473.1858, found: 473.1855.

Optical Rotation: 2ag $[\alpha]^{25}_D$ = -55.5 (c = 0.5, MeOH), 99.9% ee and *ent*-**2ag** $[\alpha]^{25}_D$ = +51.0 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ag** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 18.8 min, second peak: t_R = 23.0 min).

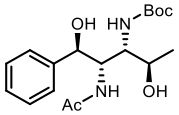


2ah

***tert*-butyl((*1R,2S,3S,4R*)-3-acetamido-1,4-dihydroxy-4-(naphthalen-2-yl)-1-phenylbutan-2-yl)carbamate (2ah)**

White solid, 24.1 mg, 98% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.82 – 7.76 (m, 1H), 7.73 – 7.67 (m, 1H), 7.59 – 7.45 (m, 4H), 7.05 – 6.95 (m, 2H), 6.92 – 6.75 (m, 4H), 6.48 (d, *J* = 8.4 Hz, 1H), 5.49 (d, *J* = 8.6 Hz, 1H), 5.31 (d, *J* = 10.9 Hz, 1H), 5.13 (d, *J* = 10.7 Hz, 1H), 4.77 – 4.70 (m, 1H), 4.56 – 4.47 (m, 1H), 4.25 (dd, *J* = 8.5, 4.3 Hz, 1H), 4.03 (dd, *J* = 8.7, 4.5 Hz, 1H), 2.09 (s, 3H), 1.48 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.1, 157.4, 139.3, 136.9, 133.1, 132.7, 128.4, 128.3, 128.1, 127.5, 127.1, 125.9, 125.5, 125.0, 123.3, 81.2, 75.6, 75.3, 53.0, 52.1, 28.3, 23.3. HRMS (ESI): calcd. for $[C_{27}H_{32}N_2NaO_5, M+Na]^+$: 487.2203, found: 487.2209.

Optical Rotation: 2ah $[\alpha]^{25}_D$ = -15.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2ah** $[\alpha]^{25}_D$ = +16.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ah** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.0 ml/min, T = 25 ℃, wavelength = 220 nm, first peak: t_R = 17.0 min, second peak: t_R = 22.8 min)



***tert*-butyl((*1R,2S,3S,4R*)-2-acetamido-1,4-dihydroxy-1-phenylpentan-3-yl)carbamate (2ai)**

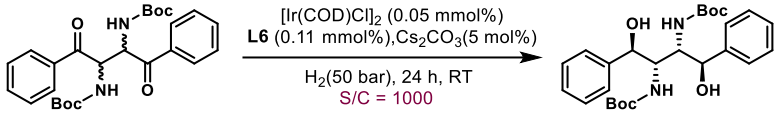
White solid, 17.1 mg, 97% yield. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.62 (d, *J* = 9.8 Hz, 1H), 7.33 – 7.18 (m, 6H), 6.39 (d, *J* = 9.6 Hz, 1H), 5.36 (d, *J* = 4.5 Hz, 1H), 4.68 (d, *J* = 4.2 Hz, 1H), 4.40 – 4.32 (m, 1H), 4.17 (td, *J* = 9.6, 2.0 Hz, 1H), 3.77 (td, *J* = 9.5, 2.0 Hz, 1H), 1.61 (s, 3H), 1.43 (s, 9H), 1.04 (d, *J* = 6.1 Hz, 3H). **^{13}C NMR** (101 MHz, DMSO) δ 170.8, 156.8, 143.8, 128.1, 127.5, 127.4, 78.7, 72.1, 66.7, 56.7, 54.5, 40.0, 28.8, 22.7, 20.4. HRMS (ESI): calcd. for $[C_{18}H_{28}N_2NaO_5, M+Na]^+$: 375.1890, found: 375.1893.

Optical Rotation: 2ah $[\alpha]^{25}_D$ = -5.2 (c = 0.5, MeOH), 99.9% ee and *ent*-**2ai** $[\alpha]^{25}_D$ = +5.1 (c = 0.5, MeOH), 99.9% ee. The absolute configuration of **2ai** was assigned by analogy. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH = 90:10,

flow rate = 1.0 ml/min, T = 25 °C, wavelength = 220 nm, first peak: t_R = 9.2 min, second peak: t_R = 14.5 min)

5. Gram-scale Reaction and Applications

1. DKR of **1a** with 0.1 mol% catalyst loading in gram scale

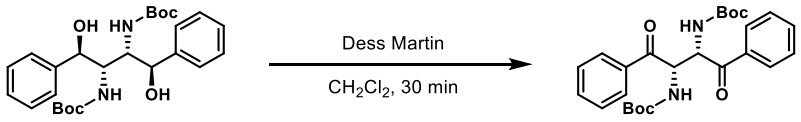


1a
2.18 mmol, 1.02 g

2a
1.01 g, 98% yield

To a 4.0 mL vial was added the catalyst precursor $[Ir(COD)Cl]_2$ (3.3 mg, 5×10^{-3} mmol, 1.0 eq.), (S_C, S_C, R_{FC})-**L6** (6.3 mg, 1.1×10^{-2}mmol, 2.2 eq.) and anhydrous *i*-PrOH (1.0 mL) in the argon-filled glovebox. The mixture was stirred for 1.0 h at 25 ℃. The resulting orange solution (436 μL) was transferred by syringe into a glass cup (50.0 mL) charged with substrate **1a** (1.02 g, 2.18 mmol), Cs_2CO_3 (35 mg, 0.11 mmol) and anhydrous THF (10.0 mL). The cup was transferred to an autoclave, which was then charged with of H_2 (50 bar) and stirred at room temperature for 24 h. The hydrogen gas was released slowly in a well-ventilated hood and the solution was concentrated and purified by flash chromatography on silica gel (CH_2Cl_2/MeOH, 10:1) to afford the target product **2a**.

2. Oxidation reaction of **2a**[2]

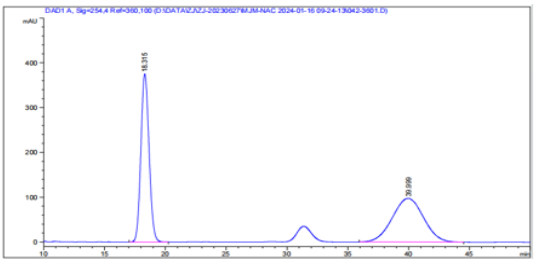


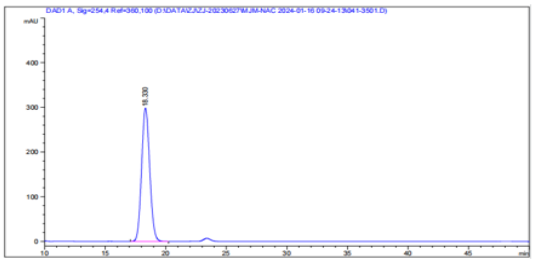
99% ee, 99% yield

To a suspension of Dess-Martin (2.1 mmol, 10 eq.) in 5 mL of anhydrous CH_2C1_2 was added the solution of **2a** (100 mg, 0.21 mmol) in 5.0 mL of CH_2C1_2 and the dark-brown mixture was stirred at room temperature for 30 min. ether (10.0 mL) was added to the reaction mixture and the mixture was filtered, and the black deposit was washed with ether. The filtrate was concentrated under reduced pressure to give the amino ketone **(*S*,*S*)-1a** (99 mg, 99% yield, 99% ee).

(*S, S*)-1a: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.7 Hz, 4H), 7.66 – 7.58 (m, 2H), 7.53 – 7.46 (m, 4H), 5.66 (d, *J* = 8.5 Hz, 2H), 5.48 (d, *J* = 8.7 Hz, 2H), 1.41 (s, 18H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 155.1, 135.0, 133.7, 128.8, 128.7, 80.3, 56.0, 28.2. (HPLC condition: Daicel Chiralcel AD-H Column, *n*-hexane/*i*-PrOH =

90:10, flow rate = 1.0 ml/min, T = 25 °C, wavelength = 254 nm, first peak: t_R = 18.3 min, second peak: t_R = 40.0 min).





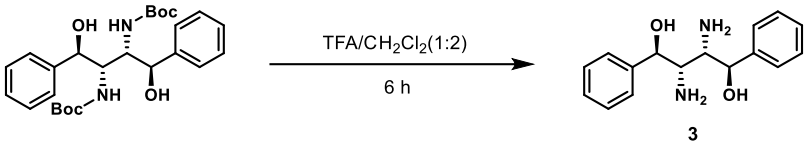
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.315	BB	0.7289	1.75126e4	374.94489	50.1023
2	39.999	BB	2.1300	1.74411e4	97.54676	49.8977
Totals :				3.49537e4	472.49165	

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.330	BB	0.7257	1.39061e4	298.37100	100.0000
Totals :				1.39061e4	298.37100	

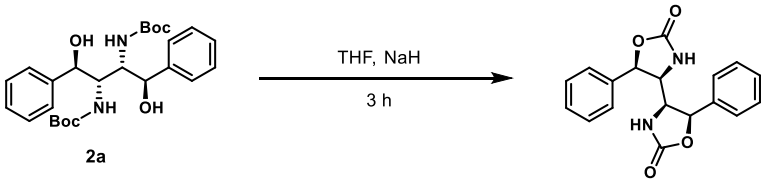
3. Deprotection of **2a**[3]



3
58 mg, 99% yield

A solution of **2a** (100.0 mg, 0.21 mmol) in CH_2C1_2 (2 mL) was treated with TFA (1 mL) and stirred for 6 hours. The solvent was concentrated under reduced pressure to give compound **3** (58.0 mg, 99% yield). The material was used in the following step without further purification. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.42 (s, 4H), 7.33 – 7.25 (m, 6H), 7.20 (s, 4H), 5.13 (d, J = 4.0 Hz, 2H), 3.38 (s, 2H). ^{13}C NMR (101 MHz, DMSO) δ 140.2, 128.9, 128.4, 126.1, 72.2, 53.0. HRMS (ESI): calcd. for $[C_{16}H_{20}N_2NaO_2, M+Na]^+$: 295.1417, found: 295.1417.

4. The synthesis of compound **4**[4]



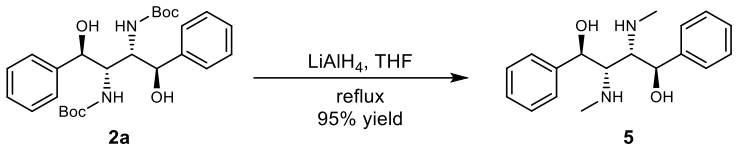
2a

4
90% yield

To an oven dried round bottom flask was added sodium hydride (60% dispersion in mineral oil, 0.25 mmol, 1.2 eq.). To this flask was added a solution of **2a** (100 mg, 0.21 mmol, 1.0 eq.) in THF (5.0 mL) slowly at 0 °C under argon. Then the flask was

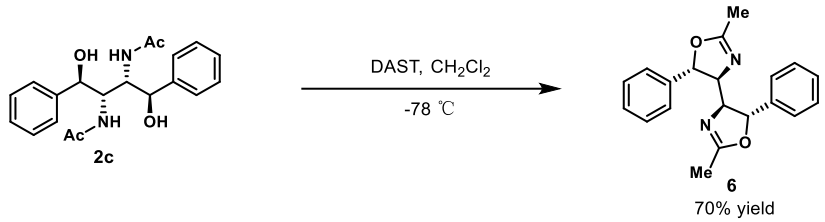
equipped with a reflux condenser and the reaction mixture was heated to reflux and reacted for 3h. The mixture was cooled down to room temperature, quenched with saturated aqueous NH_4Cl solution, and transferred to a separatory funnel. The mixture was extracted with CH_2Cl_2 three times. The combined organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated to afford the crude product, which was purified by column chromatography on silica gel (PE/EtOAc = 3/1) to afford the pure product **4** as a white solid(61.2 mg, 90% yield). ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.66 (s, 2H), 7.44 (d, J = 6.7 Hz, 6H), 7.34 – 7.29 (m, 4H), 5.25 (d, J = 6.8 Hz, 2H), 3.63 (d, J = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, DMSO) δ 158.4, 135.1, 129.4, 129.3, 127.4, 79.4, 56.0, 28.5. HRMS (ESI): calcd. for $[C_{18}H_{16}N_2NaO_4, M+Na]^+$: 347.1002, found: 347.1001.

5. The synthesis of compound **5**[5]



Compound **2a** (200 mg, 0.42 mmol) was dissolved in 25 mL of tetrahydrofuran (THF). Lithium aluminum hydride (64 mg, 1.69 mmol) was added portion-wise at 0 °C. The reaction mixture was stirred under reflux for 6 h and then quenched sequentially at 0 °C with water (2 mL), NH_4Cl aq. (4 mL), and water (2 mL). The resulting suspension was allowed to warm to room temperature and filtered using a Büchner funnel. The solid residue was washed with THF (10 mL). The combined organic phases were concentrated under reduced pressure. The aqueous layer was extracted with dichloromethane (3 × 20 mL). The combined organic layers were washed with brine (10 mL) and then dried over anhydrous Na_2SO_4. The solvents were evaporated under reduced pressure. The crude material was combined and purified by flash column chromatography on silica gel using 100:1 DCM:MeOH as the eluent. **5** (120 mg, 95% yield) was obtained as a white solid. ^{1}H NMR (400 MHz, Chloroform-d) δ 7.32 – 7.26 (m, 4H), 7.22 (d, J = 7.1 Hz, 2H), 7.12 – 7.05 (m, 4H), 4.95 (d, J = 2.8 Hz, 2H), 2.44 (s, 6H), 2.41 (d, J = 2.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.2, 128.2, 126.6, 124.7, 61.8, 33.7.

6. The synthesis of bisoxazoline ligand **6**[6]

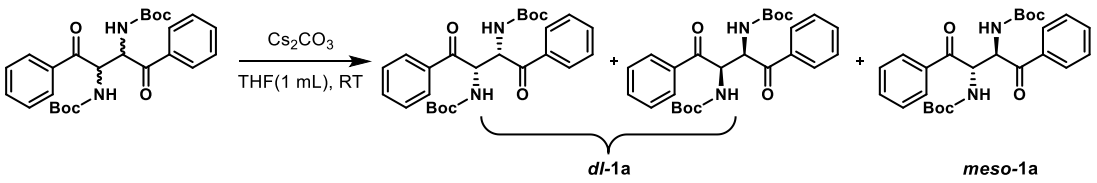


The amino alcohol **2c** (60 mg, 0.17 mmol) dissolved in CH_2Cl_2 (5 ml) was cooled at -78 ℃ under nitrogen and DAST (0.19 mmol, 1.1 eq.) was slowly added. After completion of the reaction (< 1 h), the medium was hydrolysed with crushed ice added with 4 M NH_4OH (1.0 ml) at -78 ℃. The aqueous layer was then extracted with CH_2Cl_2 (3×15 ml), and the combined organic layers dried (Na_2SO_4), filtered and concentrated leaving an oily residue, which was subjected to flash chromatography on silica gel (CH_2Cl_2: MeOH = 20:1), to give the purified **5** (38 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.24 (m, 10H), 5.42 (d, *J* = 5.9 Hz, 2H), 4.25 (d, *J* = 5.7 Hz, 2H), 2.15 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 140.6, 128.8, 128.3, 125.7, 82.8, 77.2, 14.0. HRMS (ESI): calcd. for $[C_{20}H_{20}N_2NaO_2, M+Na]^+$: 343.1417, found: 343.1420.

6. Experimental Study of Mechanism

1)The effect of base on the content of *dl*-**1a** and *meso*-**1a**

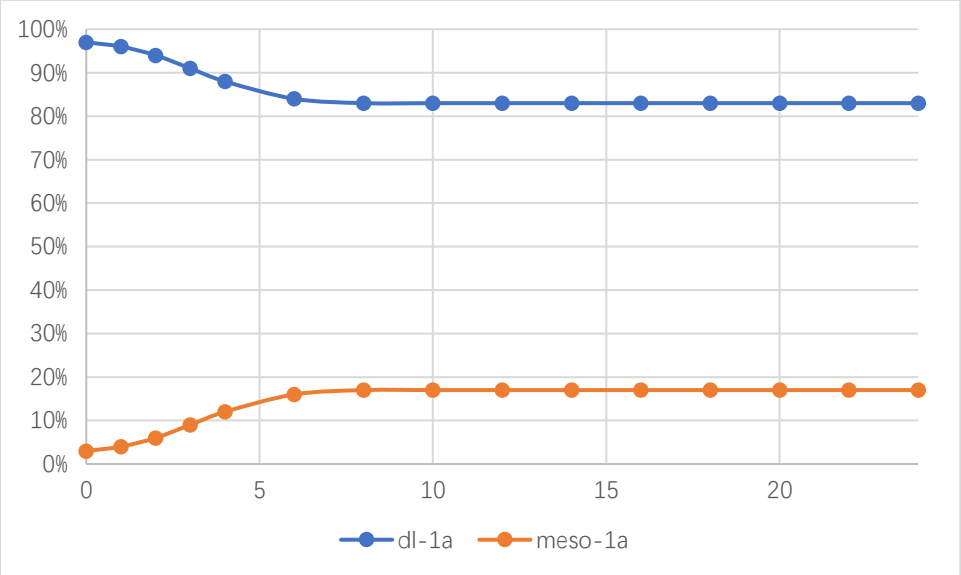
The epimerization of **1a** (23.4 mg, 0.05 mmol, dl : meso = 97 : 3) were conducted in the presence of Cs_2CO_3 (0.8 mg, 5 mol%) in THF (1 mL) at room temperature for 24 h, then the ratio of dl/ meso was detected by high performance liquid chromatography analysis on a chiral stationary phase, and the result shows that the ratio of *meso*-**1a** to *dl*-**1a** was gradually increased in the first 8 hours, then an equilibrium was reached. Further prolonging the reaction time, there is no change in the ratio of *meso*-**1a** to *dl*-**1a**, and the ratio remained at 17: 83.



Supplementary Table 3. **Variation in the content of *meso*-1a and *dl*-1a over time in the presence of base**

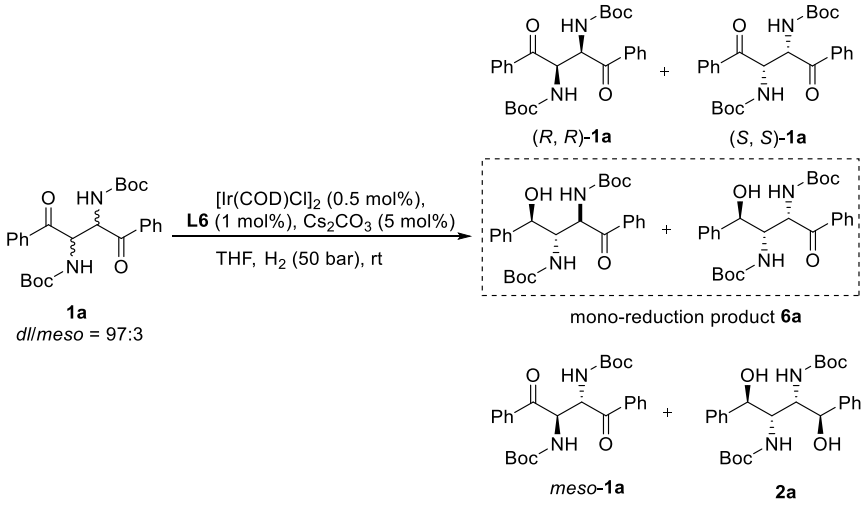
Entry	Time (h)	*dl*-**1a**	*meso*-**1a**
1	0	97%	3%
2	1	96%	4%
3	2	94%	6%
4	3	91%	9%
5	4	88%	12%
6	6	84%	16%
7	8	83%	17%
8	10	83%	17%
9	12	83%	17%
10	14	83%	17%
11	16	83%	17%
12	18	83%	17%
13	20	83%	17%

14	22	83%	17%
15	24	83%	17%



Supplementary Figure 1. Variation in the content of *meso*-**1a** and *dl*-**1a** over time in the presence of base

The change in product distribution over reaction time

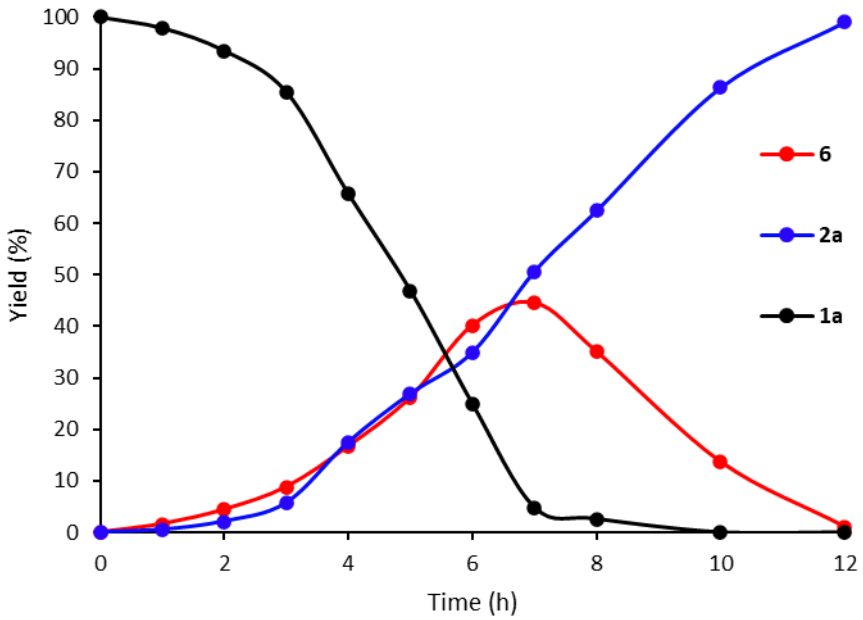


General procedure: To a 4.0 mL vial was added the catalyst precursor $[Ir(COD)Cl]_2$ (3.3 mg, 5 $\times 10^{-3}$ mmol, 1.0 eq.), (S_C, S_C, R_{FC})-L6 (6.3 mg, 1.1 $\times 10^{-2}$mmol, 2.2 eq.) and anhydrous i-PrOH (1.0 mL) in the argon-filled glovebox. The mixture was stirred for 1.0 h at 25 °C. The resulting orange solution (213 μL) was transferred by syringe into

a vial (8.0 mL) charged with substrate 2a (100 mg, 0.21 mmol), Cs_2CO_3 (3.5 mg, 0.01 mmol) and anhydrous THF (4.0 mL). The vial was transferred to an autoclave, which was then charged with of H_2 (50 bar) and stirred at room temperature for the 1 to 12 hours. The hydrogen gas was released slowly in a well-ventilated hood and the solution was concentrated and passed through a short column of silica gel (eluant: CH_2Cl_2/MeOH, 10:1) to remove the metal complex. The yield and ee value were detected by HPLC.

Supplementary Table 4: Yield variation for the hydrogenation of **1a** over time

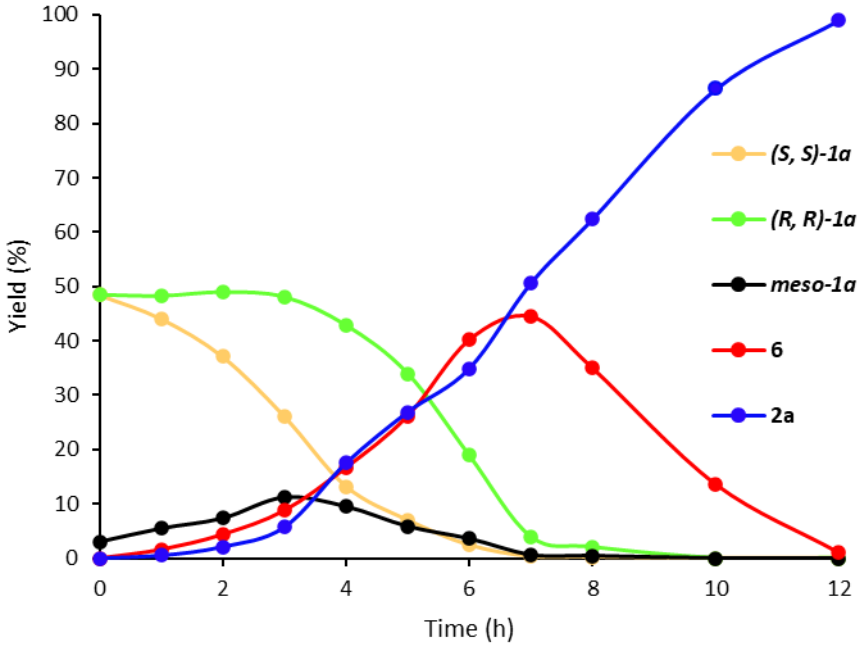
Entry	Time (h)	Yield (%)		
		6	**1a**	**2a**
0	0	0	100	0
1	1	1.6	97.9	0.5
2	2	4.4	93.5	2.1
3	3	8.8	85.5	5.7
4	4	16.7	65.7	17.5
5	5	26.2	46.9	26.9
6	6	40.2	24.9	34.9
7	7	44.5	4.8	50.6
8	8	35	2.6	62.4
9	10	13.6	trace	86.3
10	12	1	trace	99



Supplementary Figure 2. **Yield variation for the hydrogenation of 1a over time**

Supplementary Table 5: **Yield variation over time**

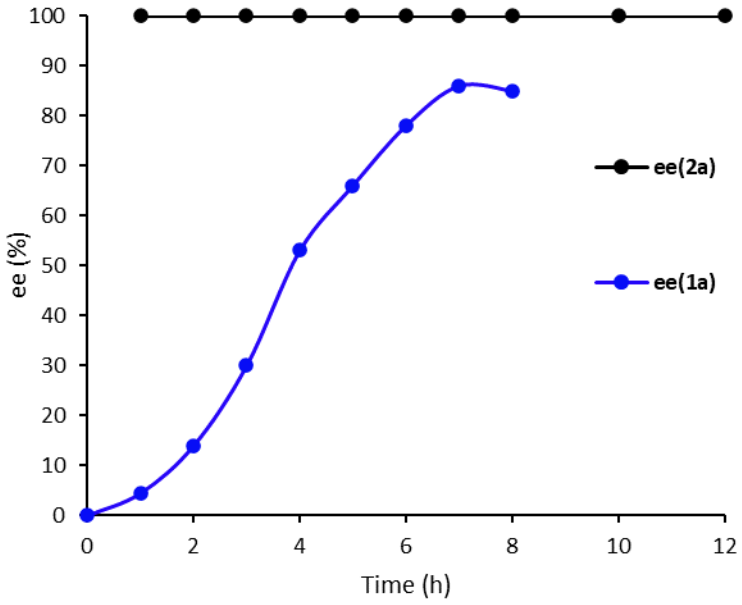
Entry	Time (h)	Yield (%)				
		(*S, S*)-**1a**	(*R, R*)-**1a**	*meso*-**1a**	**6**	**2a**
0	0	48.5	48.5	3	0	0
1	1	44.1	48.3	5.5	1.6	0.5
2	2	37.1	49	7.4	4.4	2.1
3	3	26.1	48.1	11.3	8.8	5.7
4	4	13.2	42.9	9.6	16.7	17.5
5	5	7	34	5.9	26.2	26.9
6	6	2.4	18.9	3.6	40.2	34.9
7	7	0.3	3.9	0.6	44.5	50.6
8	8	0.2	2	0.4	35	62.4
9	10	trace	trace	trace	13.6	86.3
10	12	0	0	0	1	99



Supplementary Figure 3. Yield variation over time

Supplementary Table 6. ee variation over time

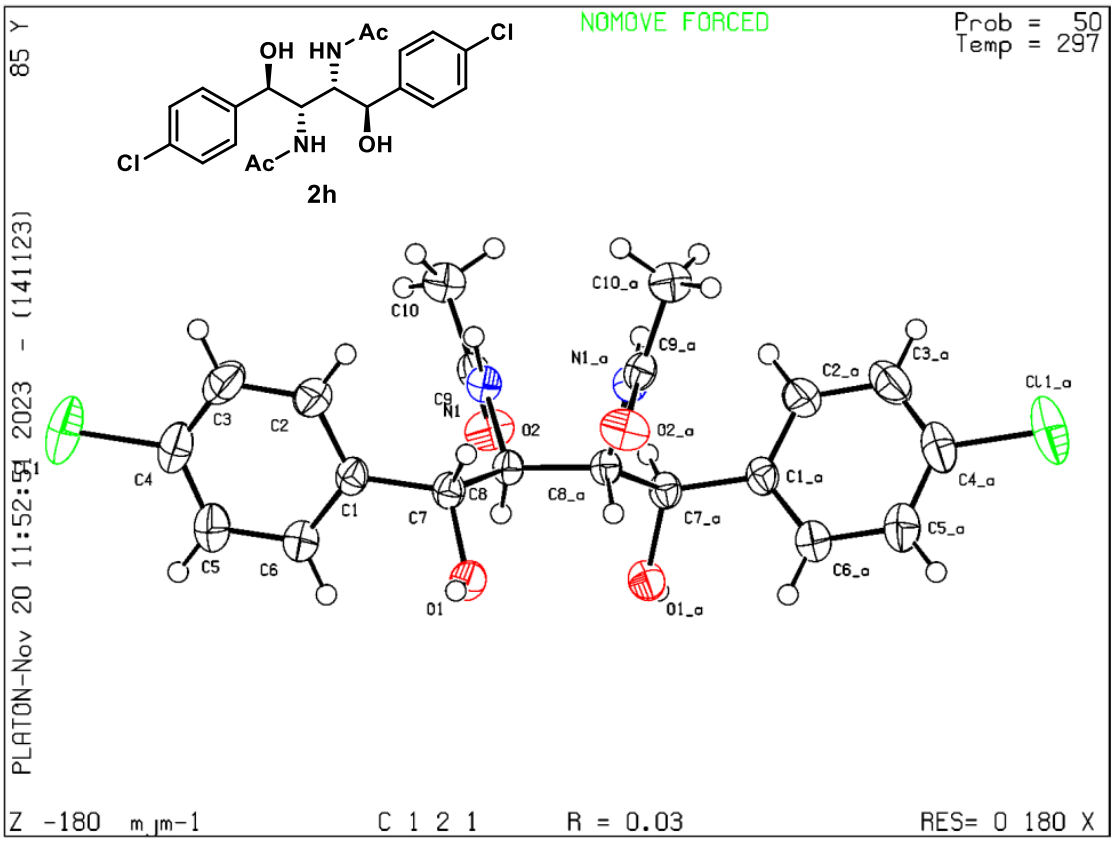
Entry	Time (h)	ee of *dl*-**1a** (%)	ee of **2a** (%)
0	0	0	-
1	1	4.5	99.9
2	2	14	99.9
3	3	30	99.9
4	4	53	99.9
5	5	66	99.9
6	6	78	99.9
7	7	86	99.9
8	8	85	99.9
9	10	-	99.9
10	12	-	99.9



Supplementary Figure 4. ee variation over time

7. The absolute configurations of 2h

Crystal **2h** was obtained through recrystallization in the solution of DCM at room temperature. The absolute configuration of **2h** was confirmed unambiguously to be (1*R*, 2*S*, 3*S*, 4*R*) by X-ray diffraction analysis, and other compounds were assigned by analogy. CCDC 2364014 contains the supplementary crystallographic data for this compound.



Supplementary Figure 5 X-ray structure of **2h**

Supplementary Table 7. Crystal data and structure refinement for 2h.

Identification code	mjm-1
Empirical formula	C20 H22 Cl2 N2 O4
Formula weight	425.29
Temperature	297.1(2) K

Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	C 1 2 1
Unit cell dimensions	a = 14.0831(3) Å a= 90°.
	b = 5.70860(10) Å b= 100.654(2)°.
	c = 12.3886(3) Å g = 90°.
Volume	978.81(4) $Å^3$
Z	2
Density (calculated)	1.443 Mg/m^3
Absorption coefficient	3.240 mm^{-1}
F(000)	444
Crystal size	0.05 x 0.04 x 0.03 mm^3
Theta range for data collection	3.630 to 74.797°.
Index ranges	-16<=h<=17, -6<=k<=7, -15<=l<=13
Reflections collected	4226
Independent reflections	1906 [R(int) = 0.0220]
Completeness to theta = 67.684°	99.0%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.57058
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1906 / 1 / 133
Goodness-of-fit on F^2	1.071
Final R indices [I>2sigma(I)]	R1 = 0.0330, wR2 = 0.0877
R indices (all data)	R1 = 0.0332, wR2 = 0.0879
Absolute structure parameter	0.016(10)
Extinction coefficient	n/a
Largest diff. peak and hole	0.173 and -0.317 $e.Å^{-3}$

Supplementary Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 2h. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	3582(1)	5013(2)	3854(1)	72(1)
O(1)	3840(1)	1003(3)	8987(1)	33(1)
O(3)	6858(1)	5022(3)	8926(2)	40(1)
N(2)	5347(1)	6246(4)	9026(2)	27(1)
C(9)	6239(2)	6580(4)	8796(2)	27(1)
C(4)	3878(2)	3790(4)	7501(2)	28(1)
C(8)	5001(1)	4019(4)	9376(2)	24(1)
C(7)	3987(2)	3408(4)	8728(2)	27(1)
C(10)	6438(2)	8954(5)	8392(2)	36(1)
C(5)	4121(2)	2056(5)	6819(2)	36(1)
C(1)	3694(2)	4551(6)	5263(2)	44(1)
C(3)	3514(2)	5883(5)	7037(2)	40(1)
C(6)	4023(2)	2416(6)	5695(2)	45(1)
C(2)	3426(2)	6283(6)	5914(3)	47(1)

Supplementary Table 9. Bond lengths [Å] and angles [°] for 2h.

Cl(1)-C(1)	1.743(3)
O(1)-H(1)	0.8200
O(1)-C(7)	1.434(3)
O(3)-C(9)	1.235(3)
N(2)-C(9)	1.352(3)
N(2)-C(8)	1.456(3)
N(2)-H(2)	0.90(4)

C(9)-C(10)	1.489(4)
C(4)-C(7)	1.514(3)
C(4)-C(5)	1.384(4)
C(4)-C(3)	1.383(4)
C(8)-C(8)#1	1.547(4)
C(8)-H(8)	0.9800
C(8)-C(7)	1.544(3)
C(7)-H(7)	0.9800
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(5)-H(5)	0.9300
C(5)-C(6)	1.390(4)
C(1)-C(6)	1.376(5)
C(1)-C(2)	1.372(5)
C(3)-H(3)	0.9300
C(3)-C(2)	1.393(4)
C(6)-H(6)	0.9300
C(2)-H(2A)	0.9300

C(7)-O(1)-H(1)	109.5
C(9)-N(2)-C(8)	124.1(2)
C(9)-N(2)-H(2)	117(3)
C(8)-N(2)-H(2)	119(3)
O(3)-C(9)-N(2)	122.1(2)
O(3)-C(9)-C(10)	121.8(2)
N(2)-C(9)-C(10)	116.1(2)
C(5)-C(4)-C(7)	121.3(2)
C(3)-C(4)-C(7)	120.2(2)
C(3)-C(4)-C(5)	118.6(2)

N(2)-C(8)-C(8)#1	111.11(13)
N(2)-C(8)-H(8)	107.8
N(2)-C(8)-C(7)	111.76(18)
C(8)#1-C(8)-H(8)	107.8
C(7)-C(8)-C(8)#1	110.3(2)
C(7)-C(8)-H(8)	107.8
O(1)-C(7)-C(4)	111.73(19)
O(1)-C(7)-C(8)	104.90(18)
O(1)-C(7)-H(7)	108.8
C(4)-C(7)-C(8)	113.70(19)
C(4)-C(7)-H(7)	108.8
C(8)-C(7)-H(7)	108.8
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(4)-C(5)-H(5)	119.5
C(4)-C(5)-C(6)	121.1(3)
C(6)-C(5)-H(5)	119.5
C(6)-C(1)-Cl(1)	119.0(2)
C(2)-C(1)-Cl(1)	119.7(2)
C(2)-C(1)-C(6)	121.3(3)
C(4)-C(3)-H(3)	119.5
C(4)-C(3)-C(2)	121.0(3)
C(2)-C(3)-H(3)	119.5
C(5)-C(6)-H(6)	120.5
C(1)-C(6)-C(5)	118.9(3)
C(1)-C(6)-H(6)	120.5

C(1)-C(2)-C(3)	119.1(3)
C(1)-C(2)-H(2A)	120.5
C(3)-C(2)-H(2A)	120.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+2

Supplementary Table 10. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 2h. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	71(1)	111(1)	32(1)	26(1)	7(1)	0(1)
O(1)	29(1)	37(1)	32(1)	5(1)	2(1)	-10(1)
O(3)	26(1)	40(1)	57(1)	5(1)	13(1)	3(1)
N(2)	22(1)	31(1)	28(1)	1(1)	5(1)	-2(1)
C(9)	23(1)	34(1)	25(1)	-4(1)	5(1)	-4(1)
C(4)	23(1)	35(1)	25(1)	2(1)	-2(1)	-6(1)
C(8)	20(1)	29(1)	23(1)	0(1)	3(1)	-2(1)
C(7)	23(1)	32(1)	25(1)	1(1)	2(1)	-2(1)
C(10)	31(1)	37(1)	42(1)	0(1)	12(1)	-2(1)
C(5)	37(1)	40(1)	29(1)	2(1)	5(1)	1(1)
C(1)	38(1)	62(2)	29(1)	12(1)	2(1)	-8(1)
C(3)	45(1)	36(1)	36(1)	5(1)	-1(1)	2(1)
C(6)	48(2)	57(2)	29(1)	1(1)	8(1)	-2(1)
C(2)	48(2)	45(2)	44(2)	17(1)	-5(1)	-2(1)

Supplementary Table 11. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for 2h.

	x	y	z	U(eq)
H(1)	3260	744	8924	50
H(8)	5448	2794	9229	29
H(7)	3508	4371	9005	32
H(10A)	6787	9860	8989	54
H(10B)	5838	9719	8101	54
H(10C)	6817	8812	7826	54
H(5)	4353	628	7119	43
H(3)	3326	7041	7482	48
H(6)	4177	1233	5241	54
H(2A)	3190	7704	5609	57
H(2)	4940(30)	7490(80)	8940(30)	57(10)

Supplementary Table 12. Torsion angles [°] for 2h.

Cl(1)-C(1)-C(6)-C(5)	179.4(2)
Cl(1)-C(1)-C(2)-C(3)	179.6(2)
N(2)-C(8)-C(7)-O(1)	167.89(17)
N(2)-C(8)-C(7)-C(4)	45.5(3)
C(9)-N(2)-C(8)-C(8)#1	103.7(2)
C(9)-N(2)-C(8)-C(7)	-132.6(2)
C(4)-C(5)-C(6)-C(1)	1.1(4)
C(4)-C(3)-C(2)-C(1)	0.9(4)
C(8)-N(2)-C(9)-O(3)	-4.2(4)
C(8)-N(2)-C(9)-C(10)	176.8(2)
C(8)#1-C(8)-C(7)-O(1)	-67.97(18)
C(8)#1-C(8)-C(7)-C(4)	169.68(15)
C(7)-C(4)-C(5)-C(6)	-179.7(2)
C(7)-C(4)-C(3)-C(2)	178.7(2)
C(5)-C(4)-C(7)-O(1)	-31.4(3)

C(5)-C(4)-C(7)-C(8)	87.1(3)
C(5)-C(4)-C(3)-C(2)	-2.3(4)
C(3)-C(4)-C(7)-O(1)	147.5(2)
C(3)-C(4)-C(7)-C(8)	-94.0(3)
C(3)-C(4)-C(5)-C(6)	1.3(4)
C(6)-C(1)-C(2)-C(3)	1.7(4)
C(2)-C(1)-C(6)-C(5)	-2.7(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+2

8. References

(1) Wang, Y.; Yang, M.; Lao, C.; Jiang, Z. Potassium-Base-Mediated Autoxidative Diastereoselective Homocoupling of *N* -Acyl-2-Aminoacetophenones. *Org. Lett.* **2022**, *24*, 2625–2629.

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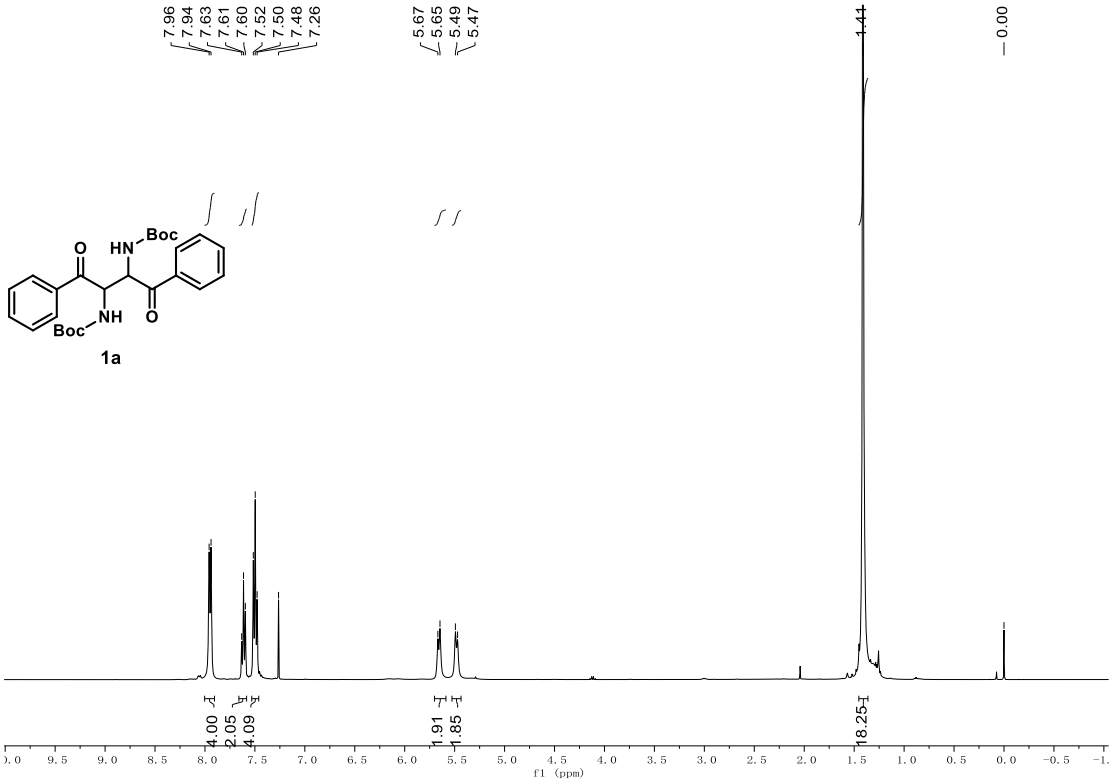
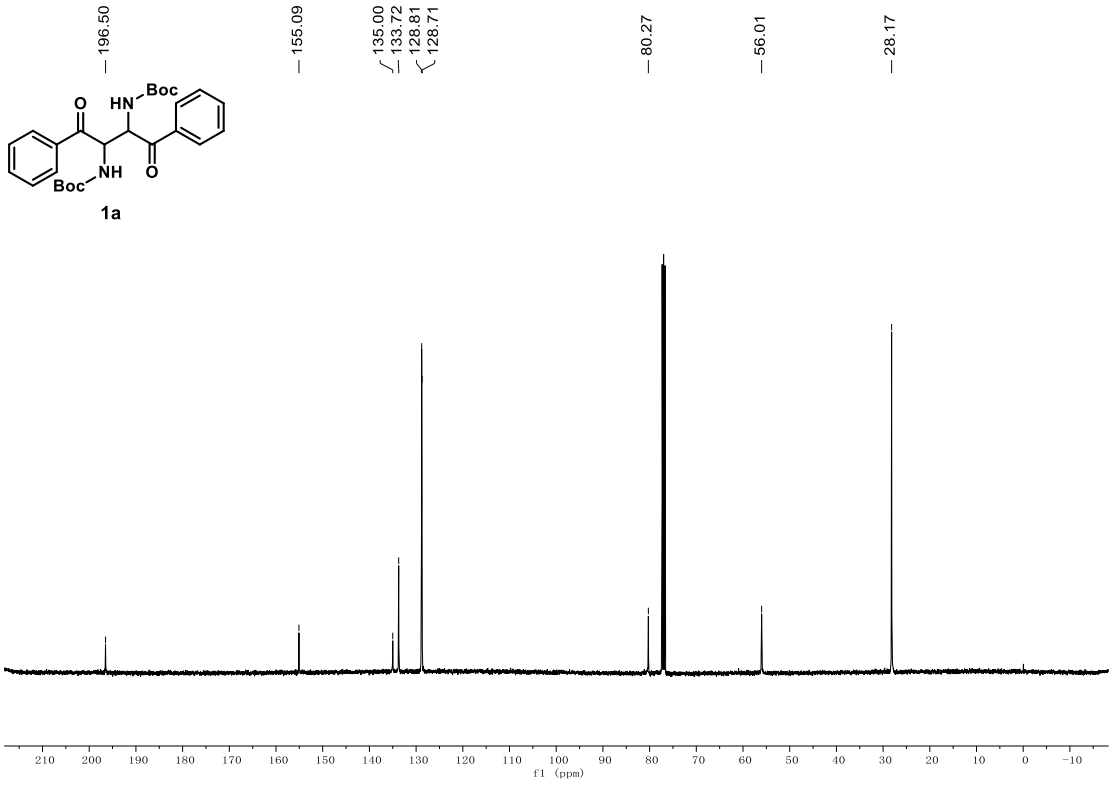
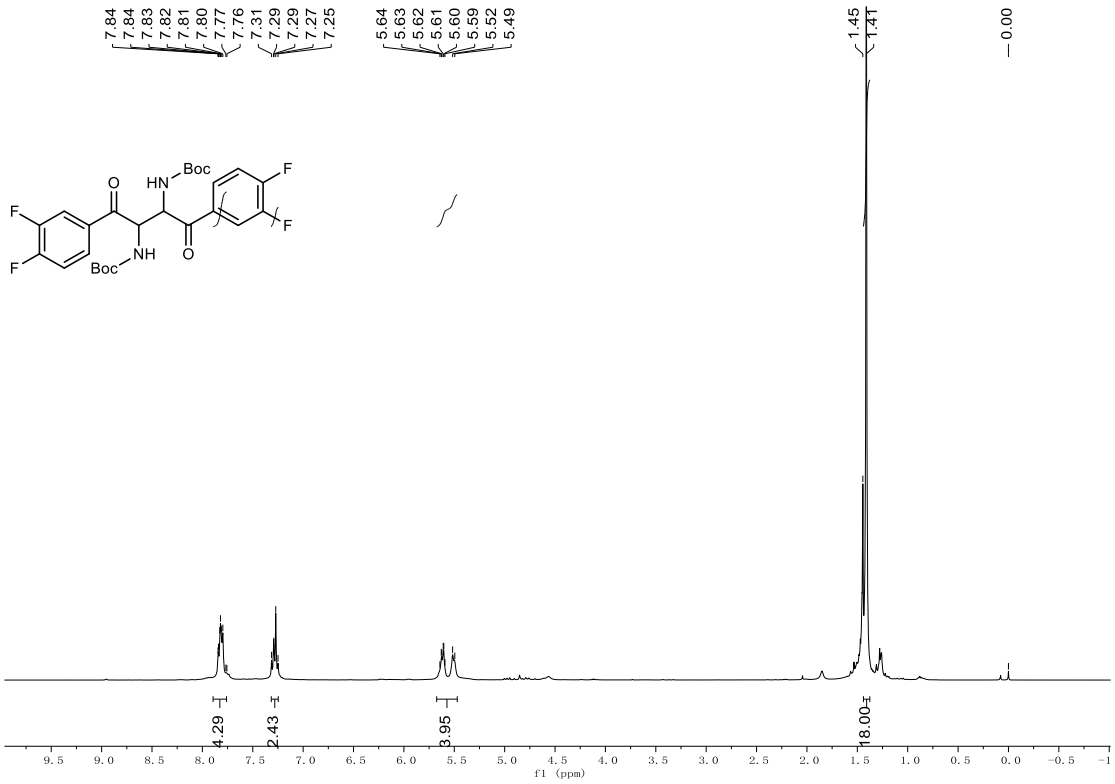
9. NMR Spectra of New Compounds



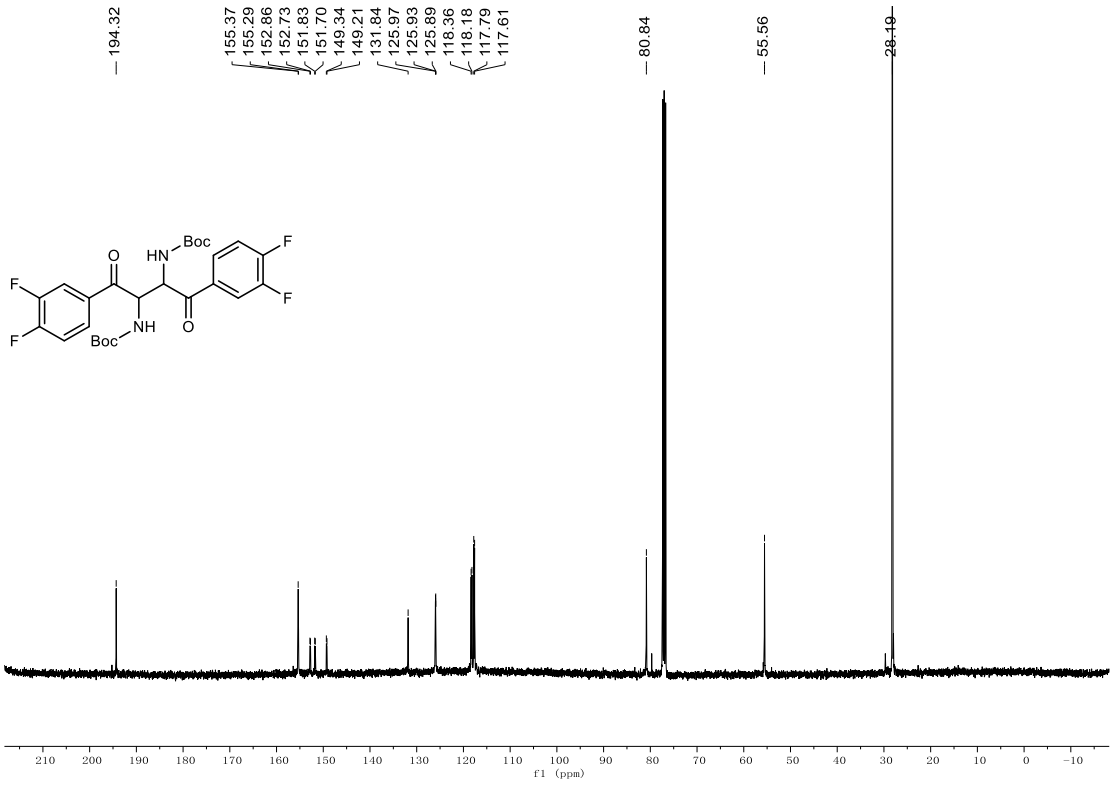
Supplementary Figure 6. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1a



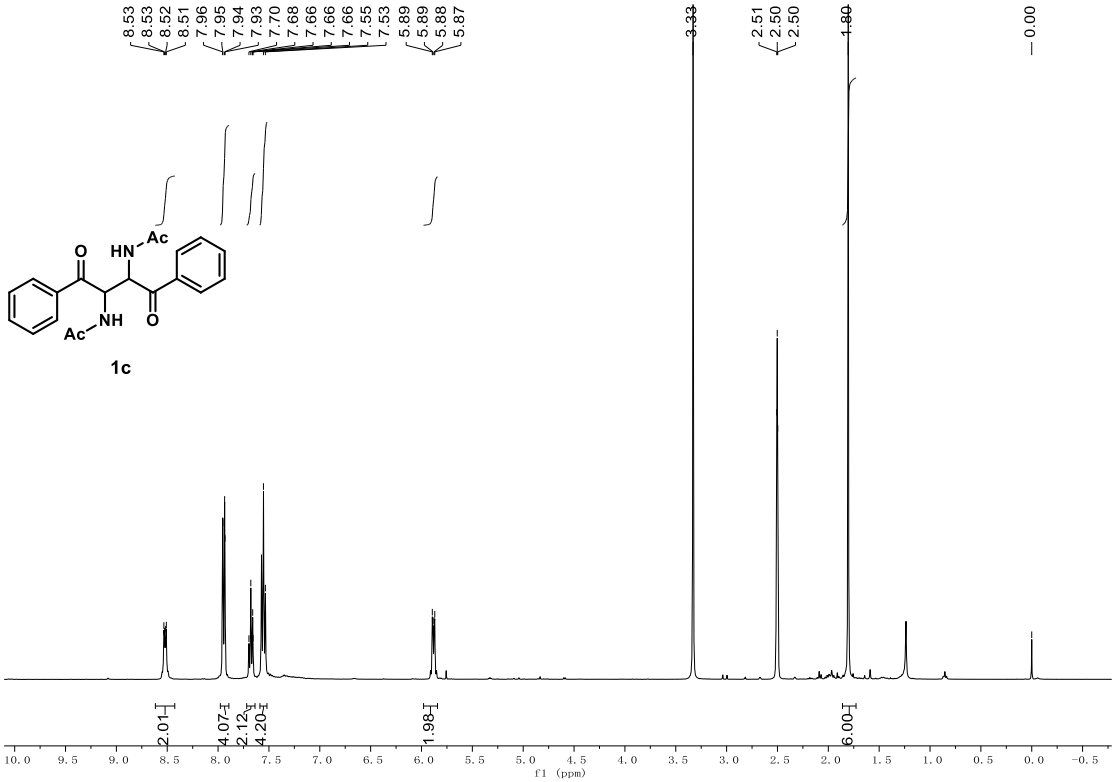
Supplementary Figure 7. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1a



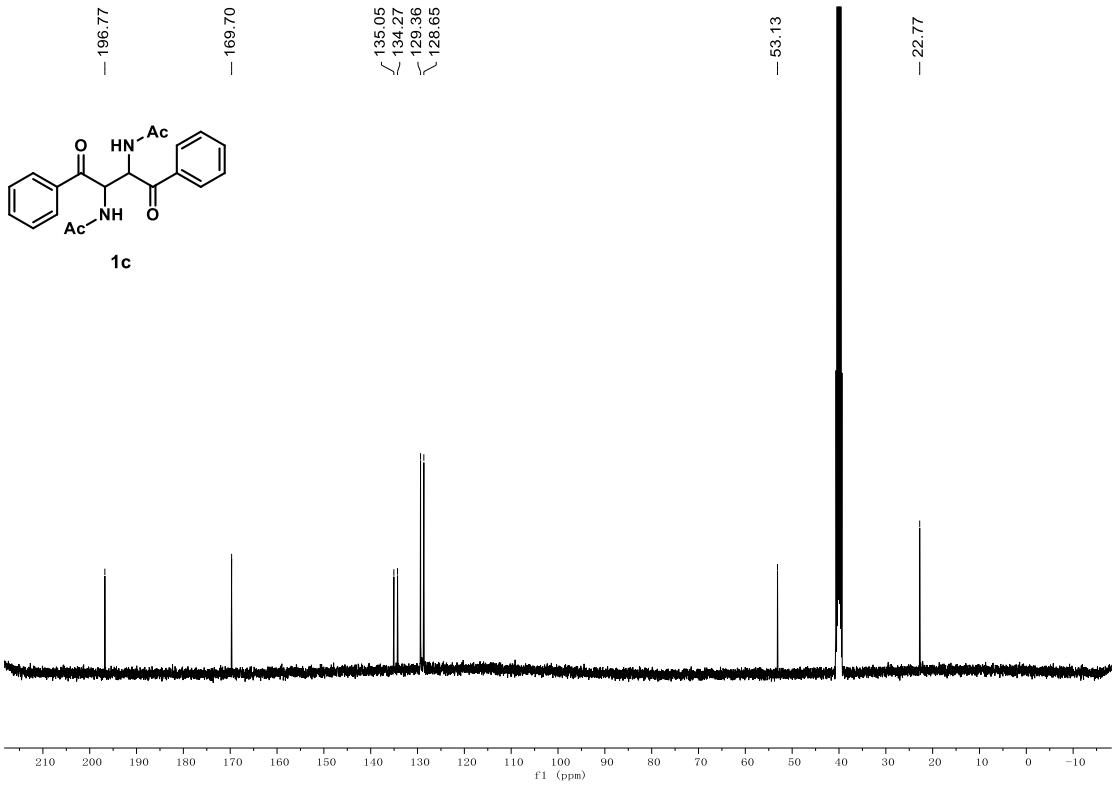
Supplementary Figure 8. ^{1}H NMR (100 MHz, $CDCl_3$) spectra for compound 1b



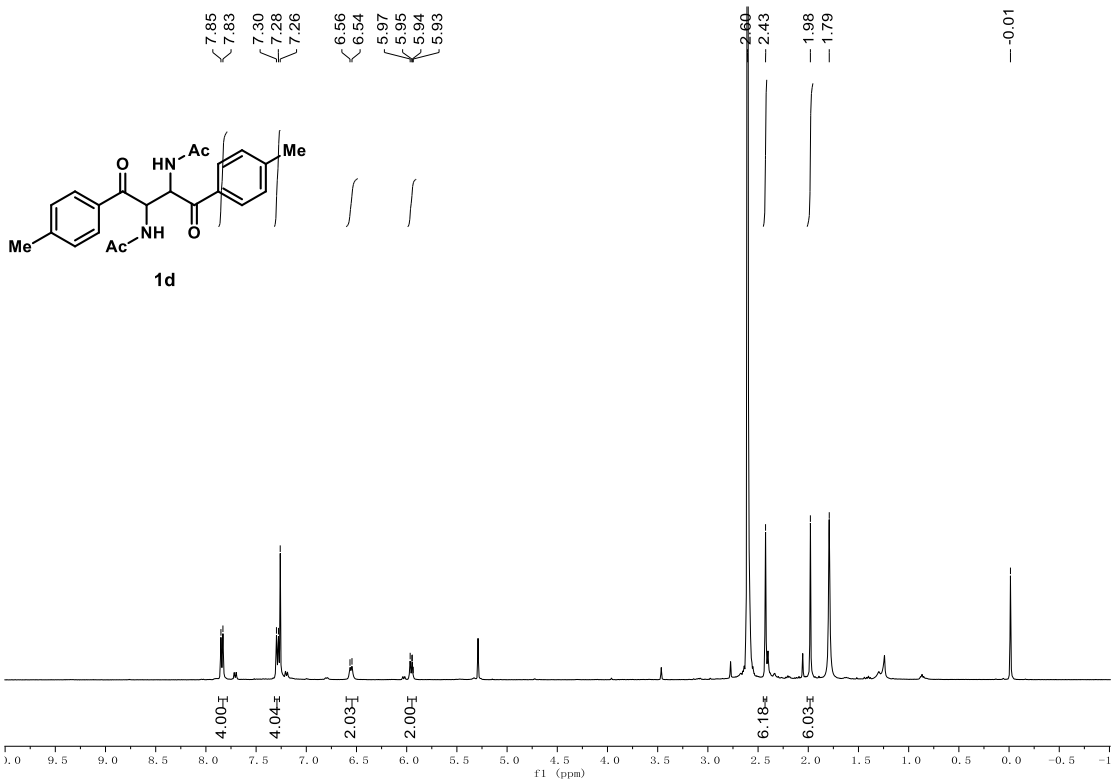
Supplementary Figure 9. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1b



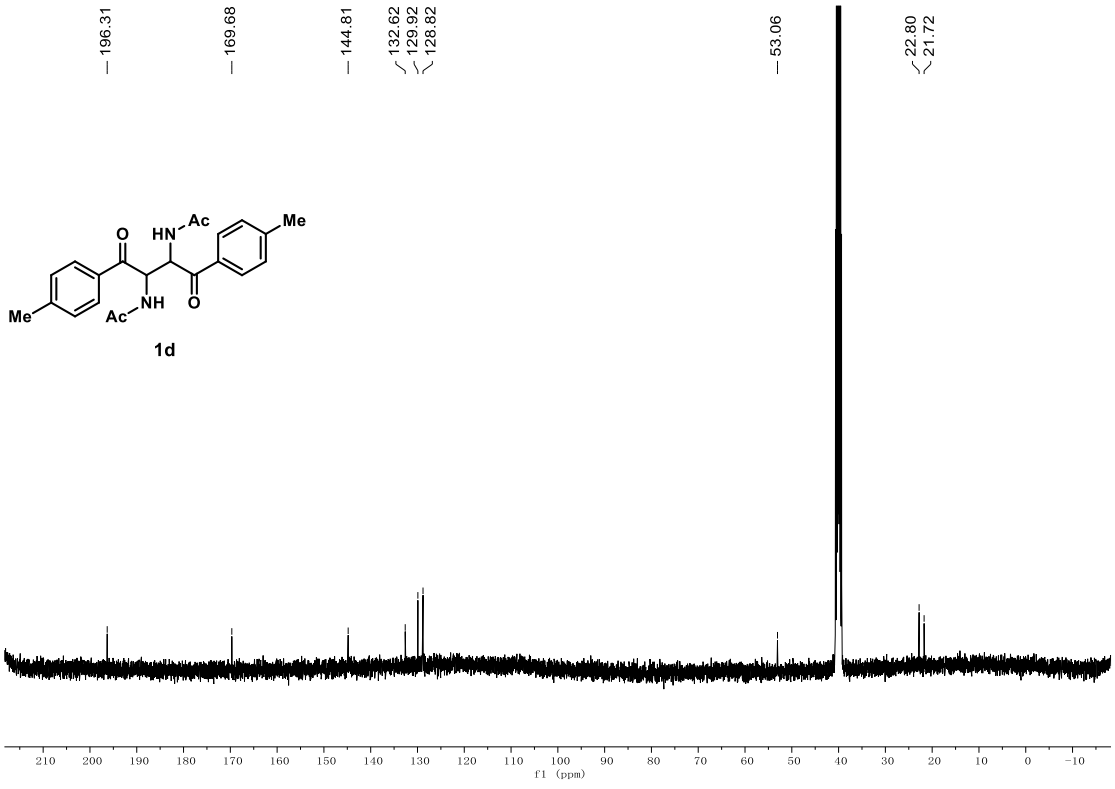
Supplementary Figure 10. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1c



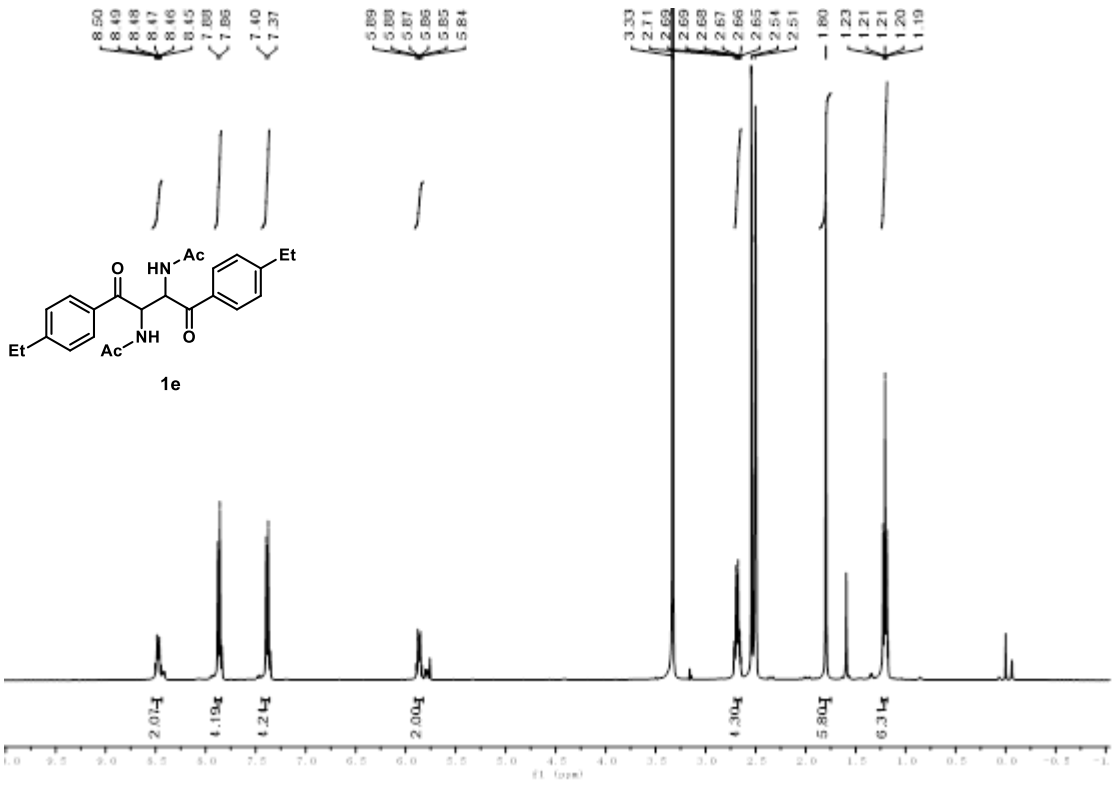
Supplementary Figure 11. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1c



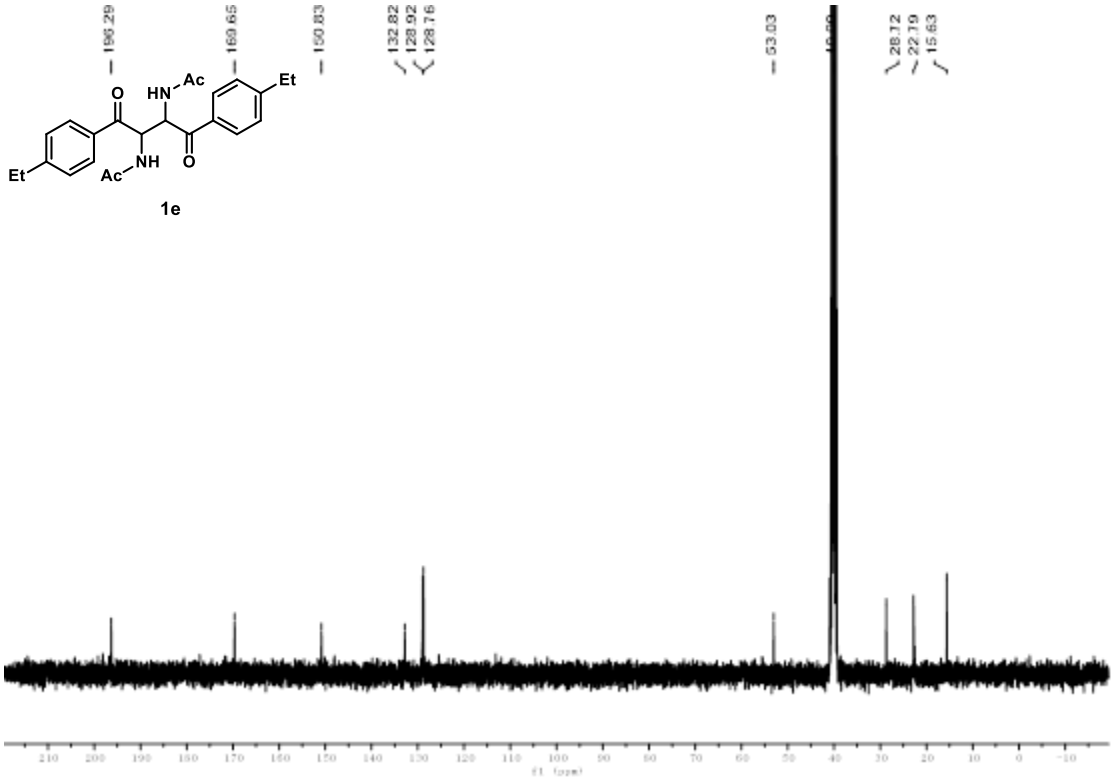
Supplementary Figure 12. ^{1}H NMR (100 MHz, $CDCl_3$) spectra for compound 1d



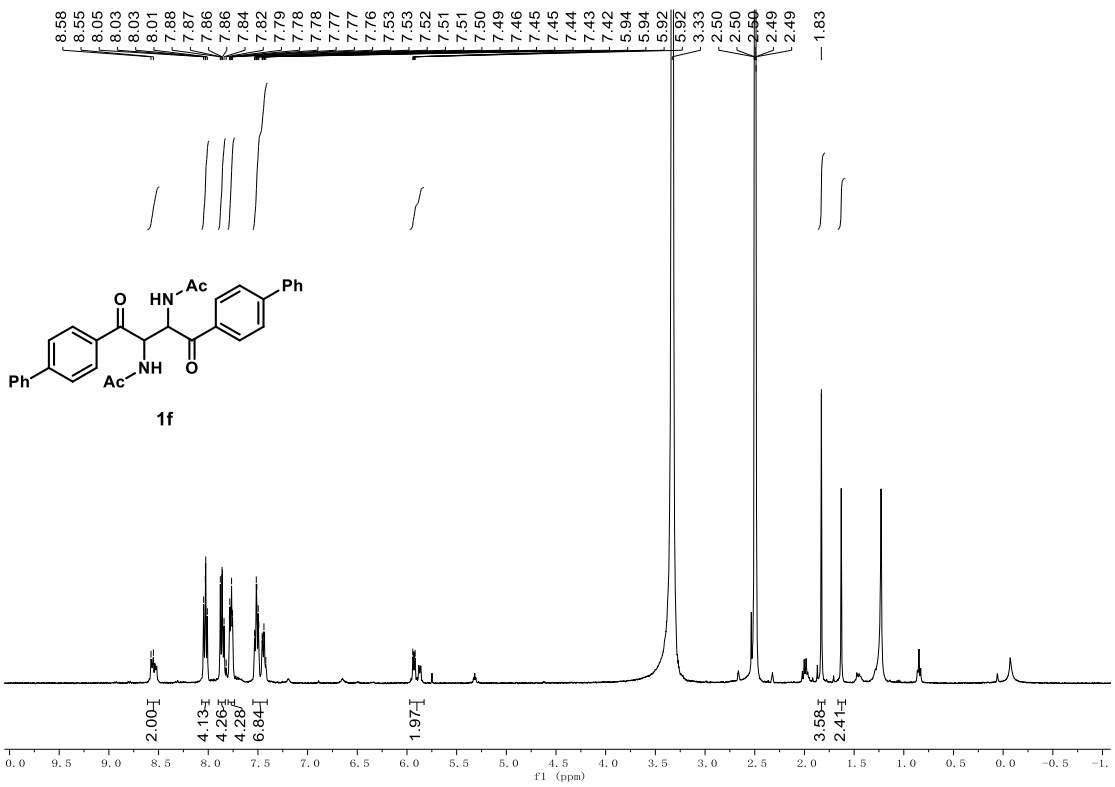
Supplementary Figure 13. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1d



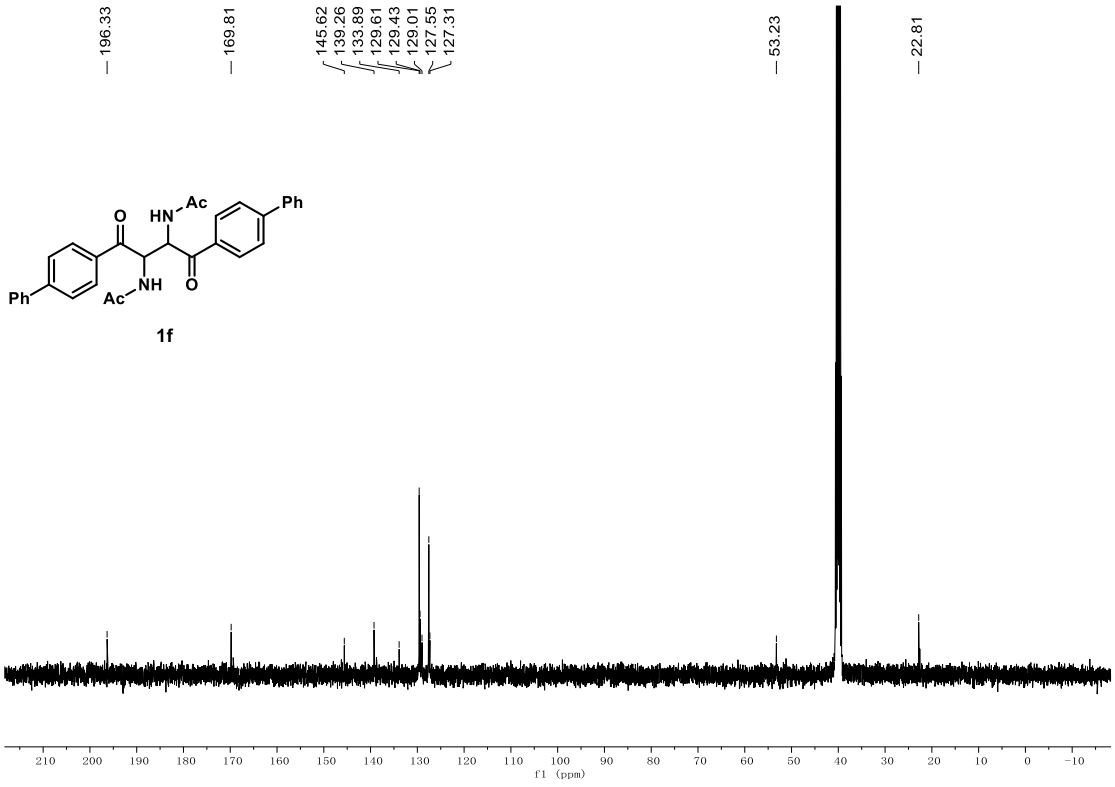
Supplementary Figure 14. ^{1}H NMR (100 MHz, $CDCl_3$) spectra for compound 1e



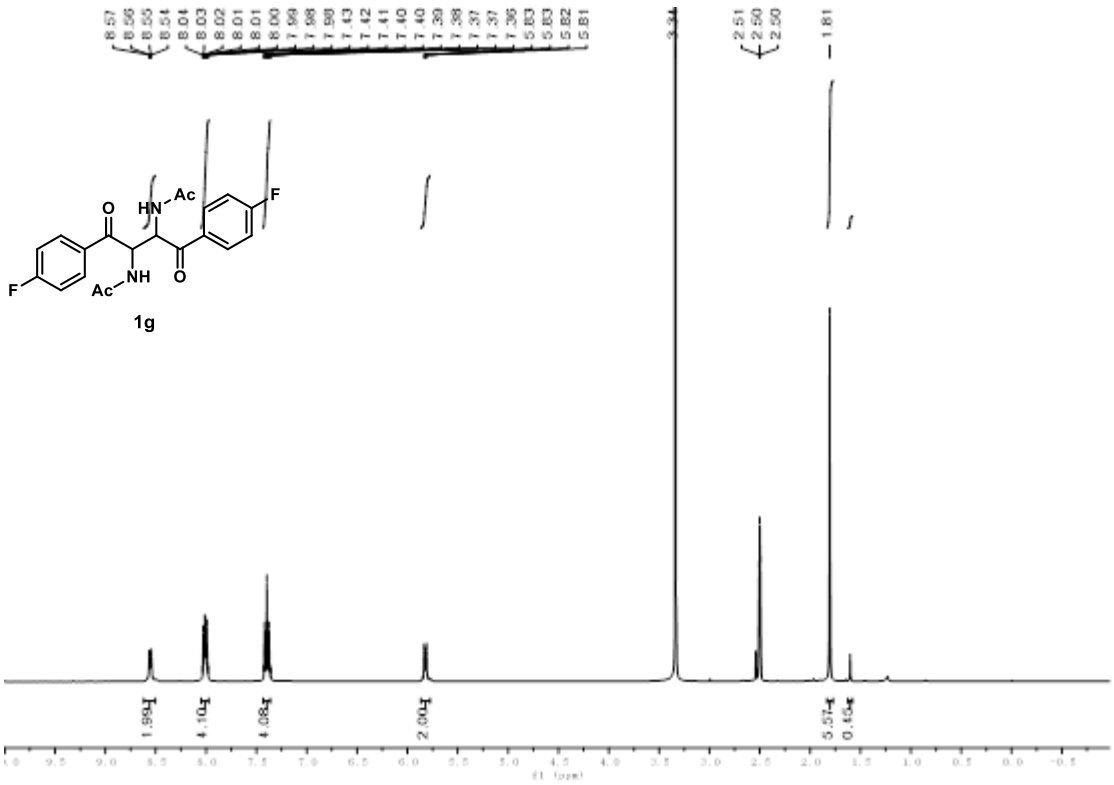
Supplementary Figure 15. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1e



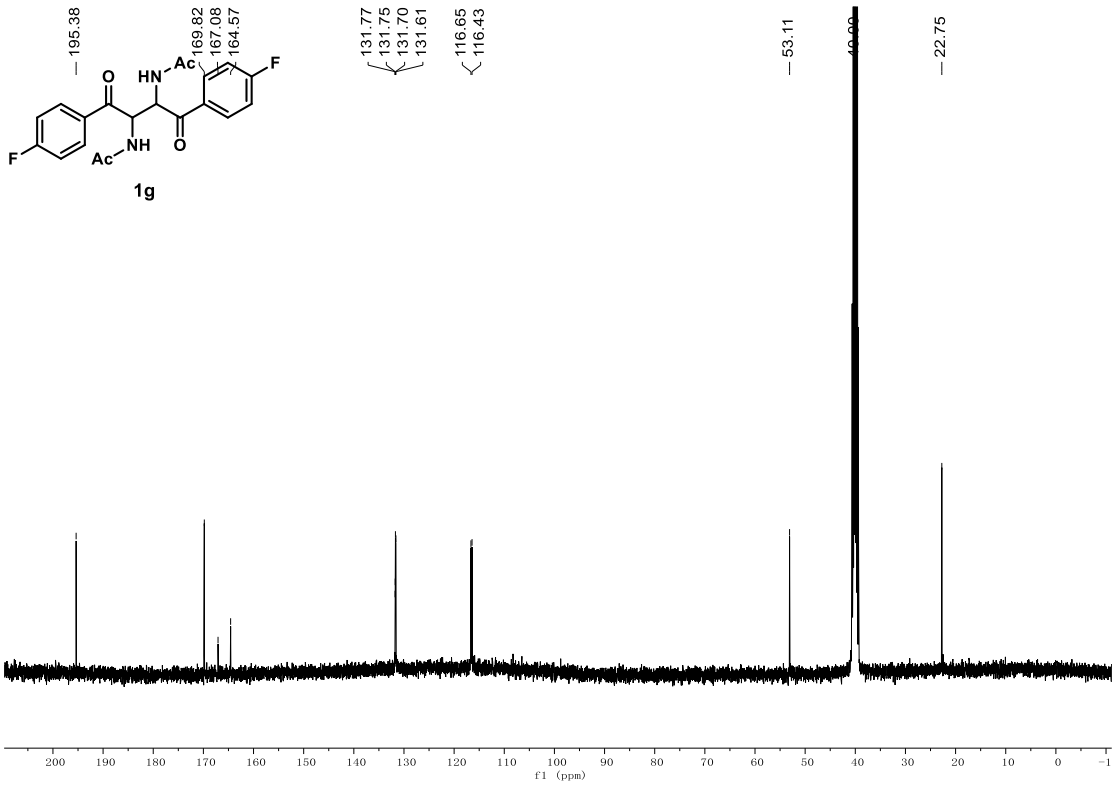
Supplementary Figure 16. ^{1}H NMR (100 MHz, $CDCl_3$) spectra for compound 1f



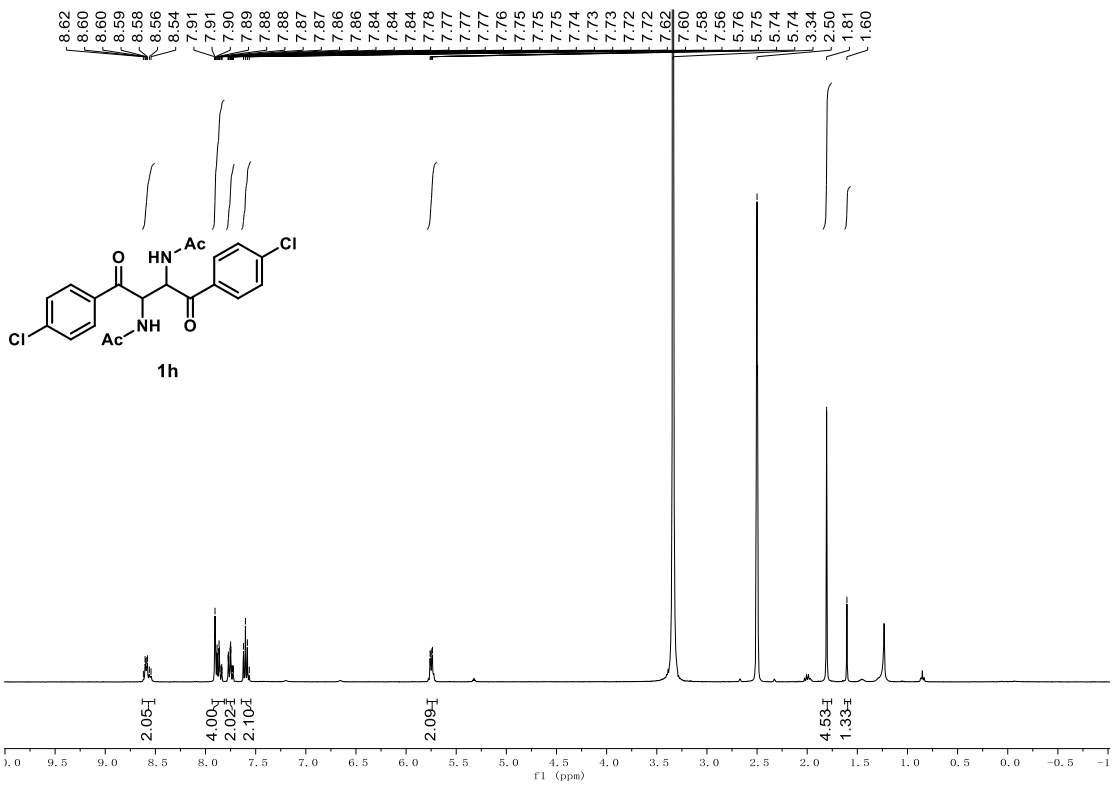
Supplementary Figure 17. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1f



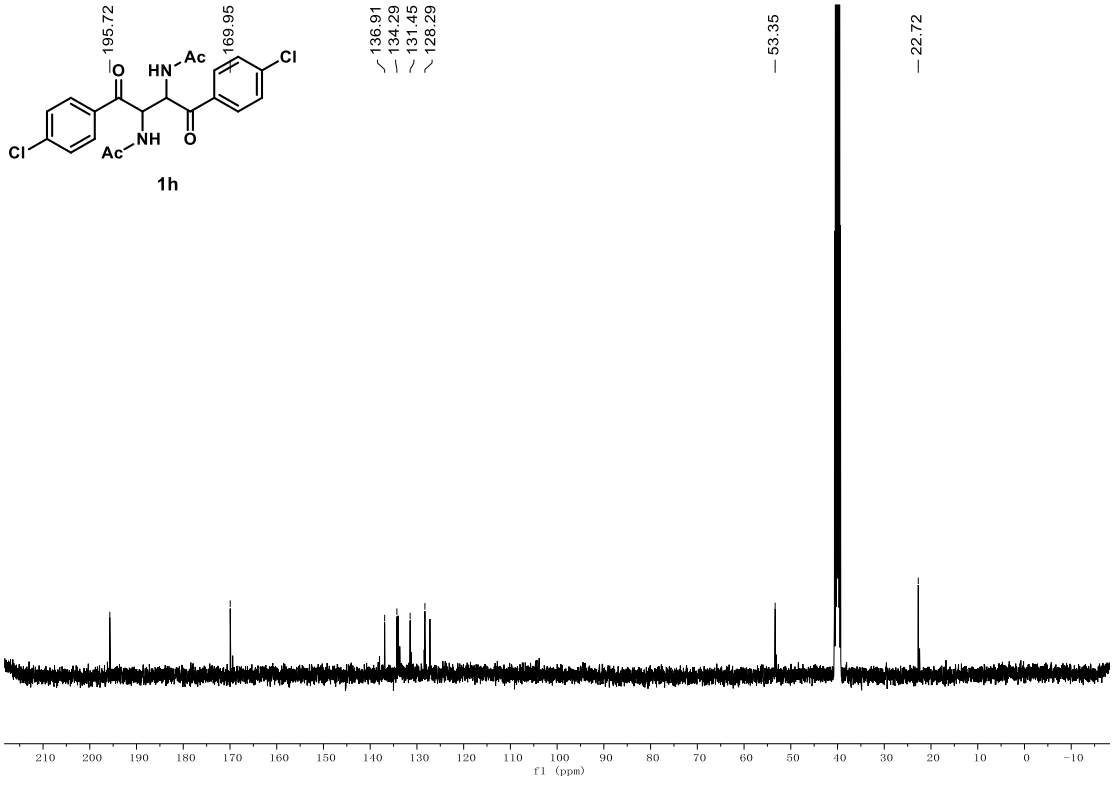
Supplementary Figure 18. ^{1}H NMR (100 MHz, $CDCl_3$) spectra for compound 1g



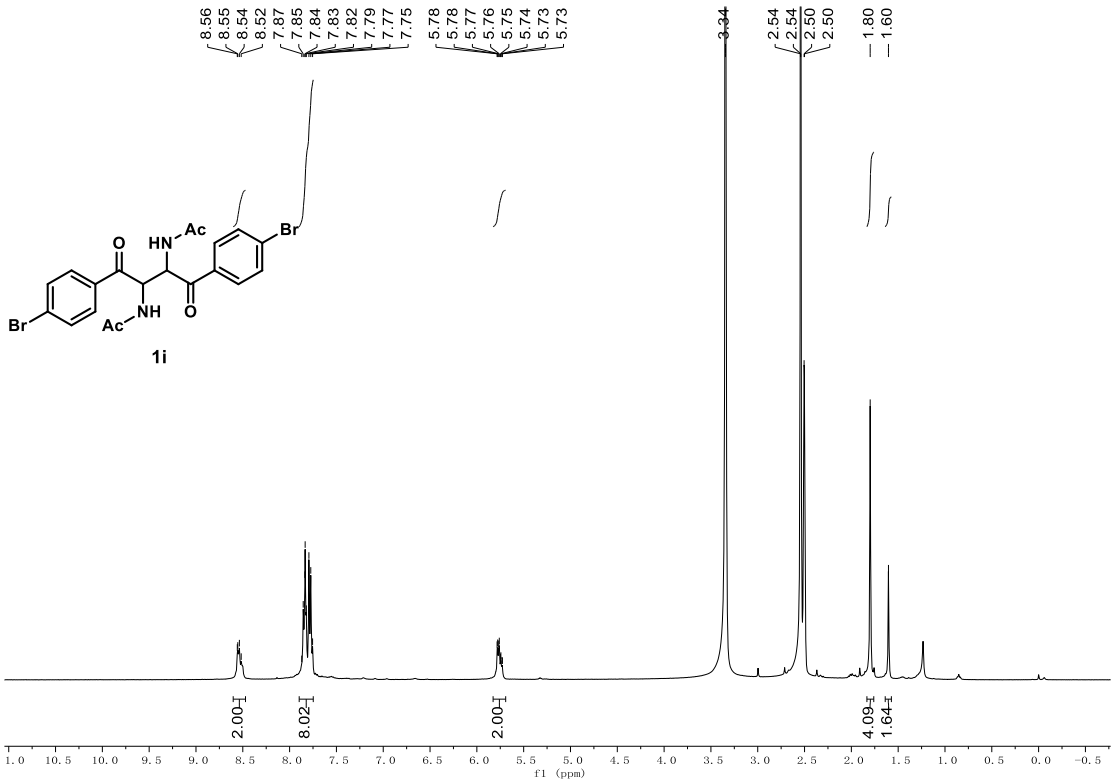
Supplementary Figure 19. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1g



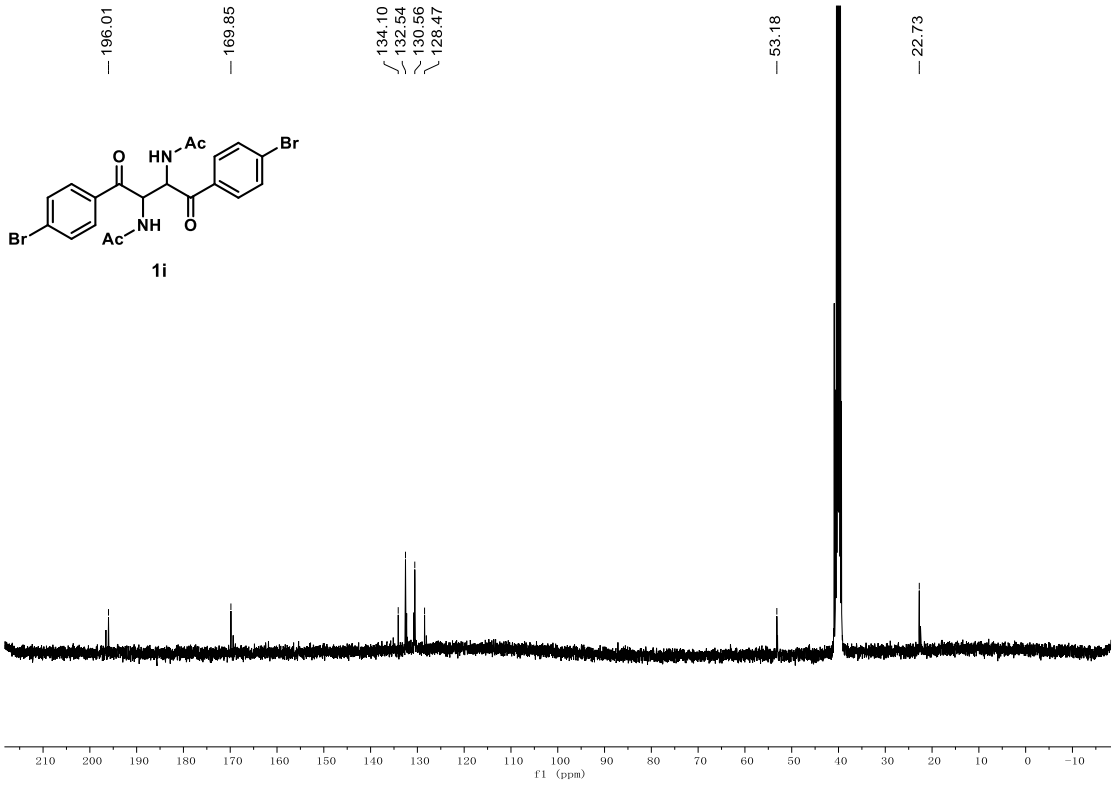
Supplementary Figure 20. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1h



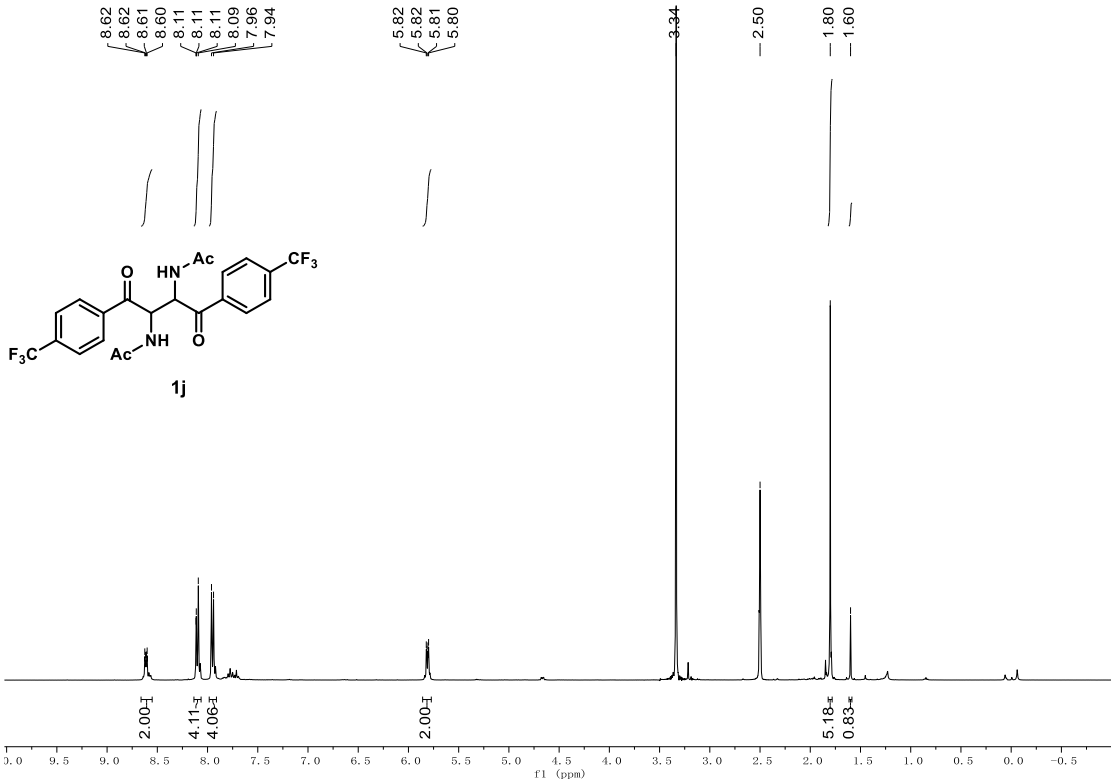
Supplementary Figure 21. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1h



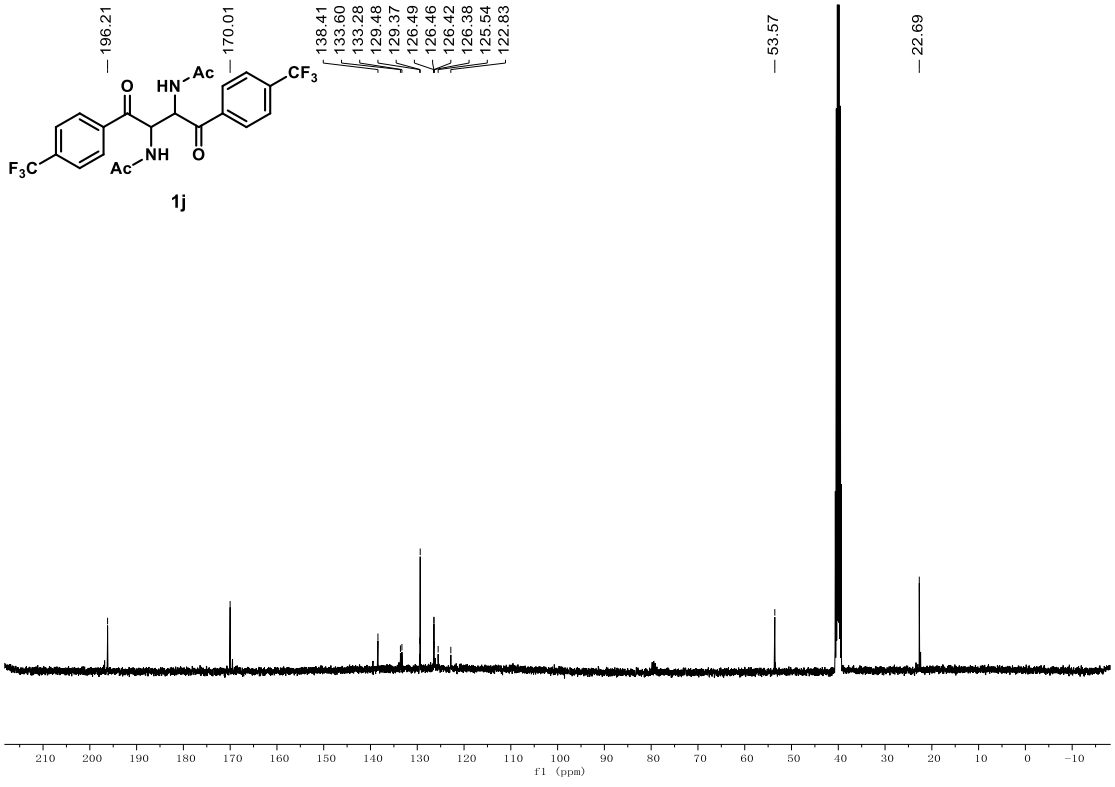
Supplementary Figure 22. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1i



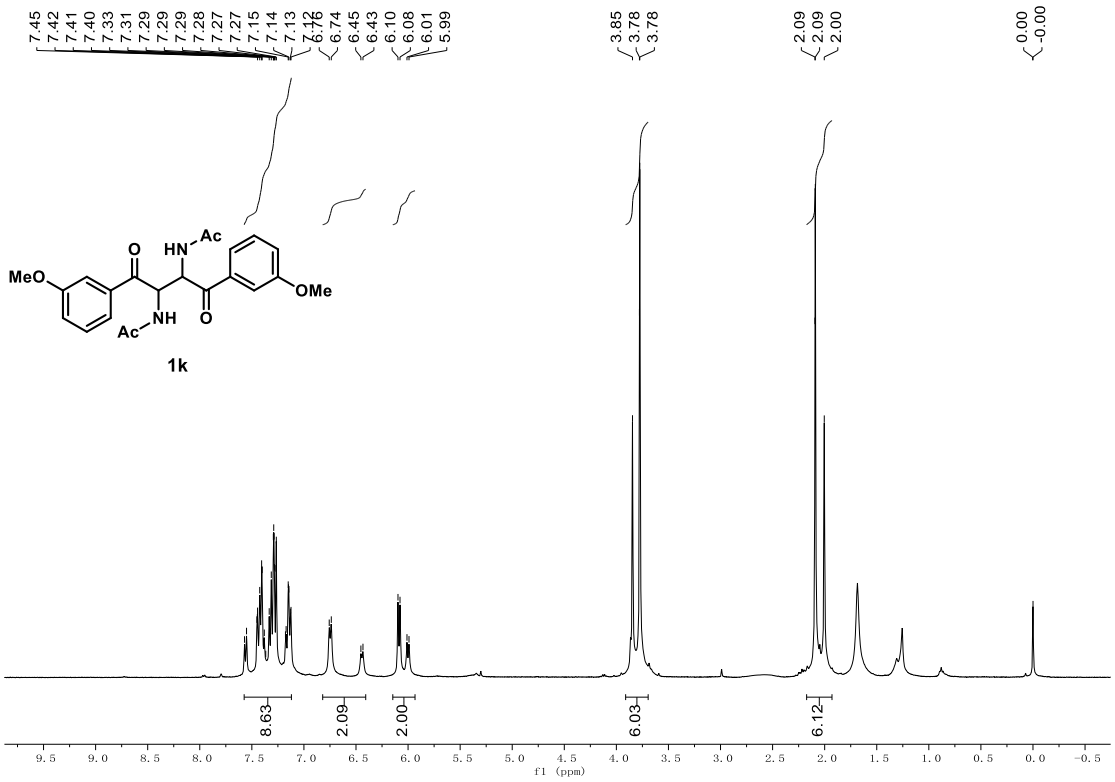
Supplementary Figure 23. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1i



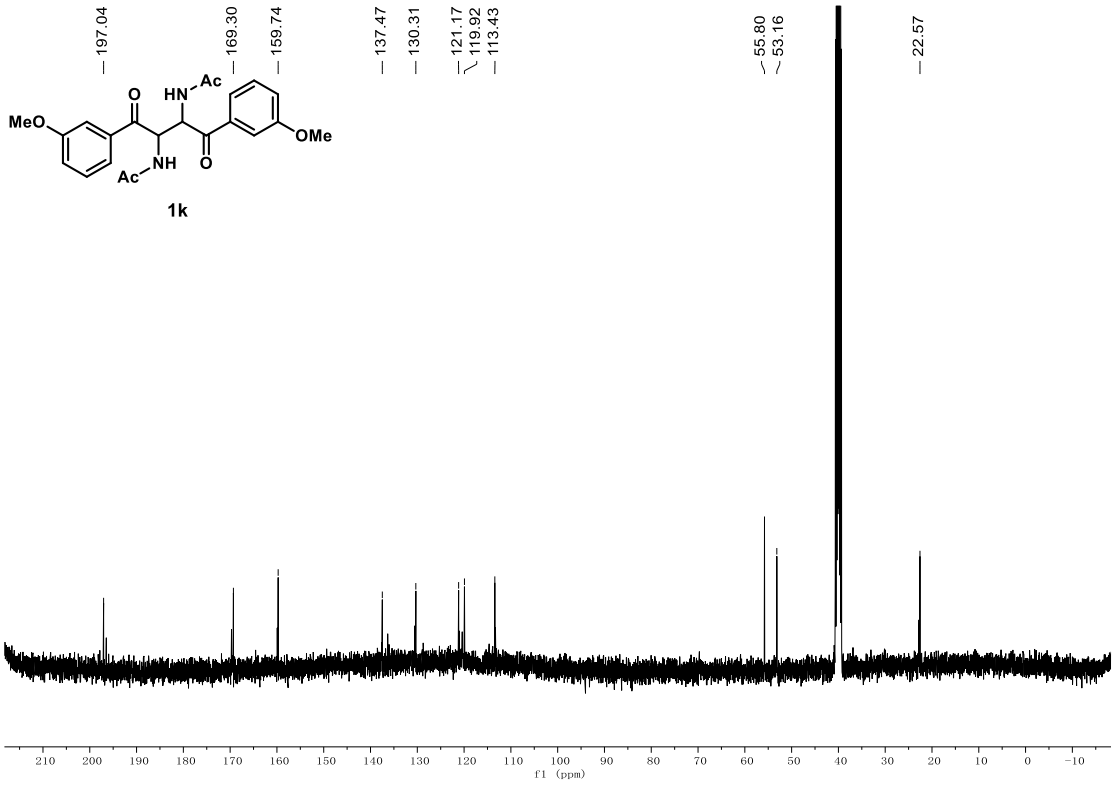
Supplementary Figure 24. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1j



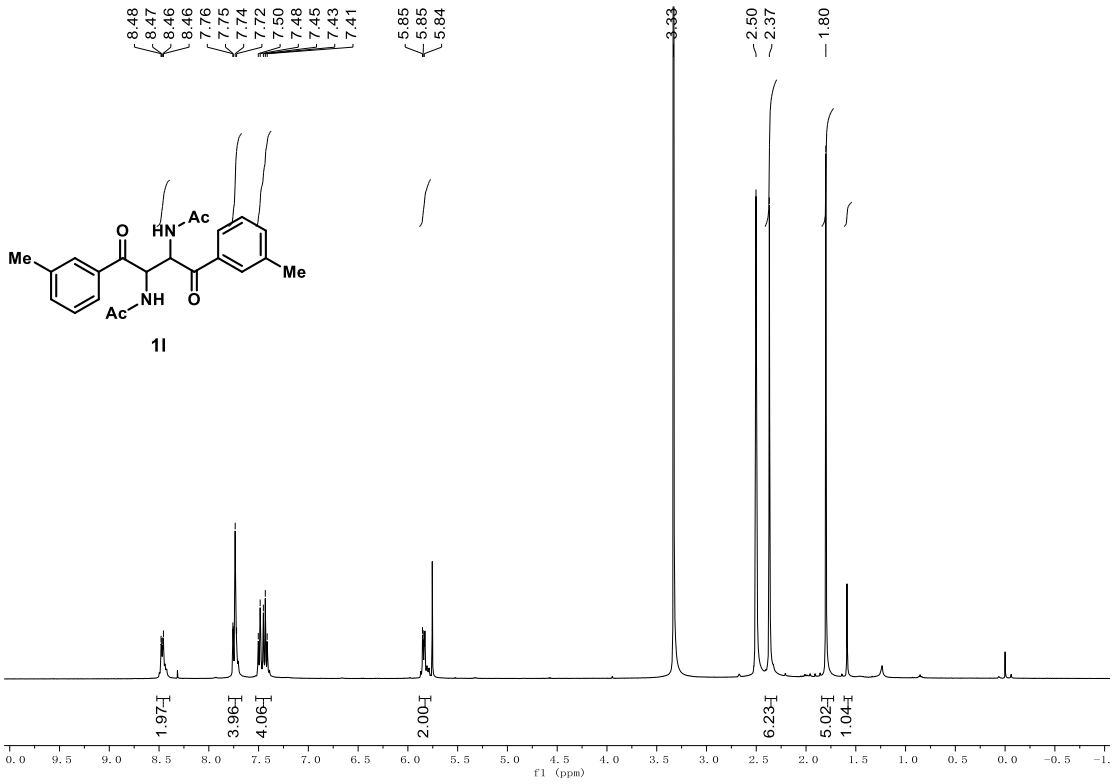
Supplementary Figure 25. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1j



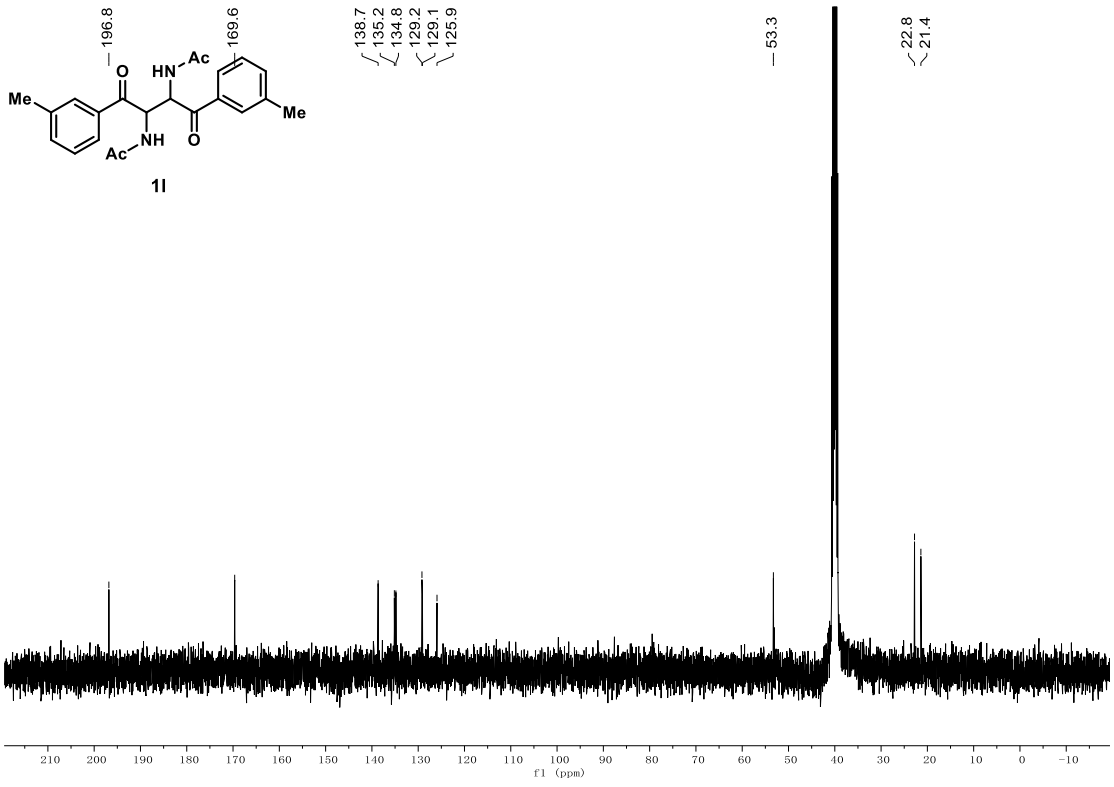
Supplementary Figure 26. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1k



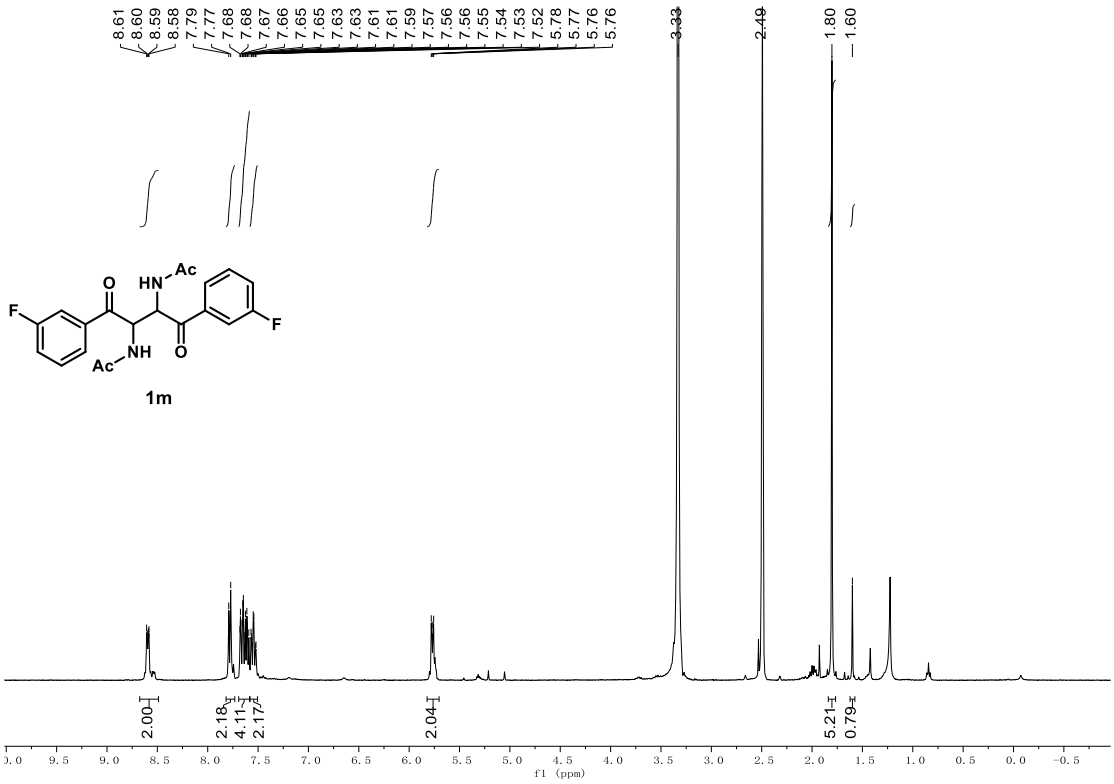
Supplementary Figure 27. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1k



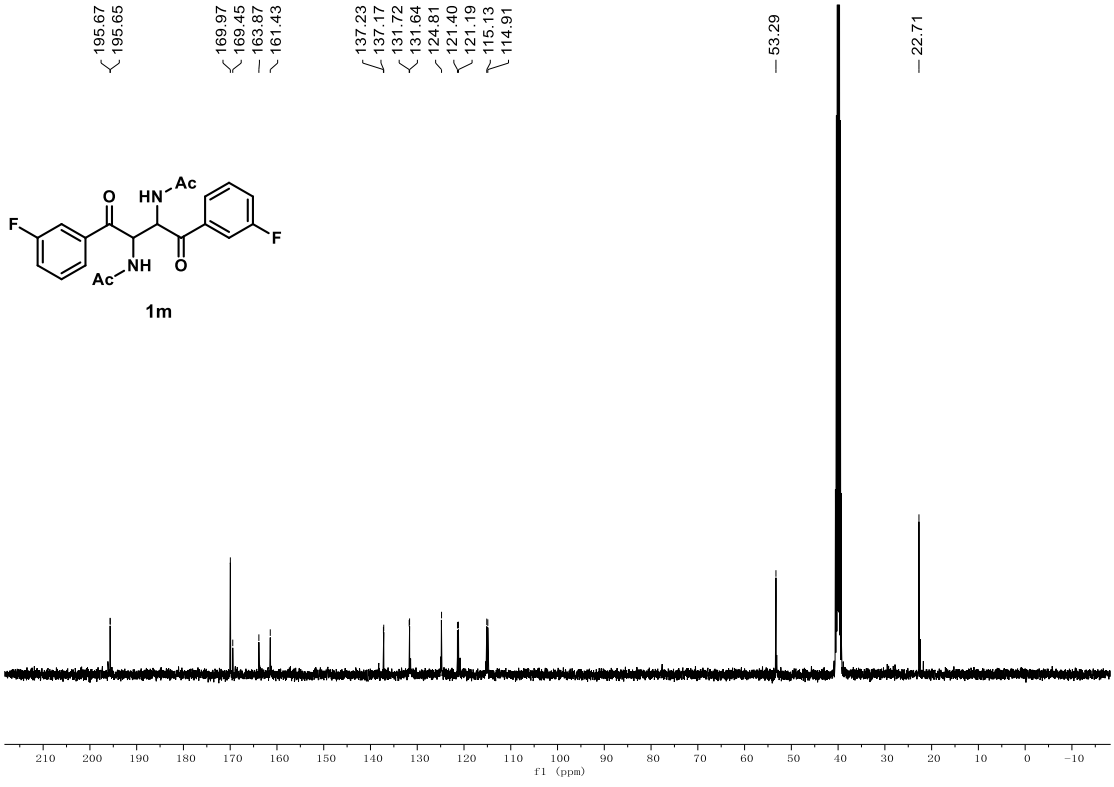
Supplementary Figure 28. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1l



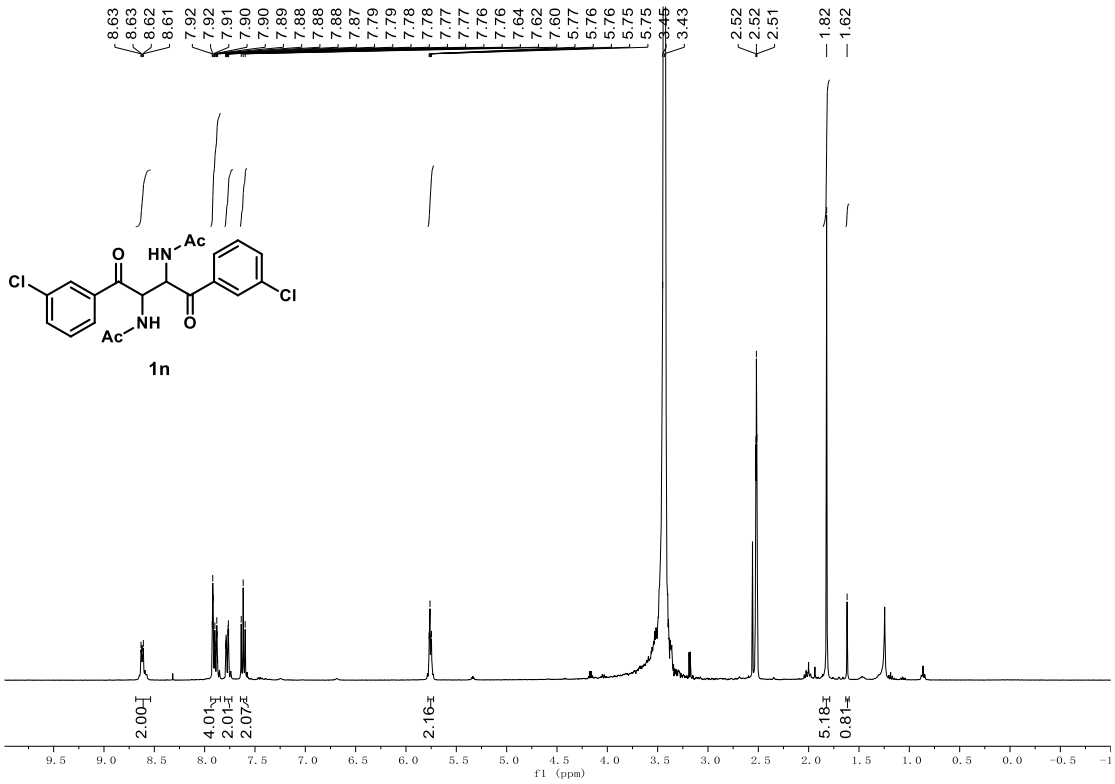
Supplementary Figure 29. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1l



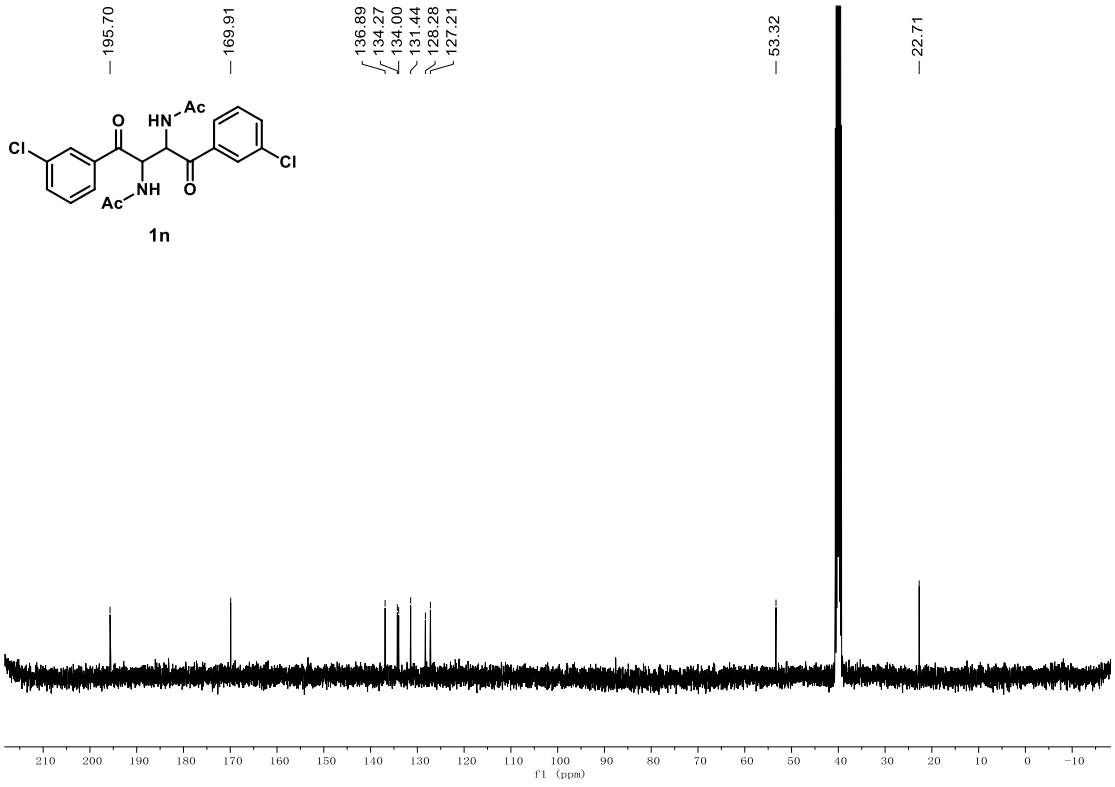
Supplementary Figure 30. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1m



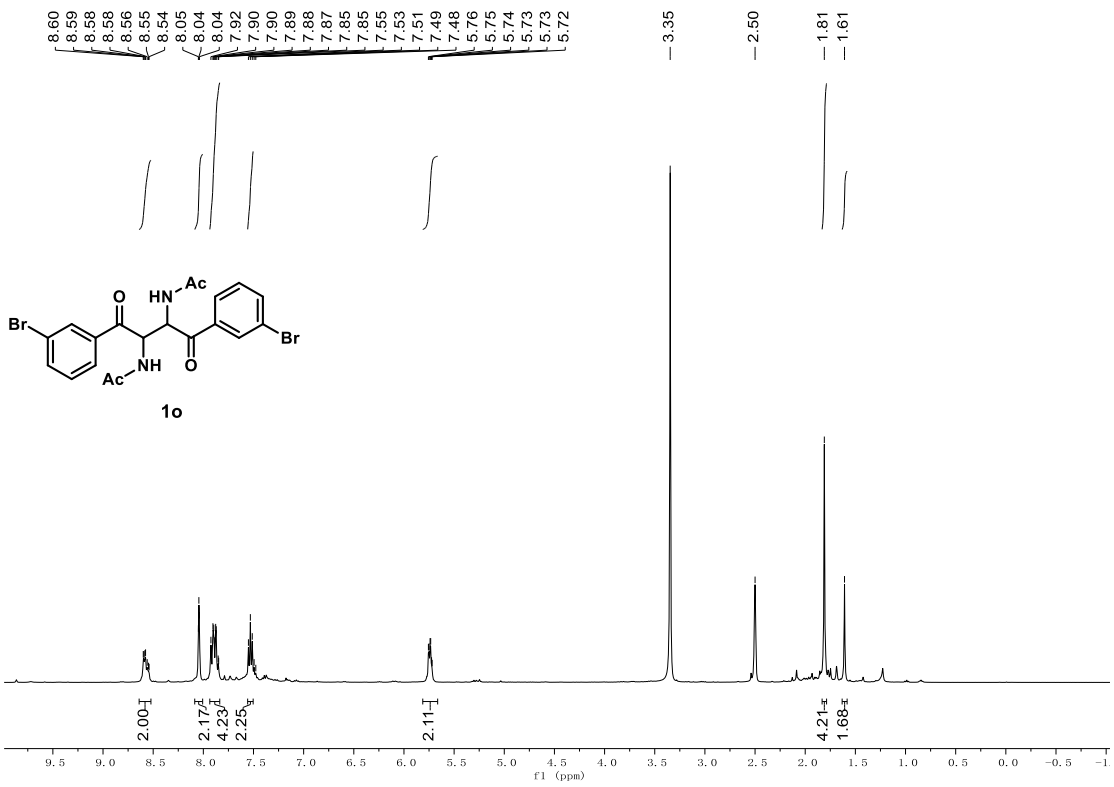
Supplementary Figure 31. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1m



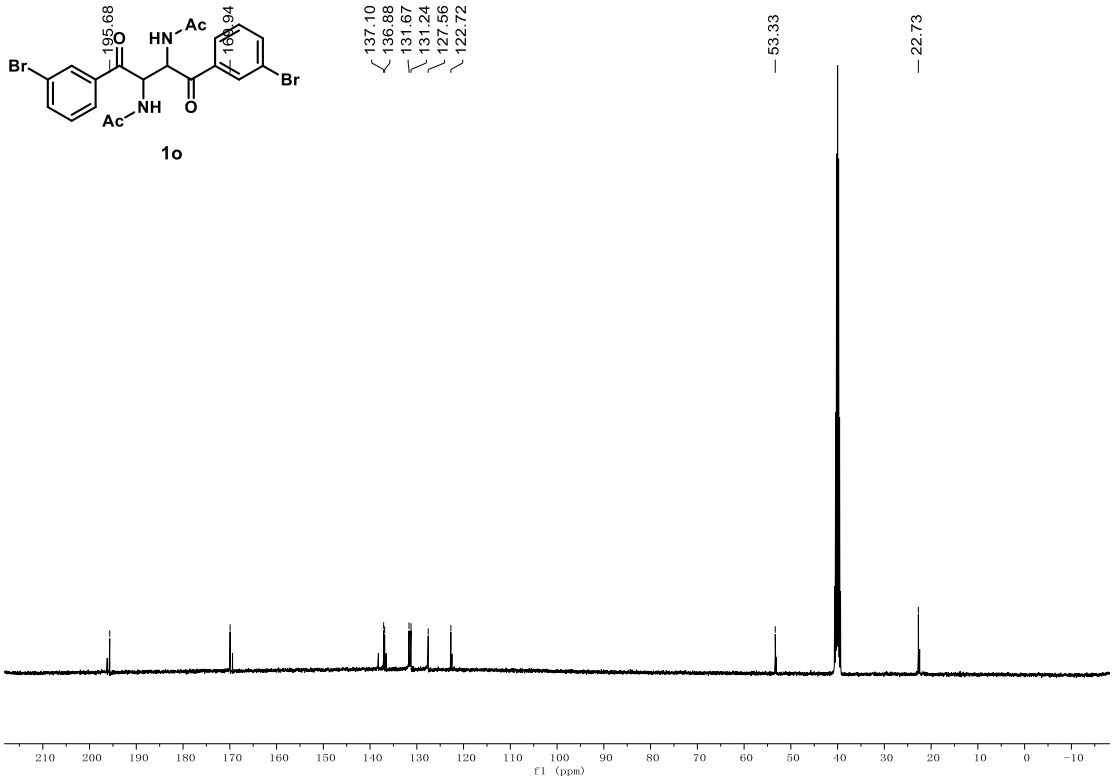
Supplementary Figure 32. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1n



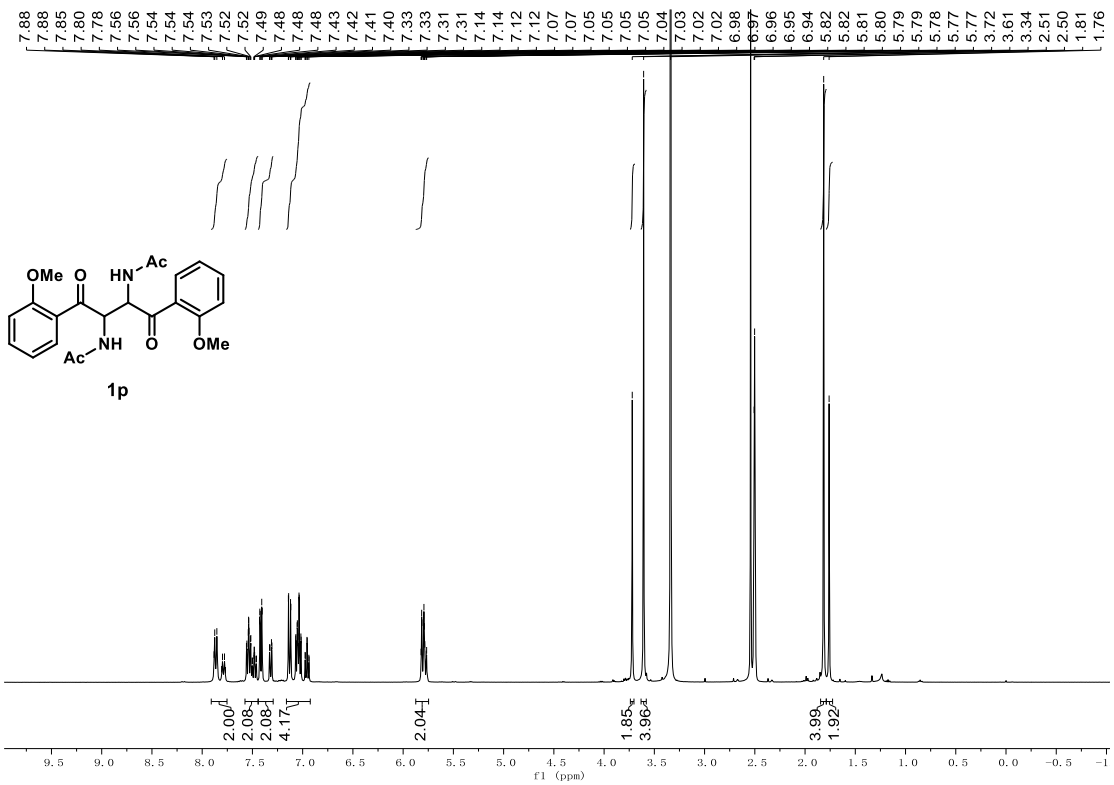
Supplementary Figure 33. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1n



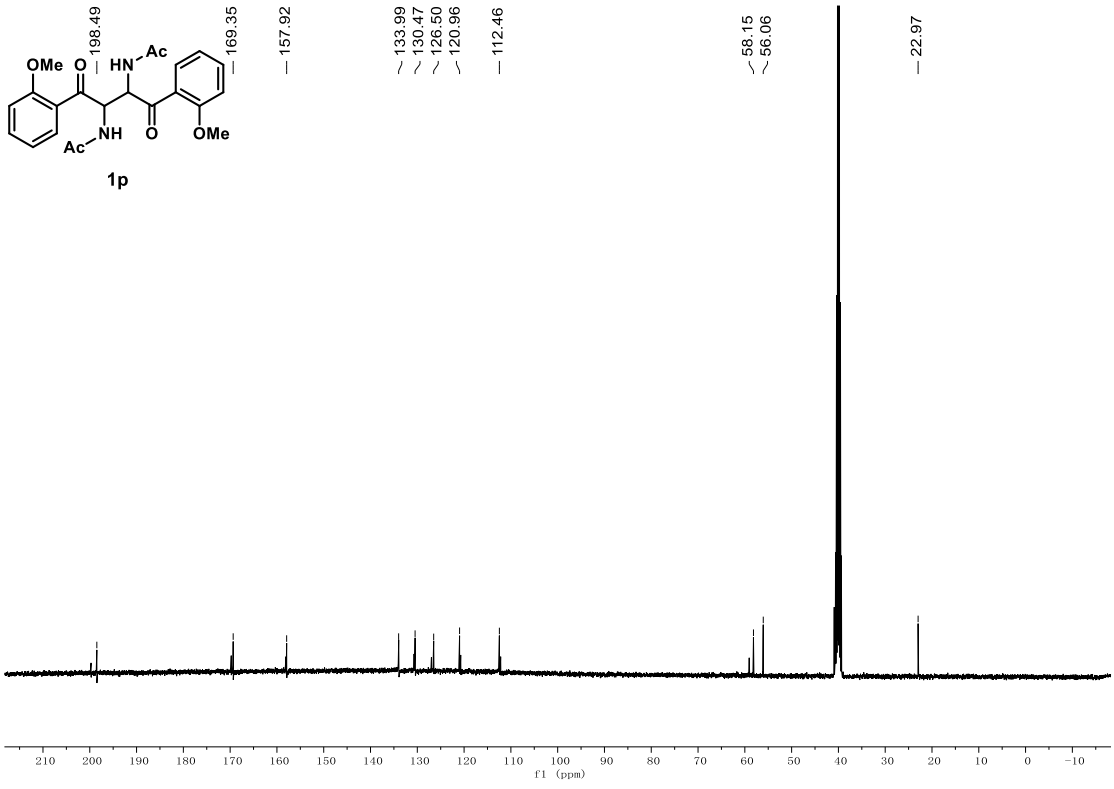
Supplementary Figure 34. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1o



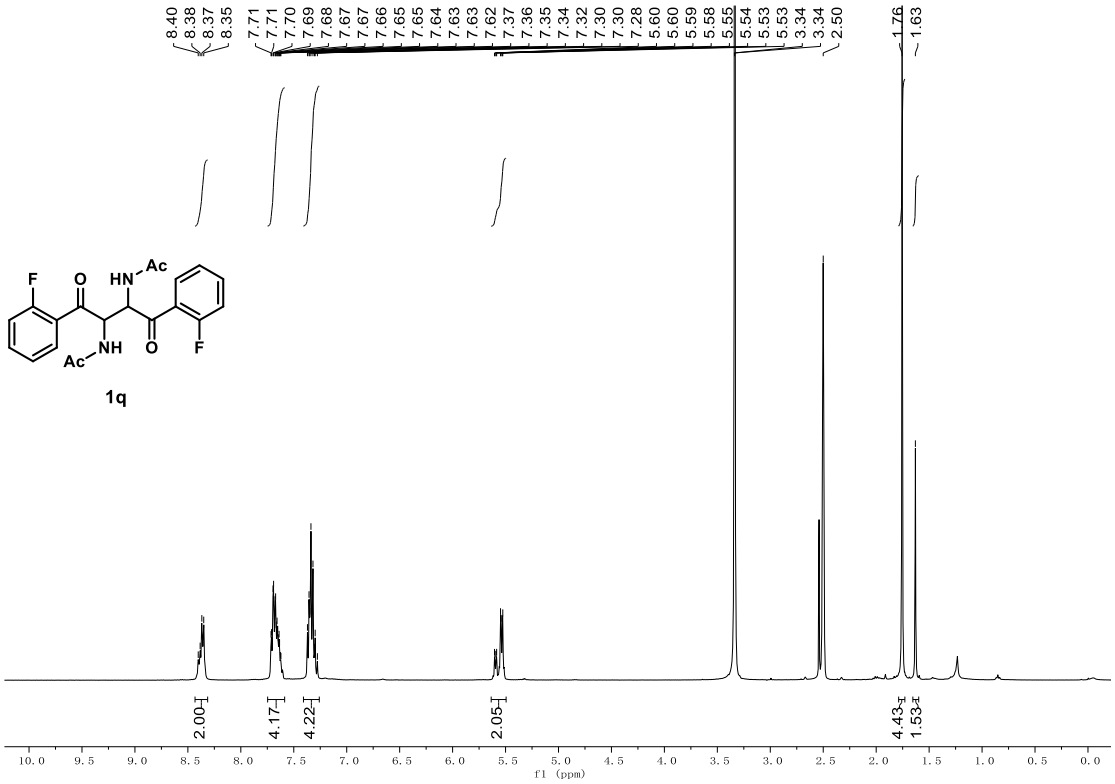
Supplementary Figure 35. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1o



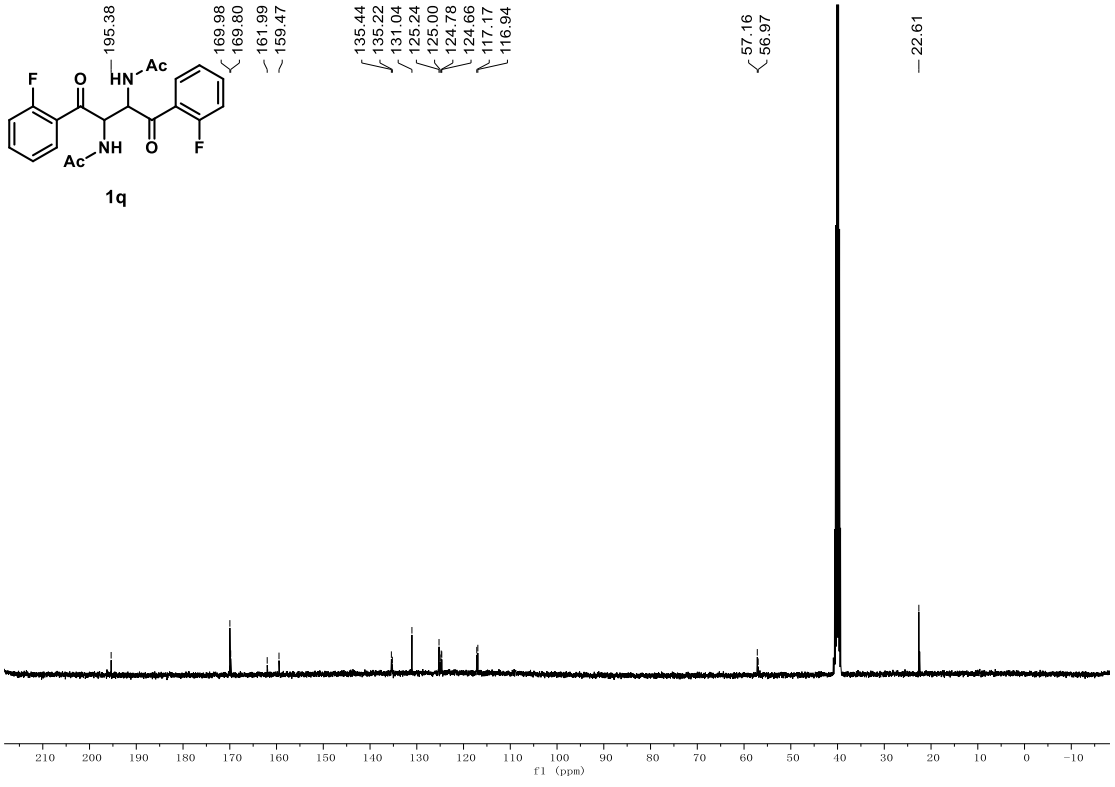
Supplementary Figure 36. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1p



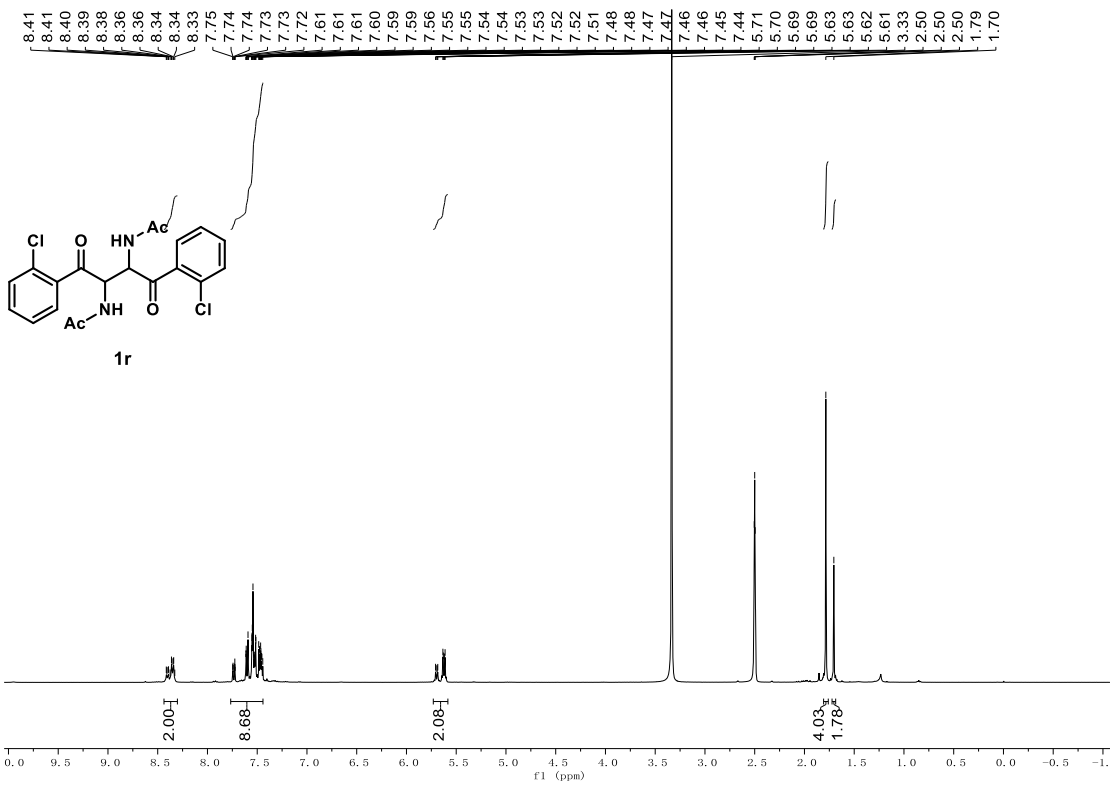
Supplementary Figure 37. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1p



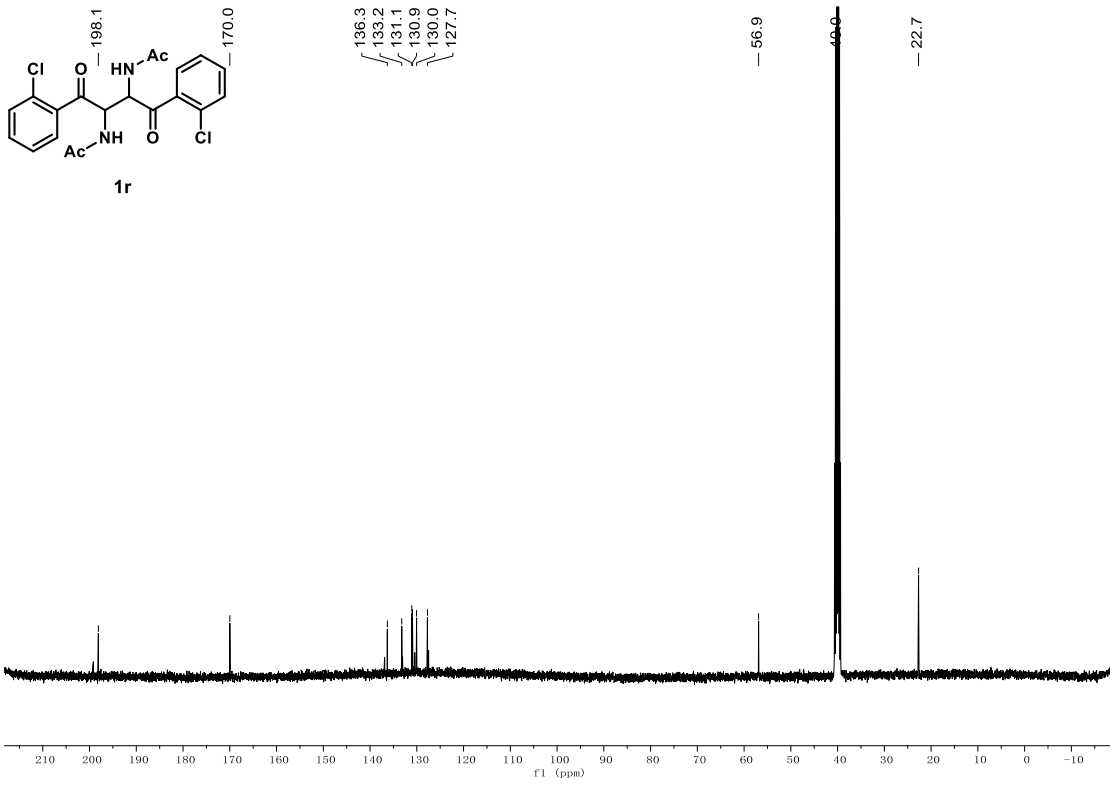
Supplementary Figure 38. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1q



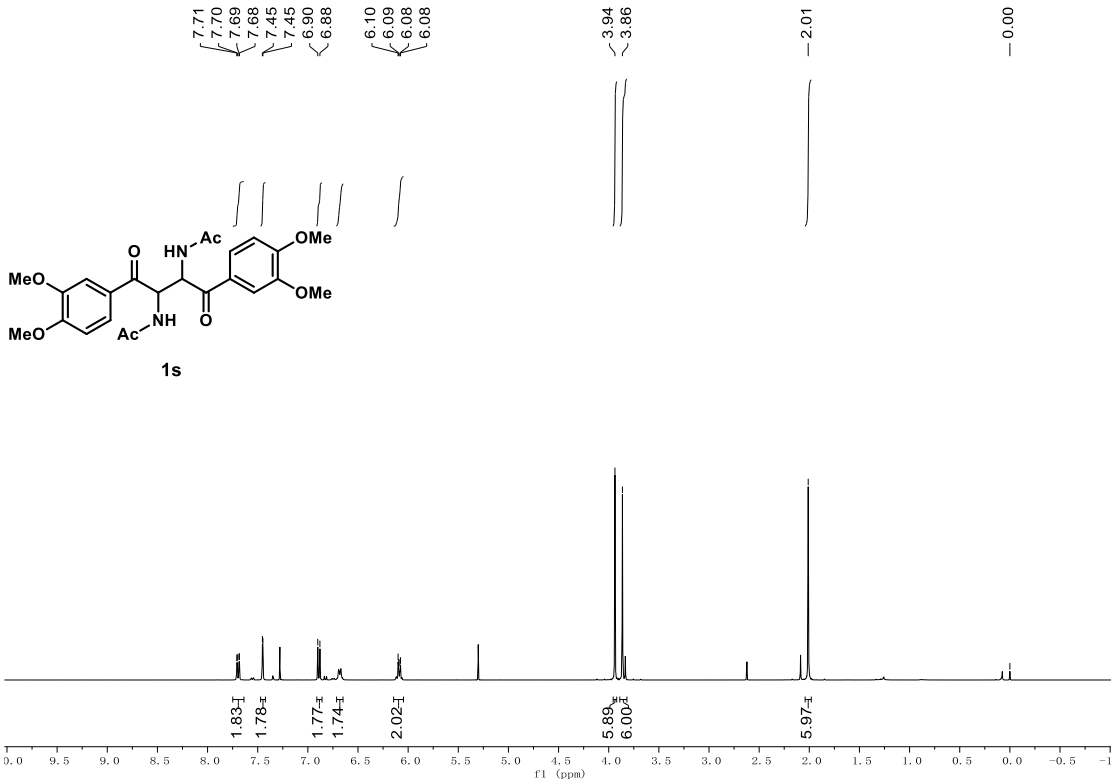
Supplementary Figure 39. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1q



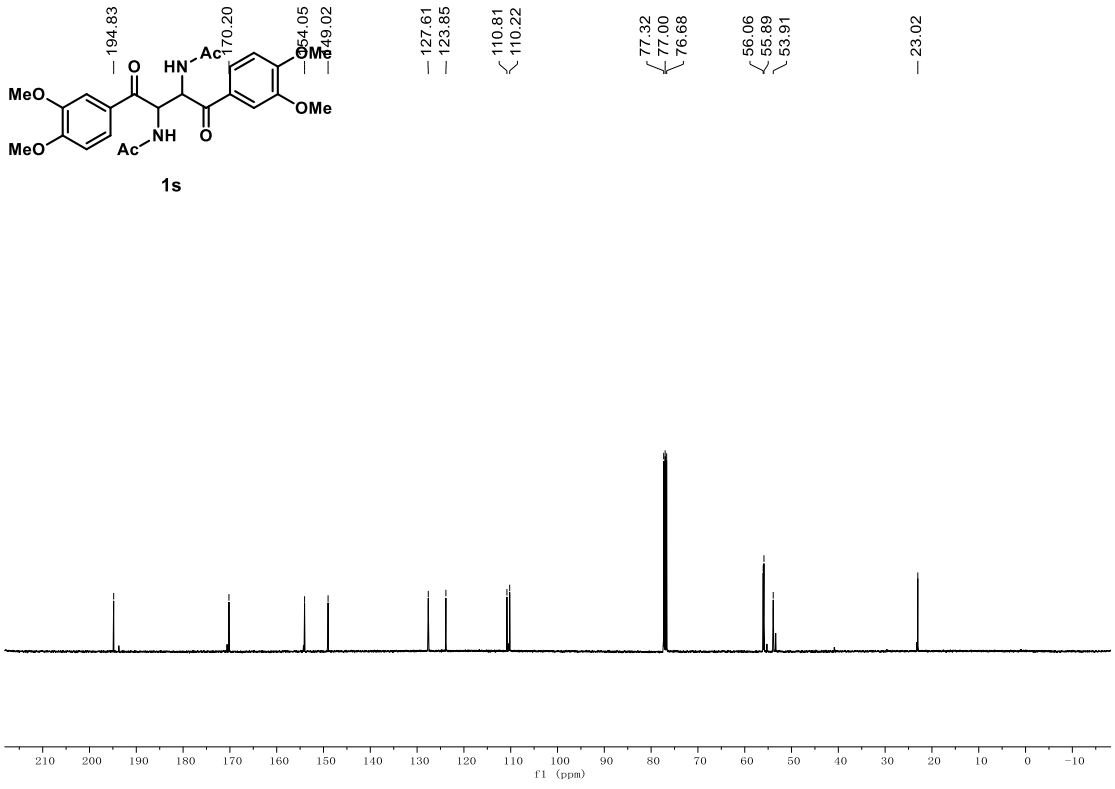
Supplementary Figure 40. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1r



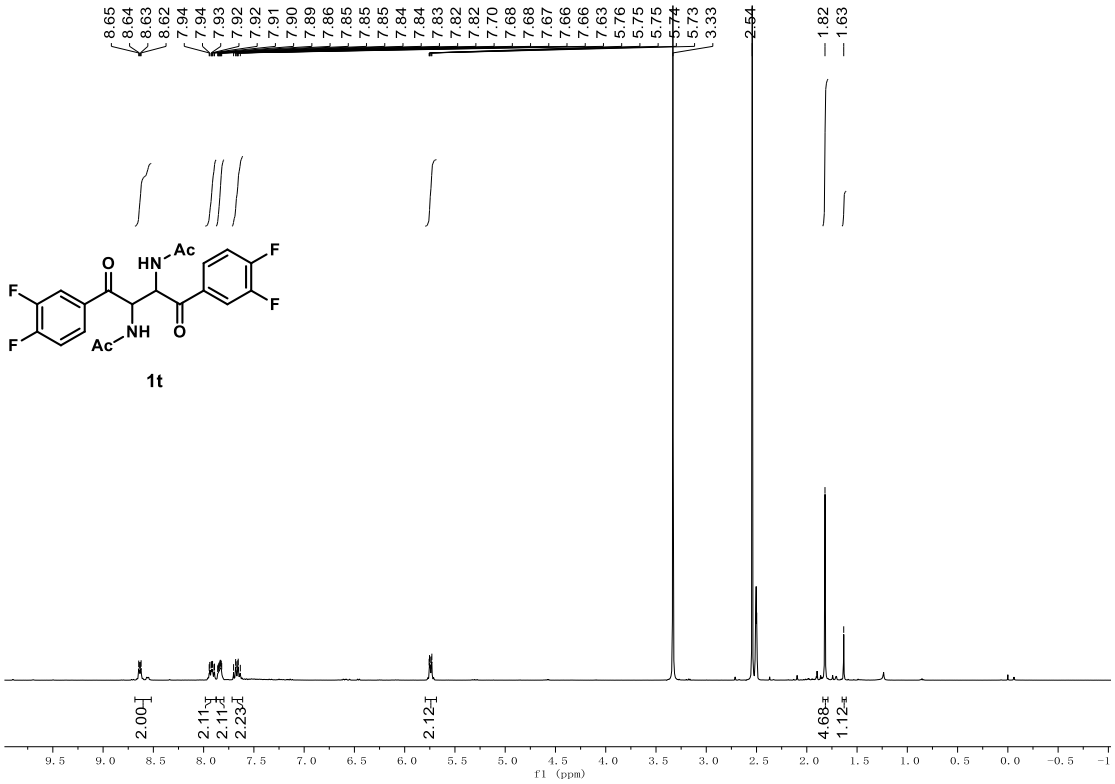
Supplementary Figure 41. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1r



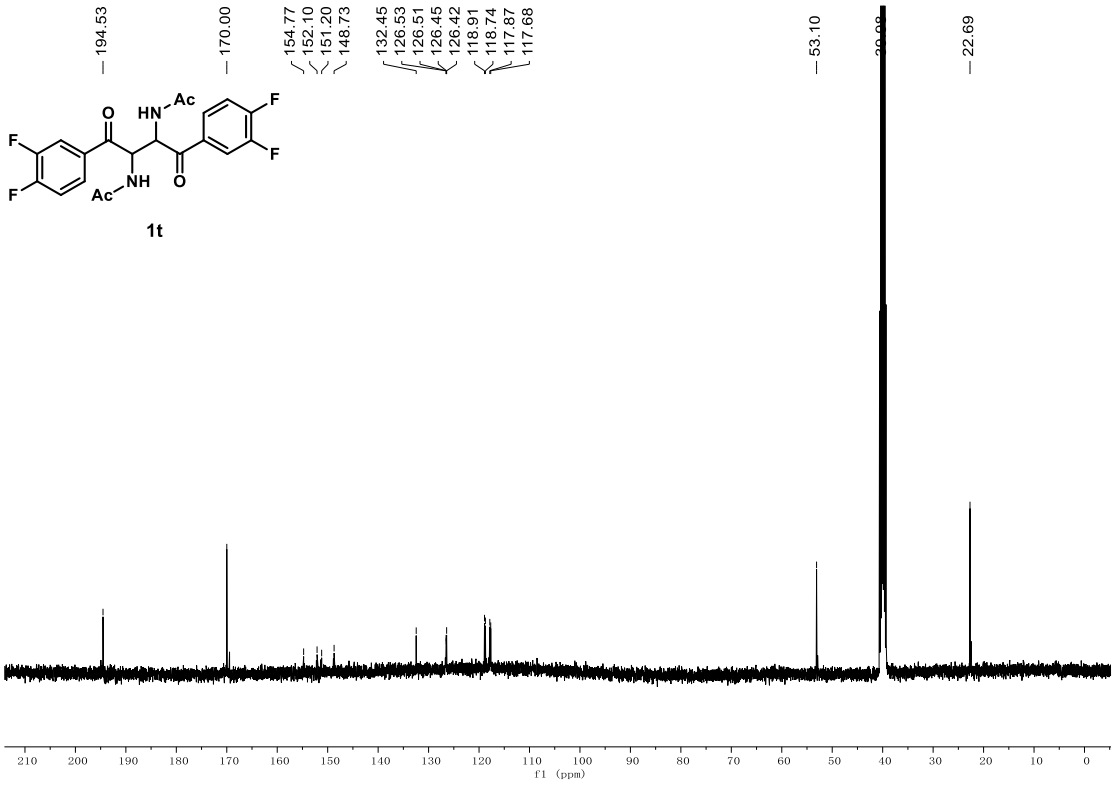
Supplementary Figure 42. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1s



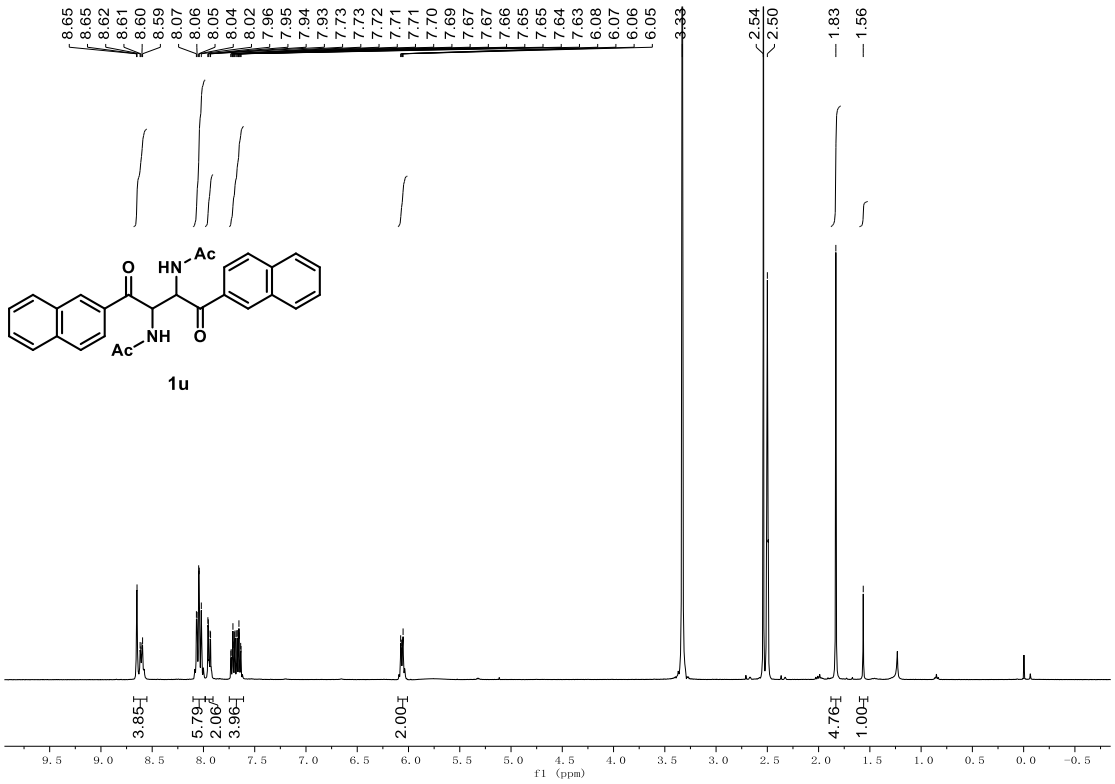
Supplementary Figure 43. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1s



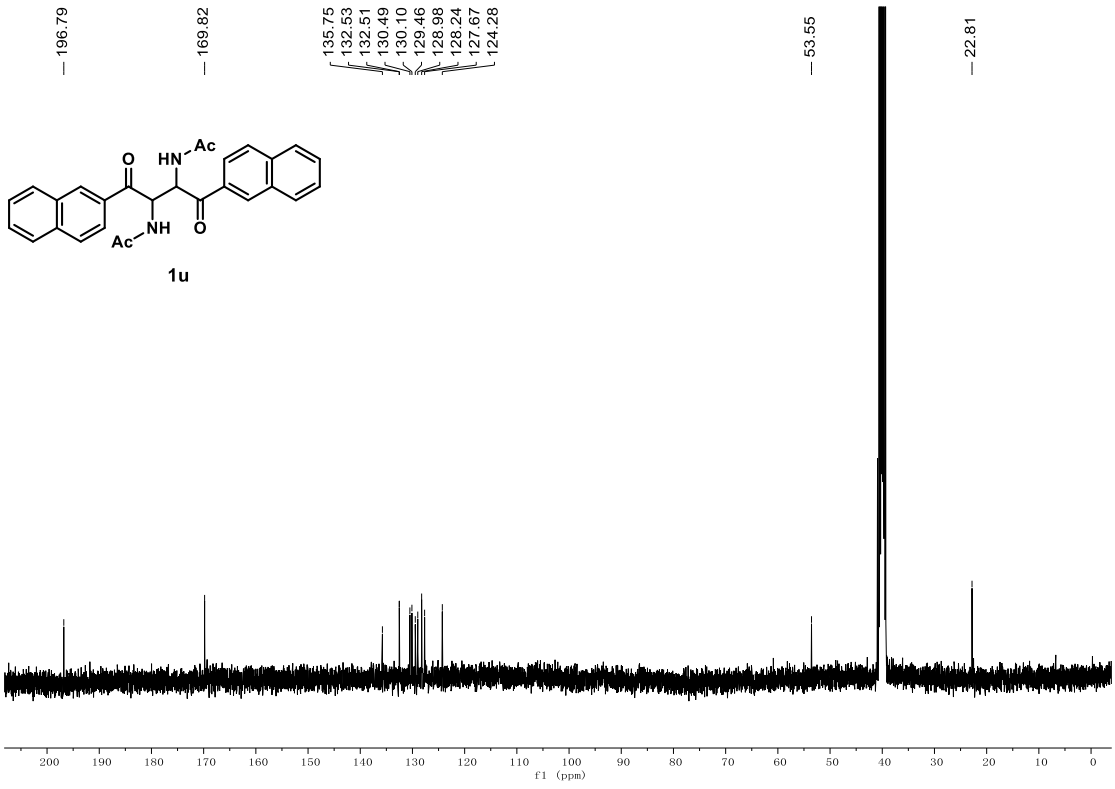
Supplementary Figure 44. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1t



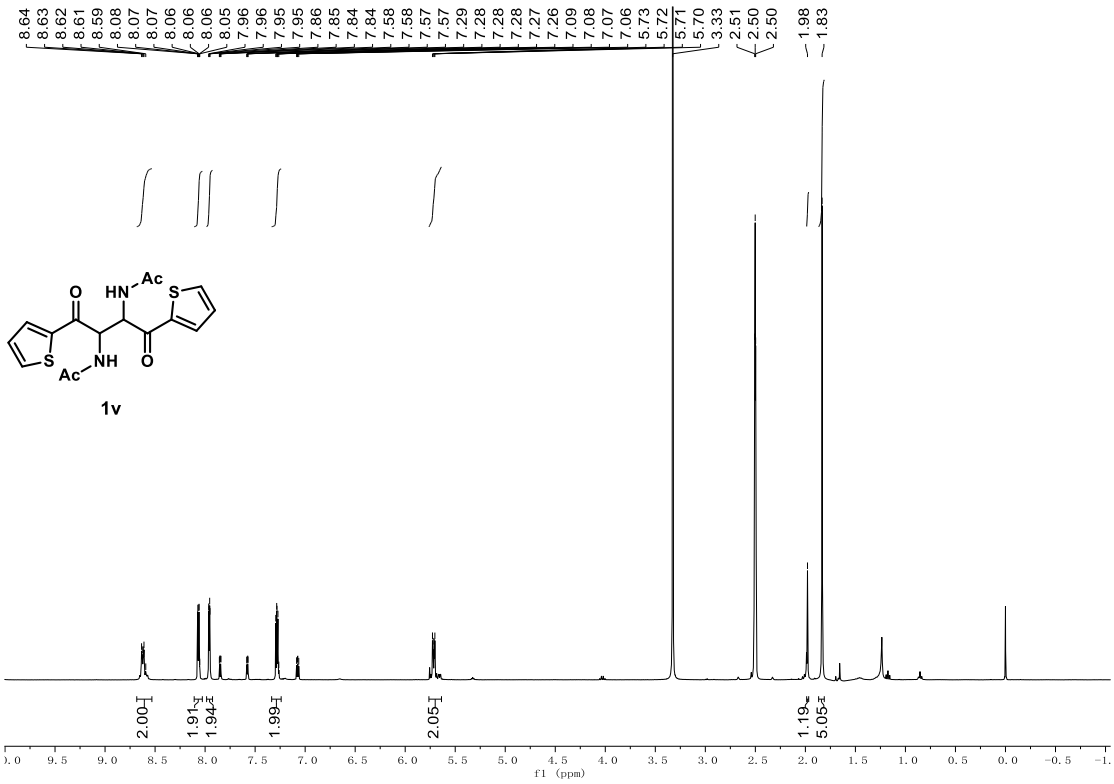
Supplementary Figure 45. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1t



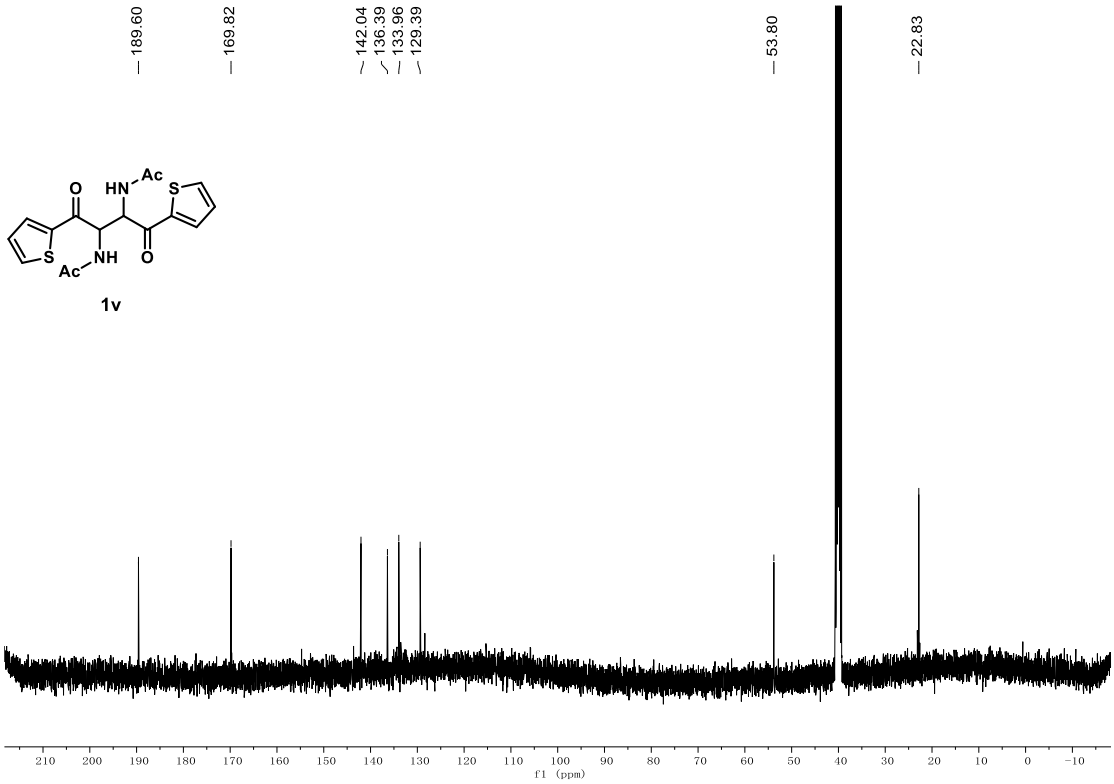
Supplementary Figure 46. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1u



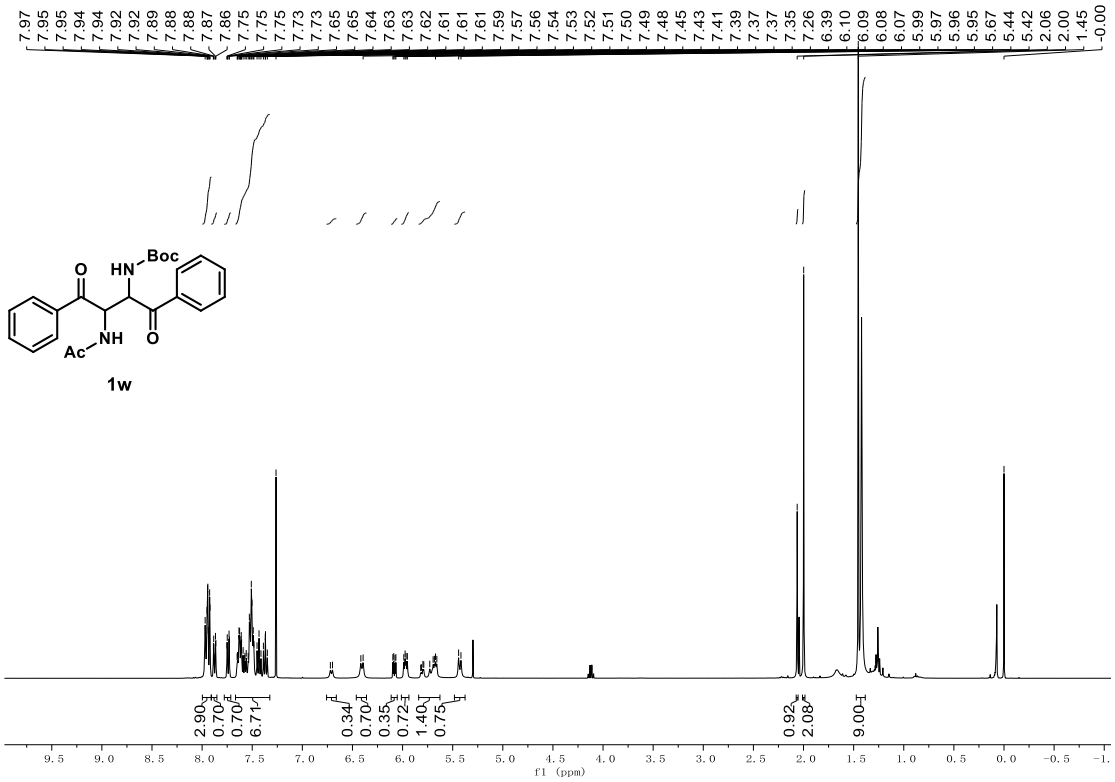
Supplementary Figure 47. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1u



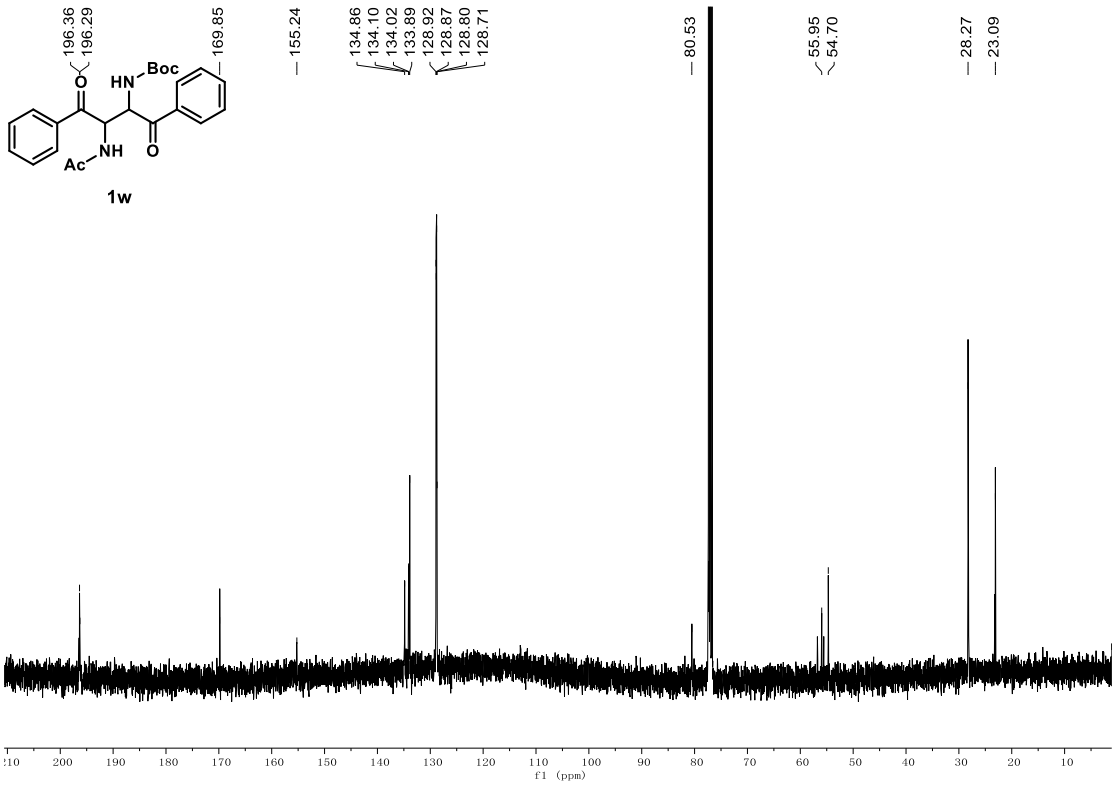
Supplementary Figure 48. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1v



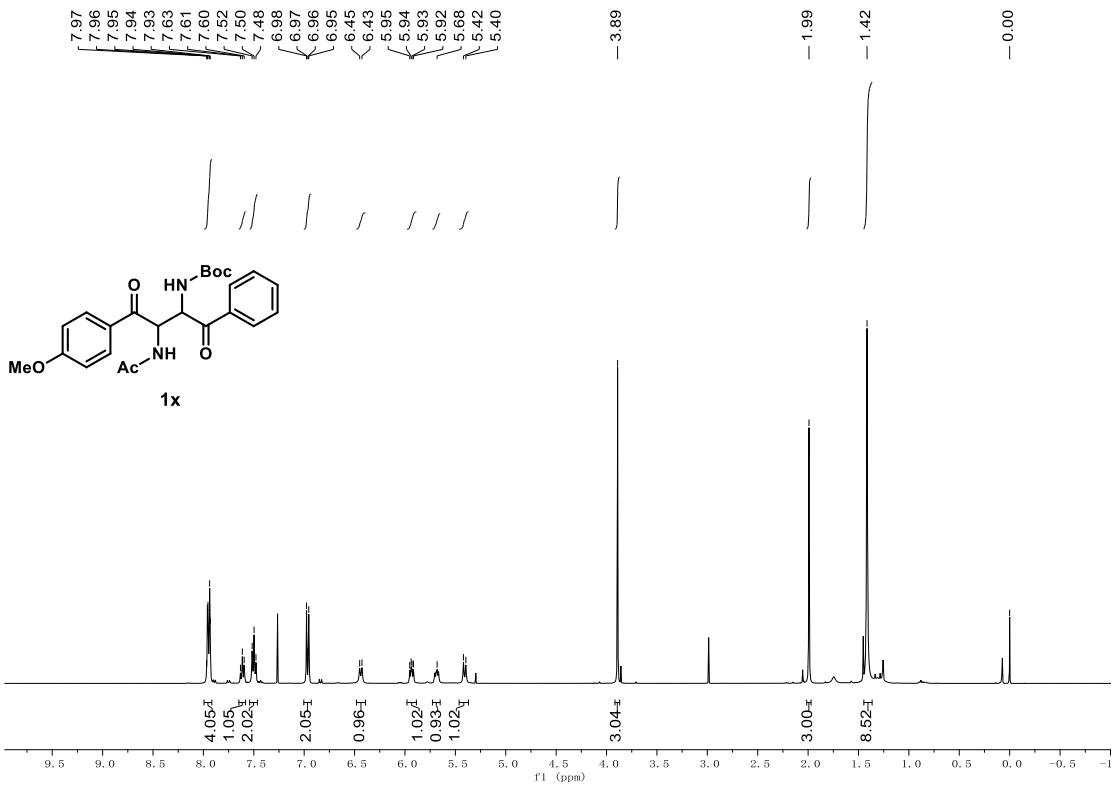
Supplementary Figure 49. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1v



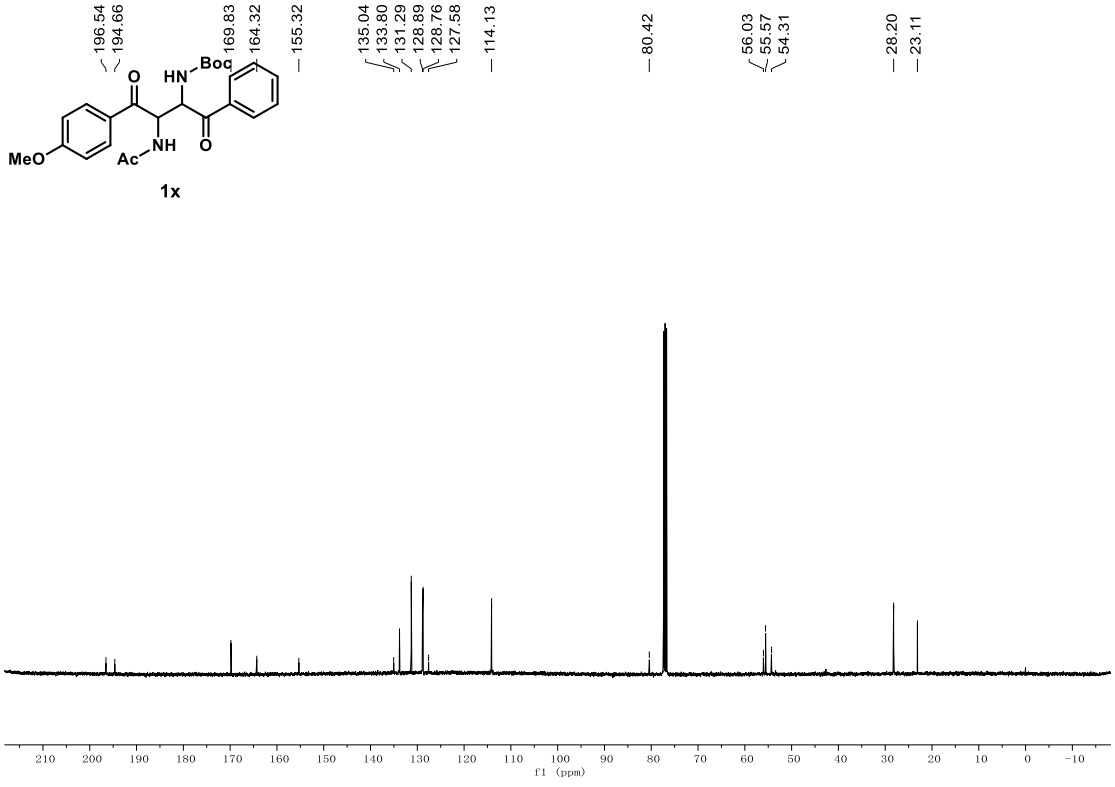
Supplementary Figure 50. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1w



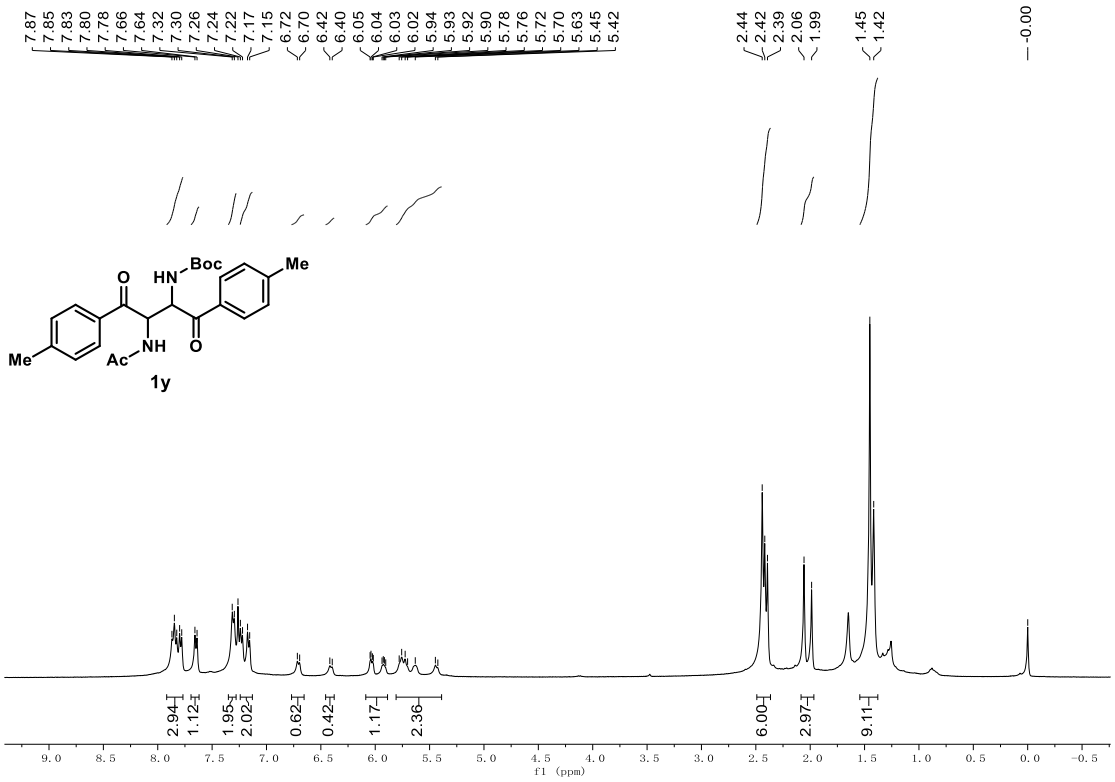
Supplementary Figure 51. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1w



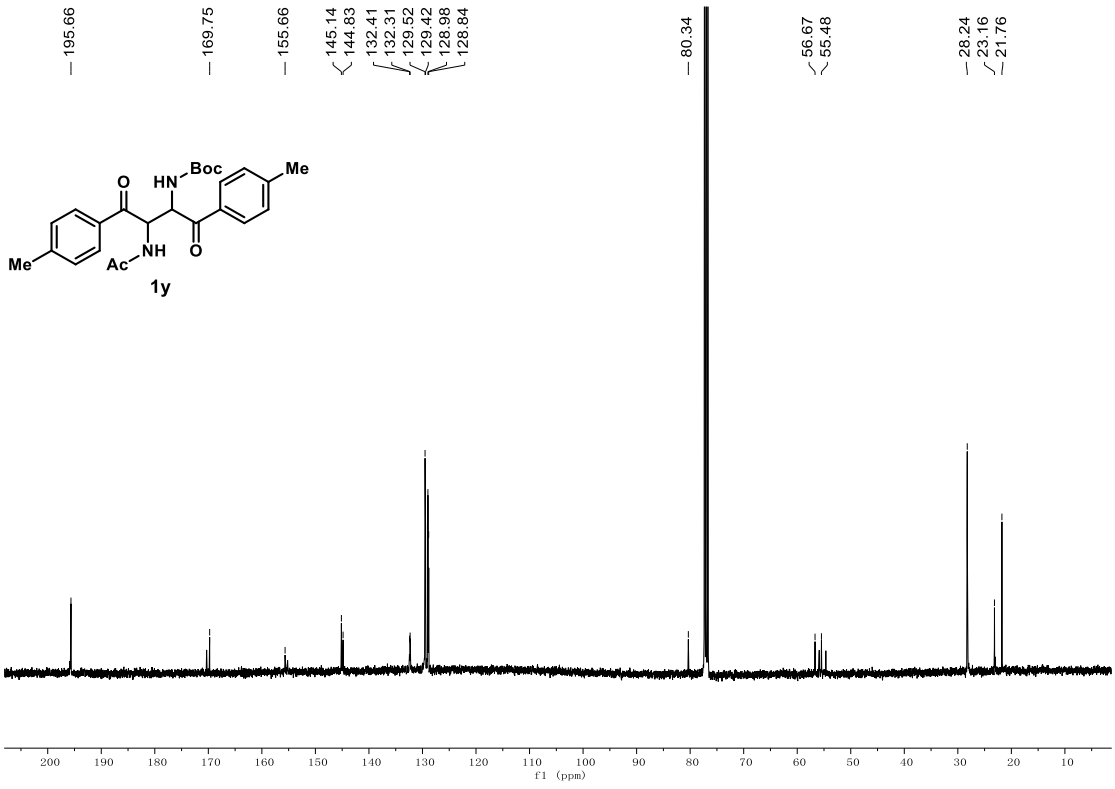
Supplementary Figure 52. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1x



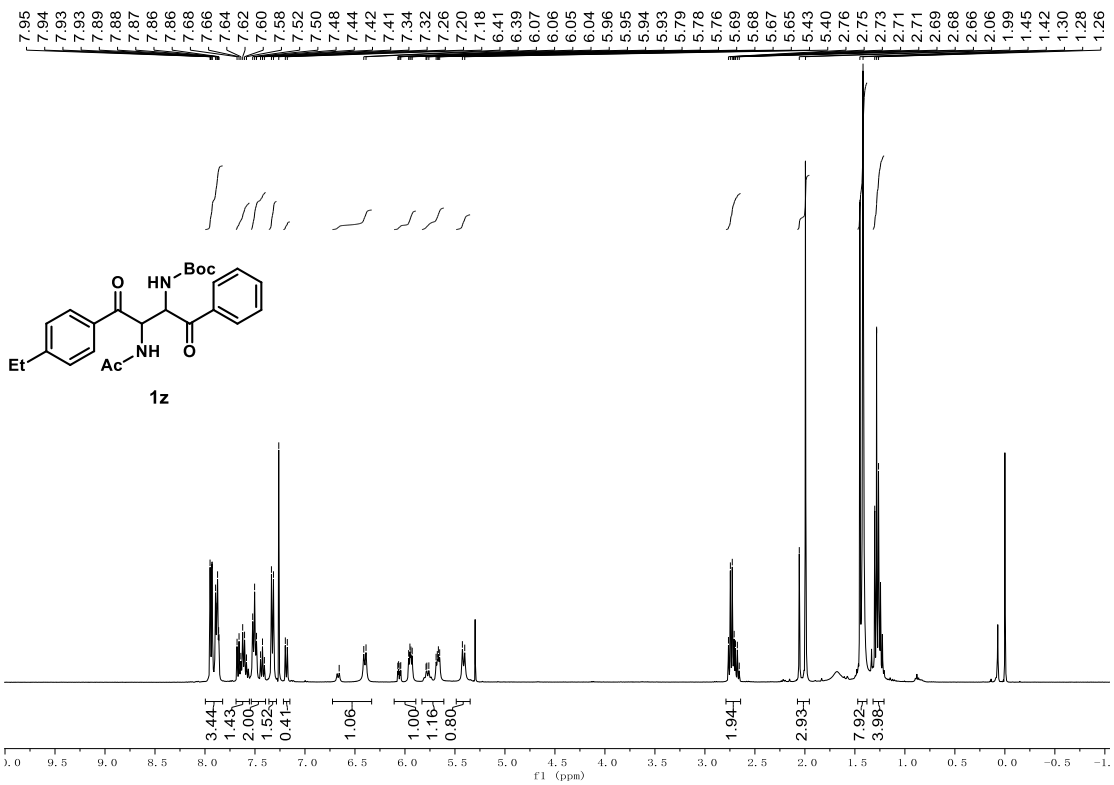
Supplementary Figure 53. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1x



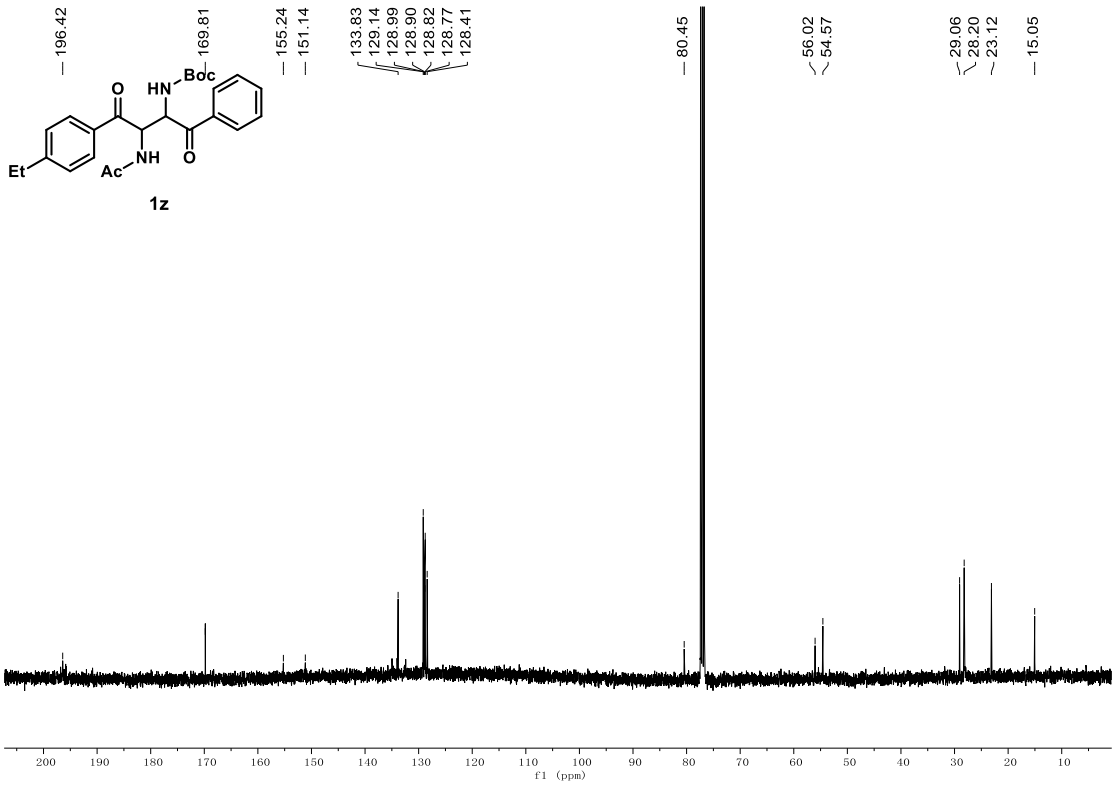
Supplementary Figure 54. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1y



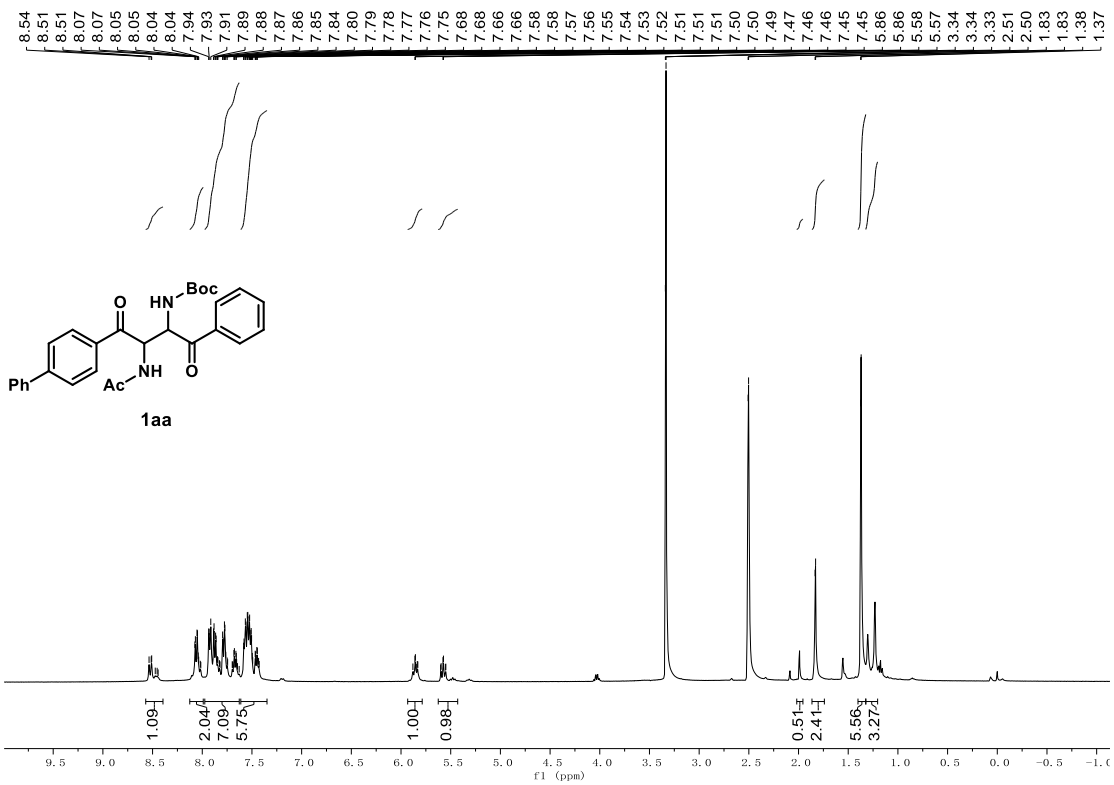
Supplementary Figure 55. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1y



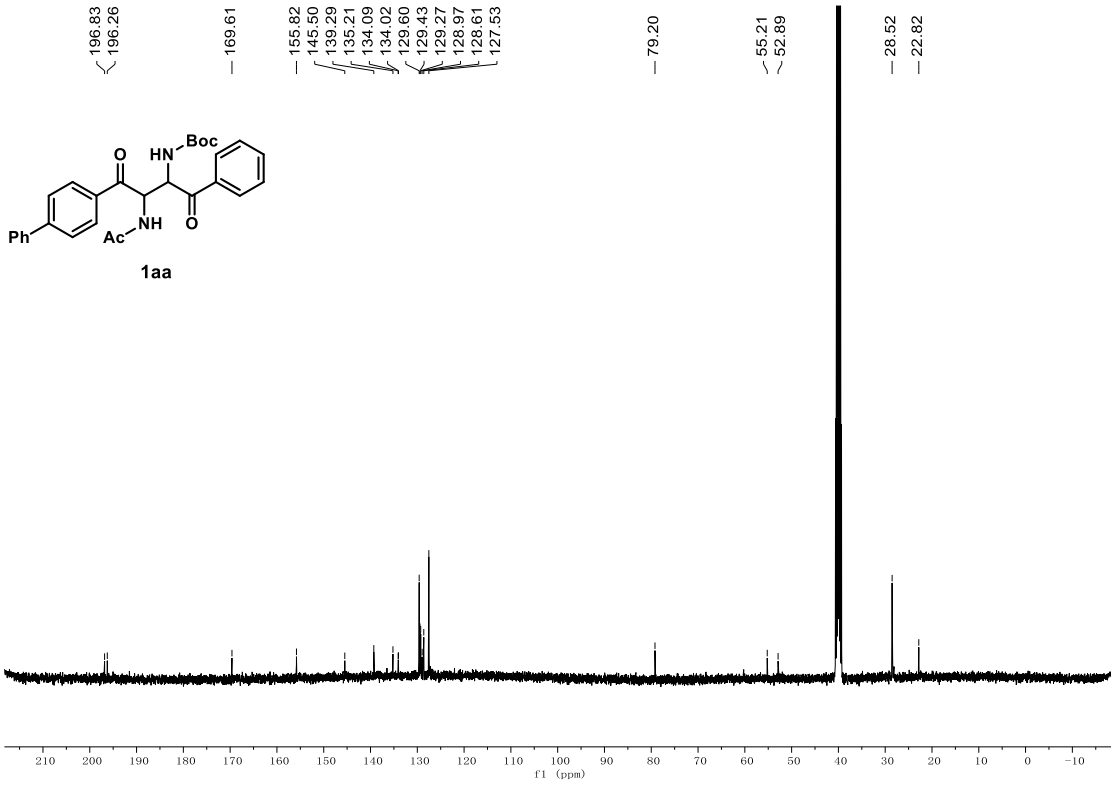
Supplementary Figure 56. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1z



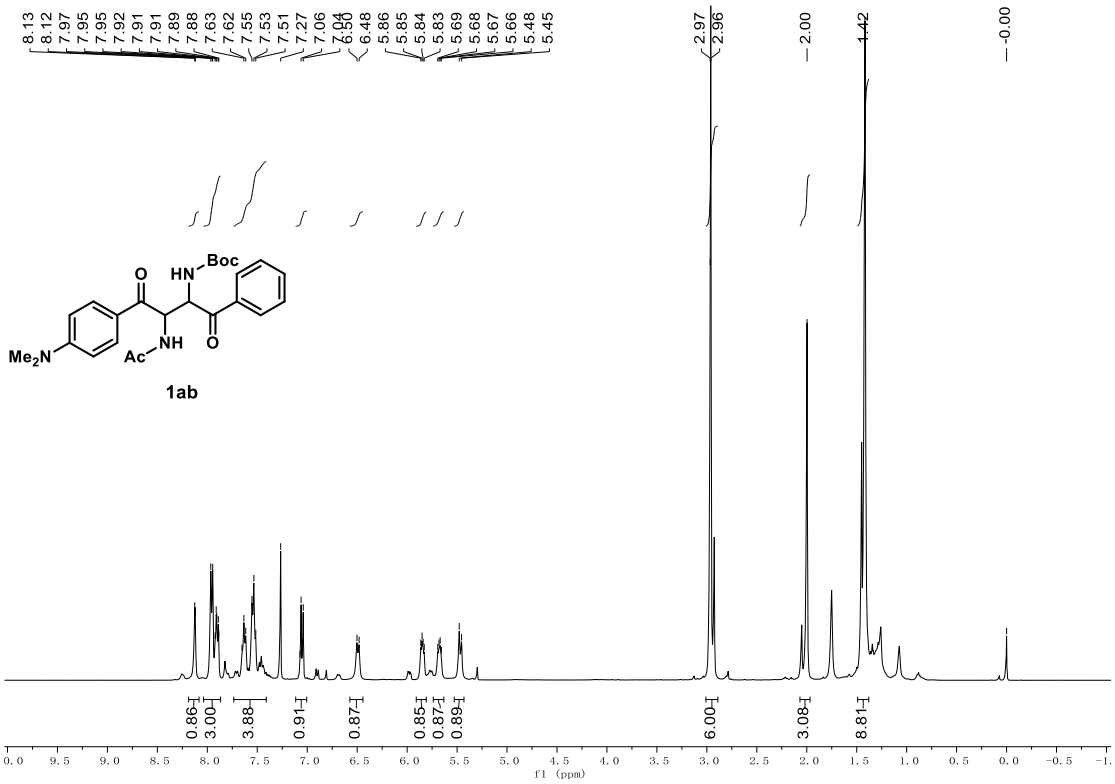
Supplementary Figure 57. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1z



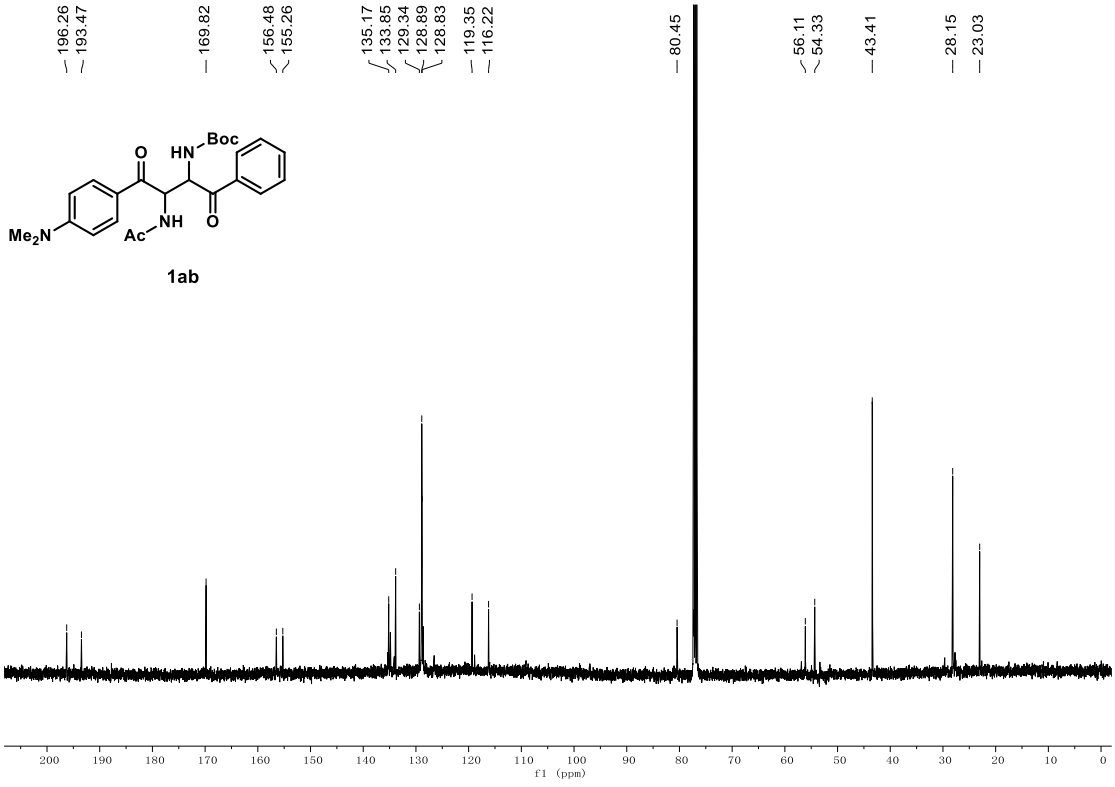
Supplementary Figure 58. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1aa



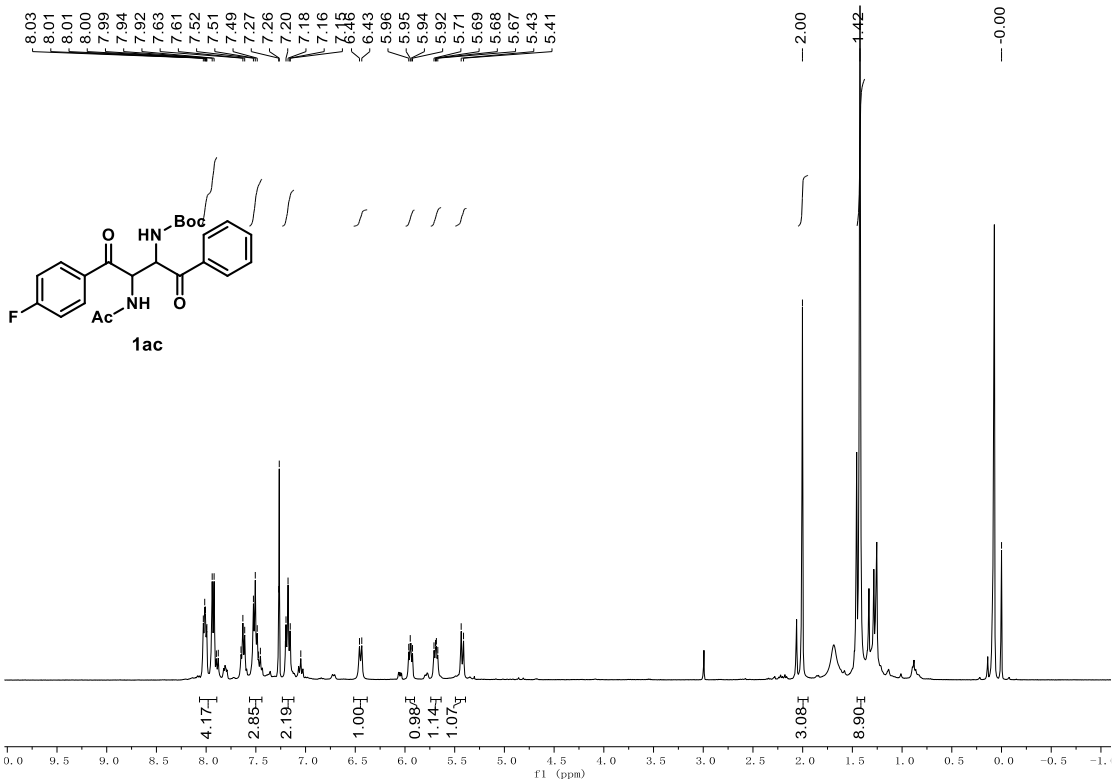
Supplementary Figure 59. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1aa



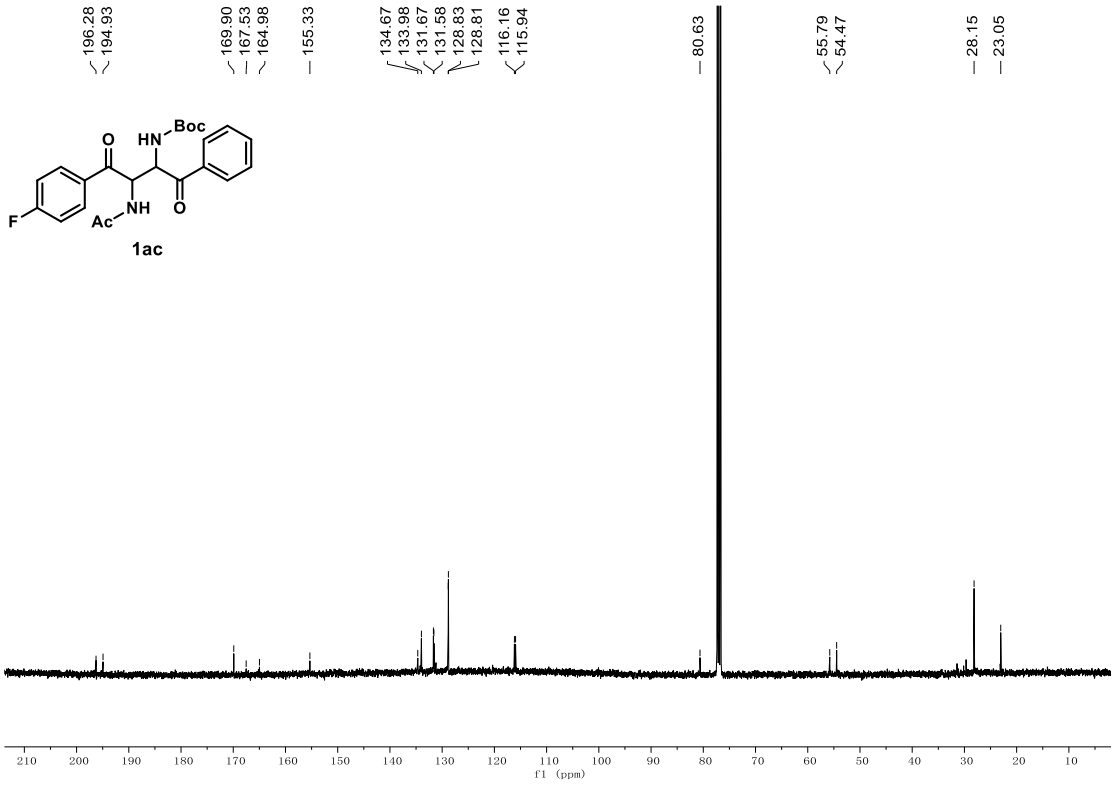
Supplementary Figure 60. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ab



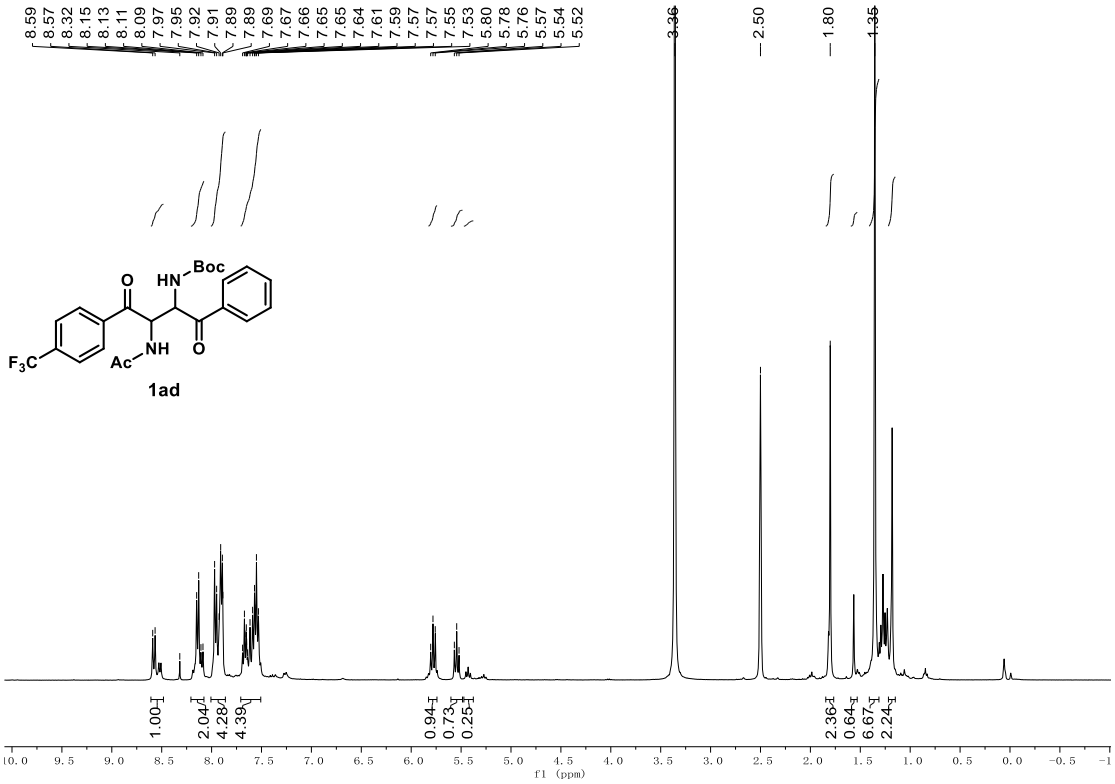
Supplementary Figure 61. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ab



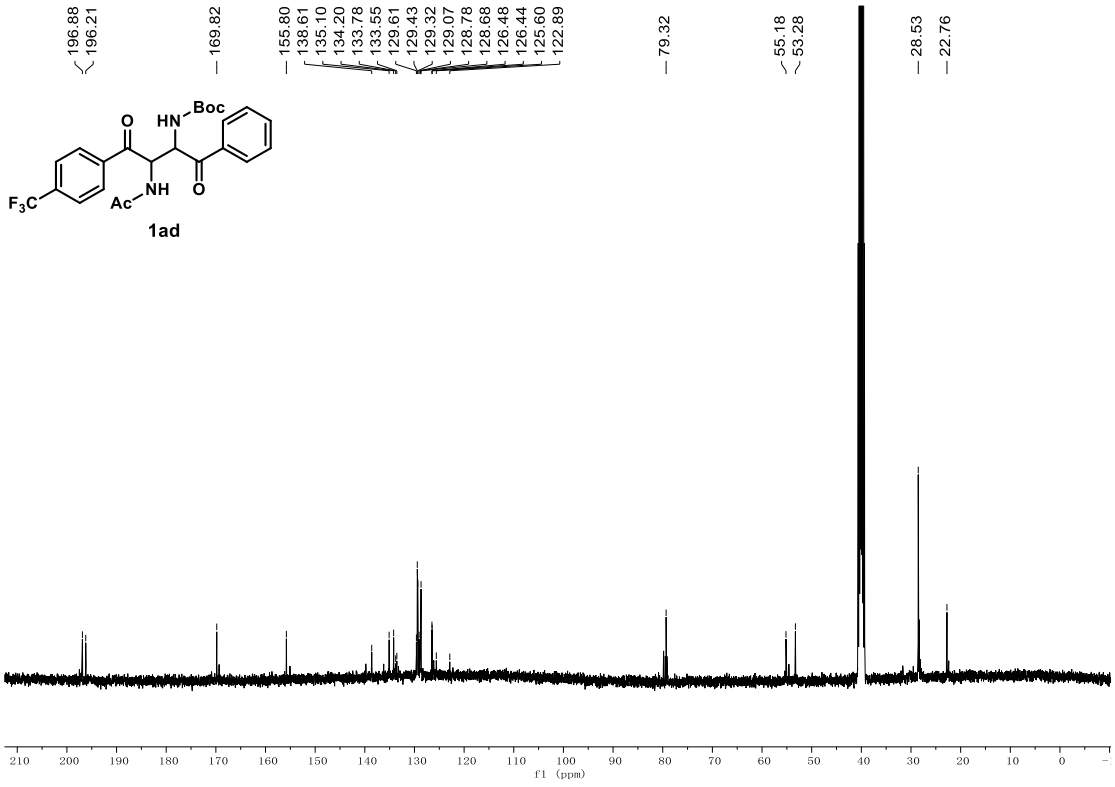
Supplementary Figure 62. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ac



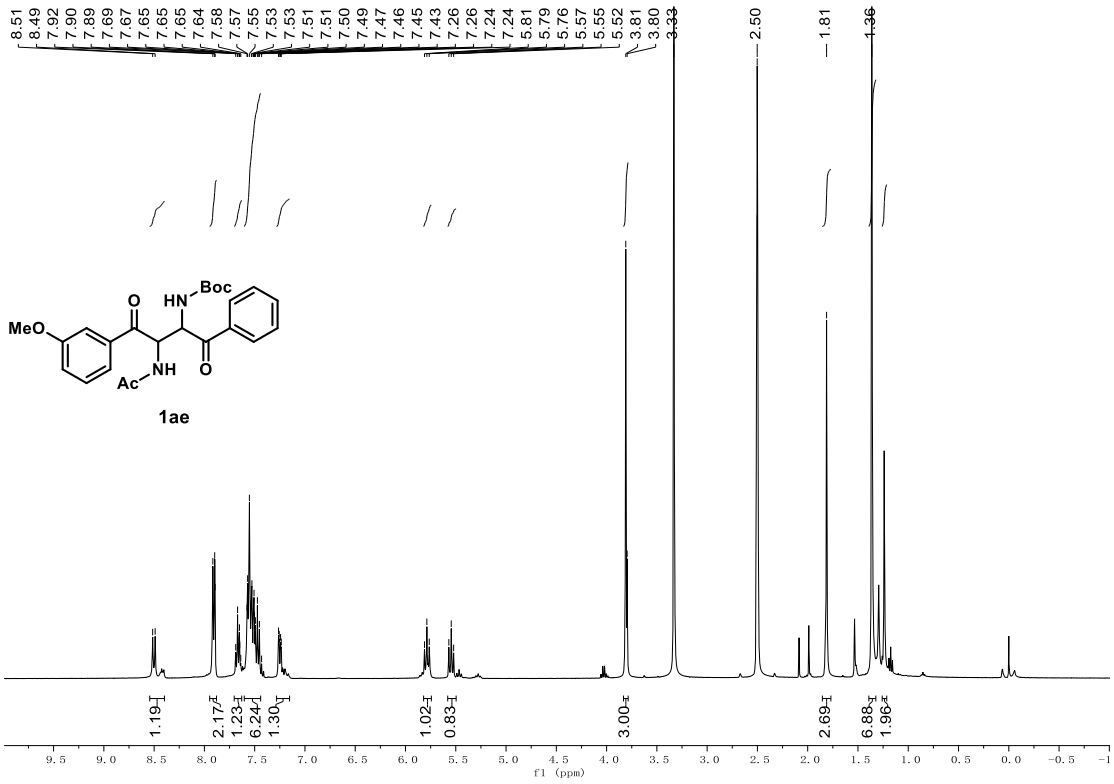
Supplementary Figure 63. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ac



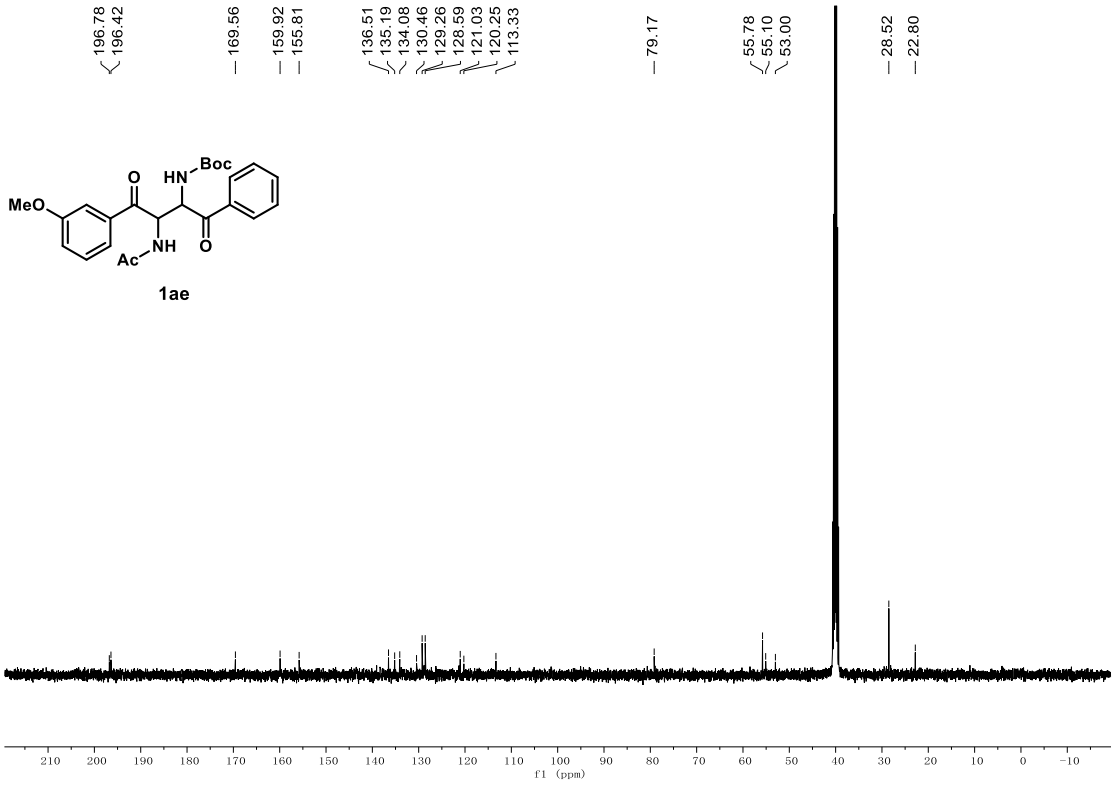
Supplementary Figure 64. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ad



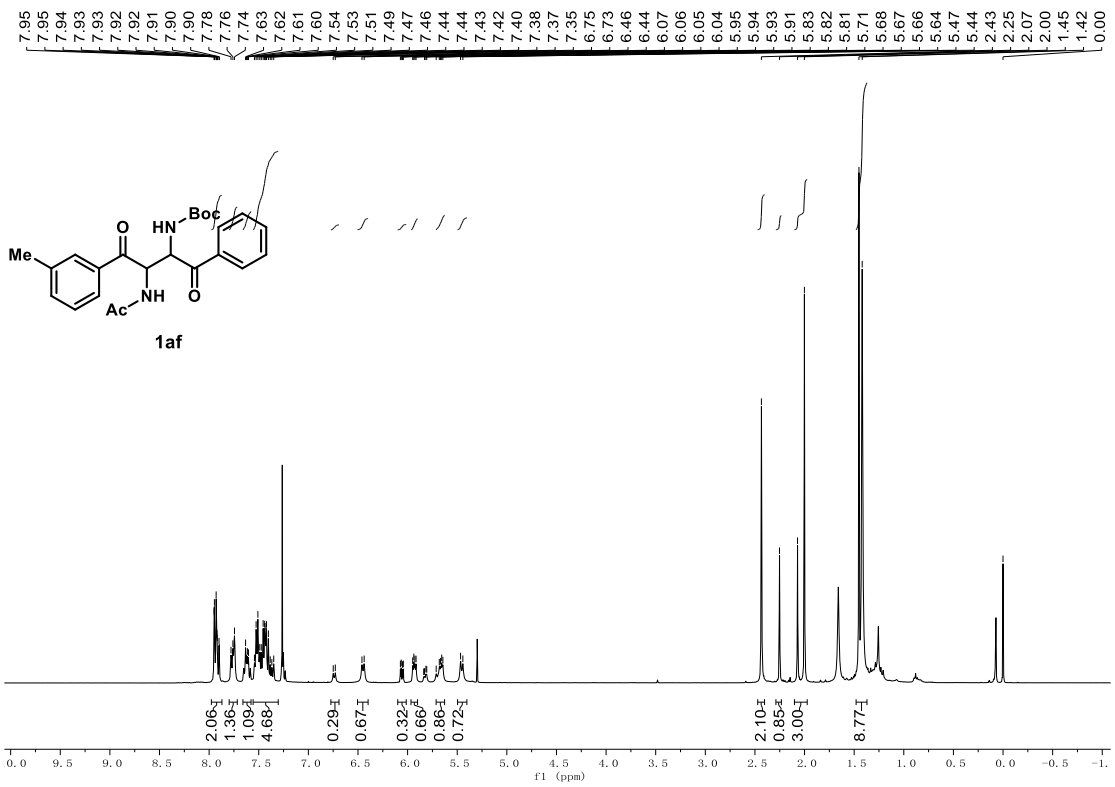
Supplementary Figure 65. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ad



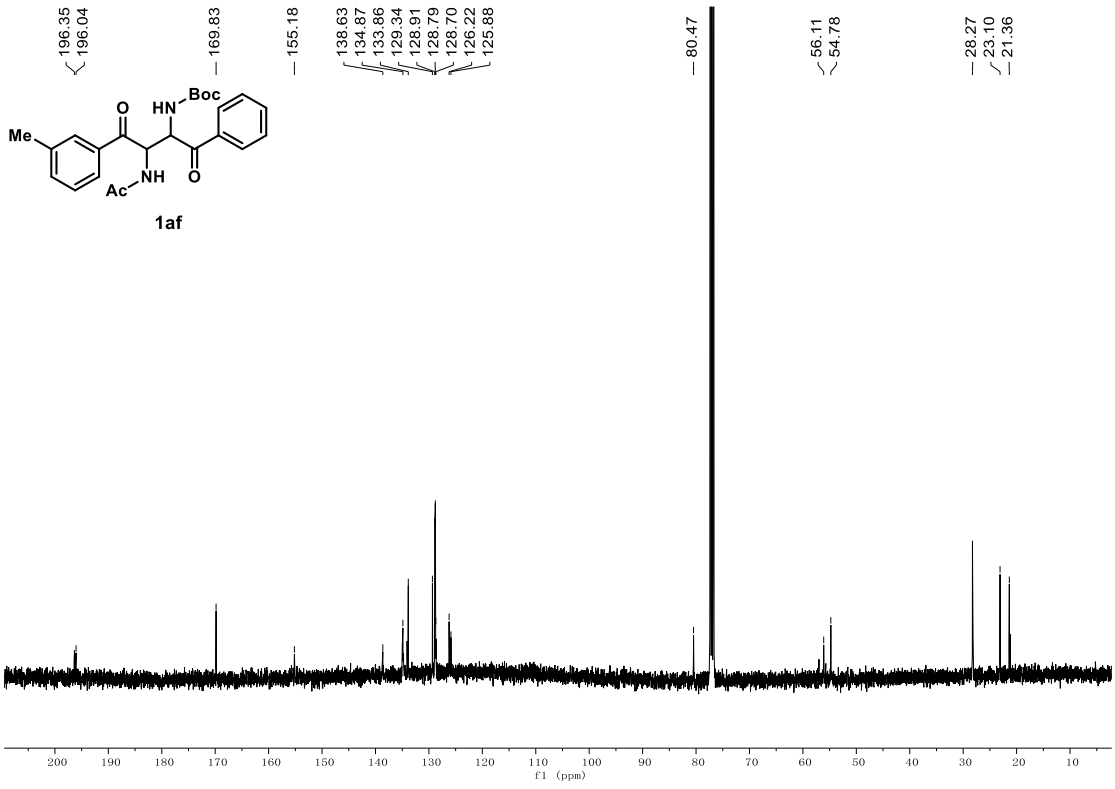
Supplementary Figure 66. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ae



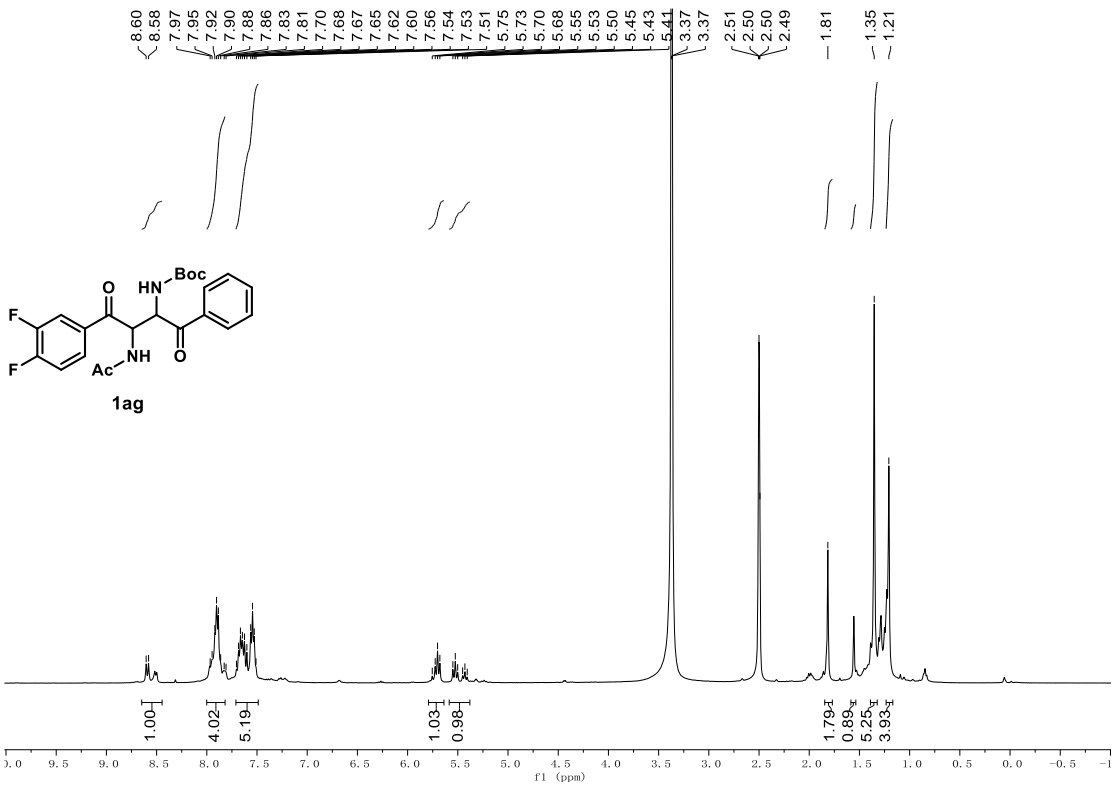
Supplementary Figure 67. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ae



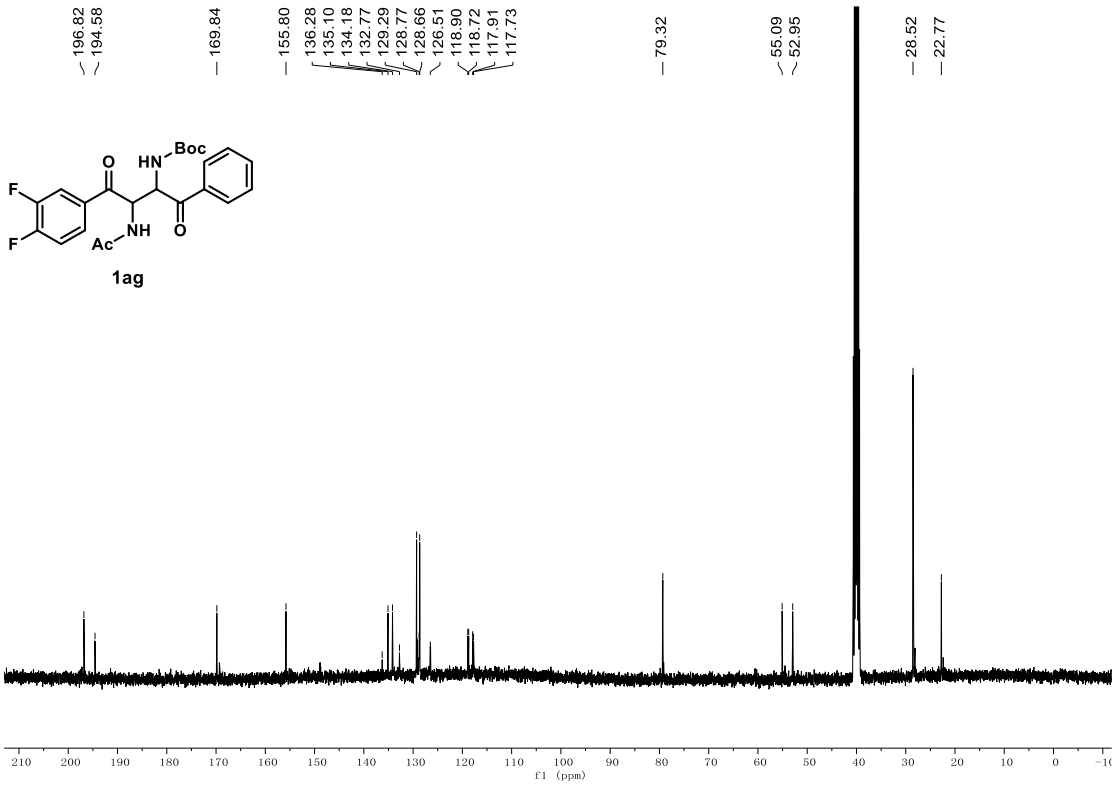
Supplementary Figure 68. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1af



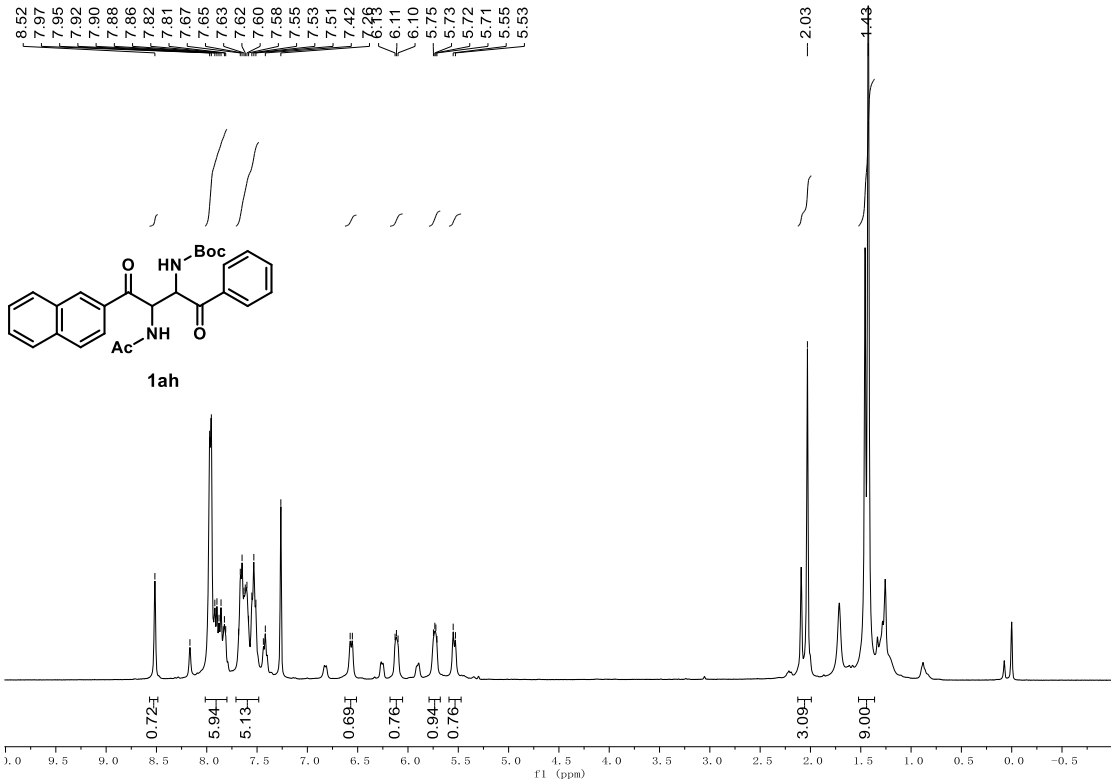
Supplementary Figure 69. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1af



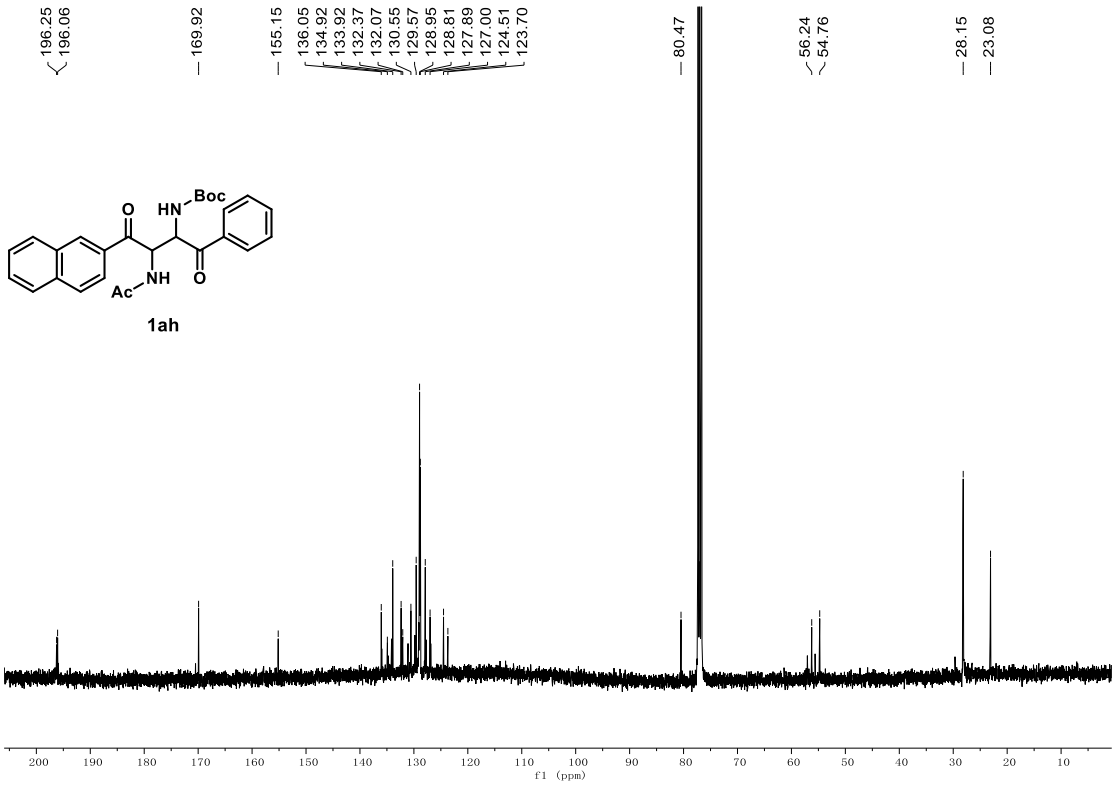
Supplementary Figure 70. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ag



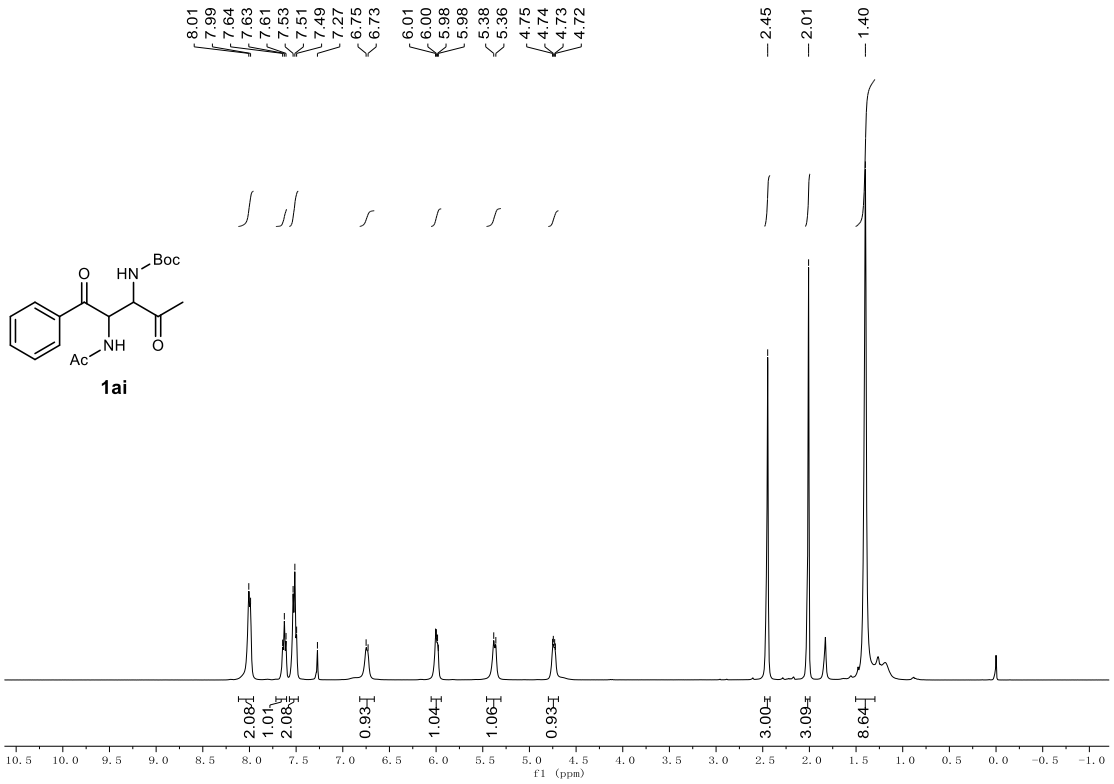
Supplementary Figure 71. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ag



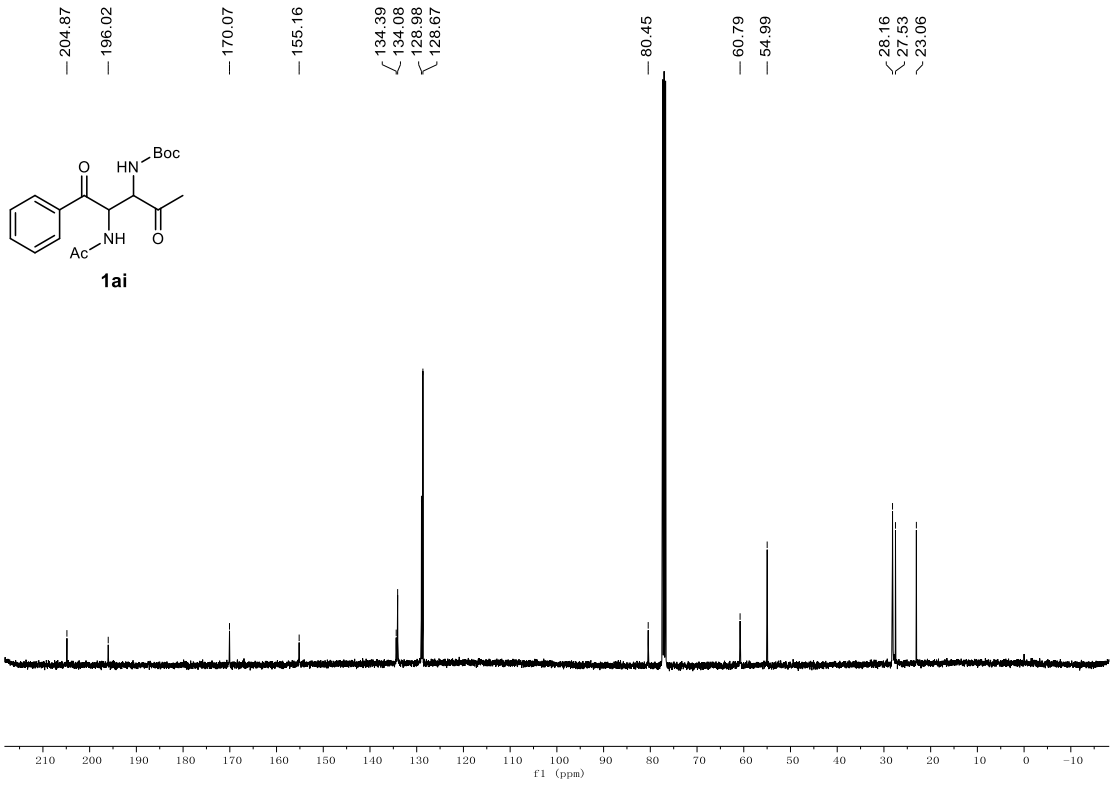
^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ah



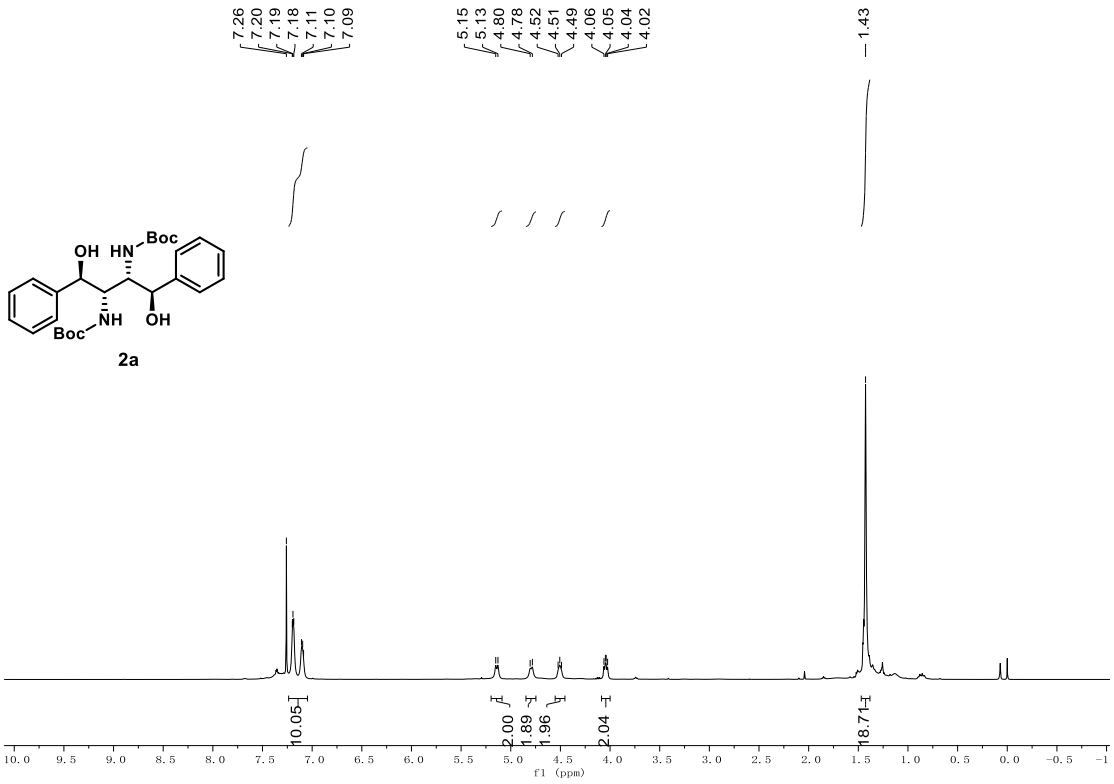
Supplementary Figure 72. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ah



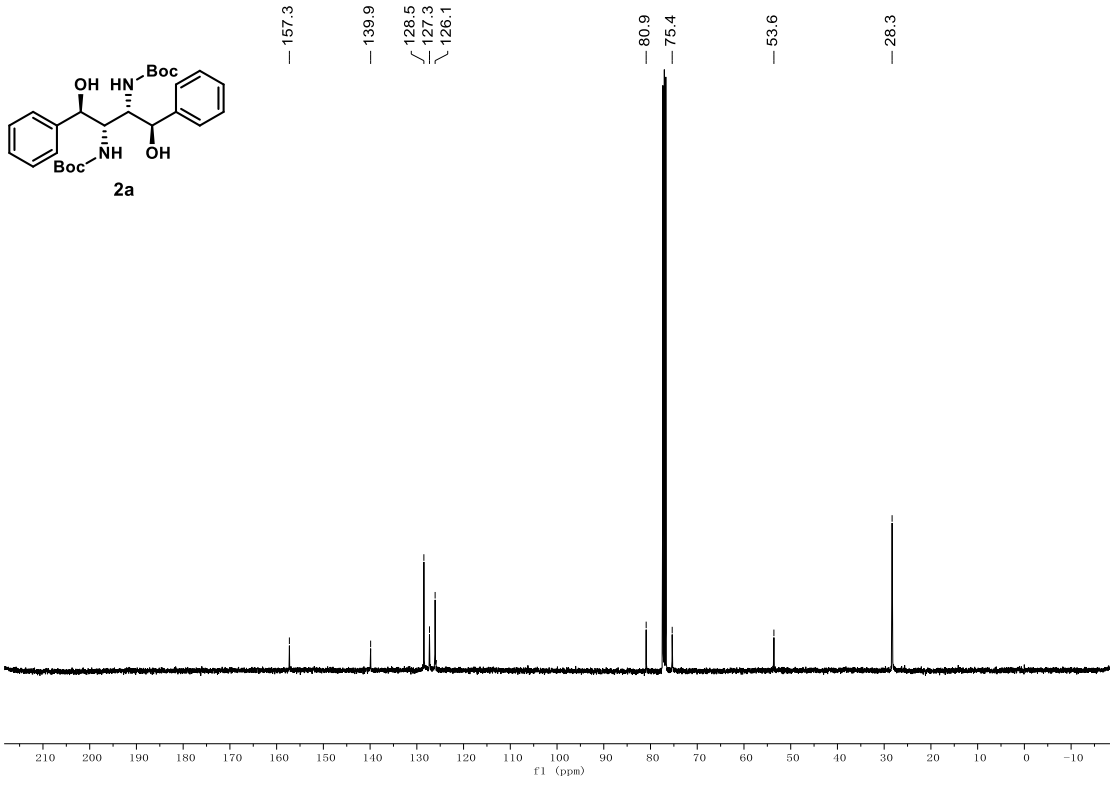
^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 1ai



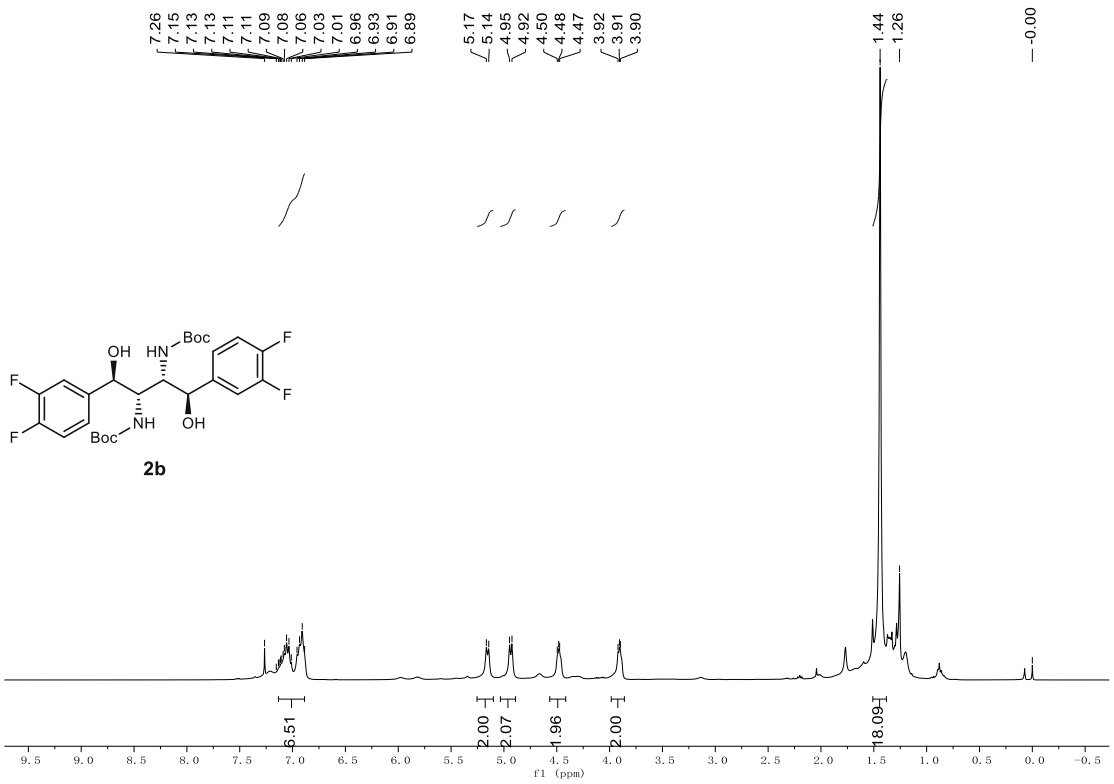
Supplementary Figure 73. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 1ai



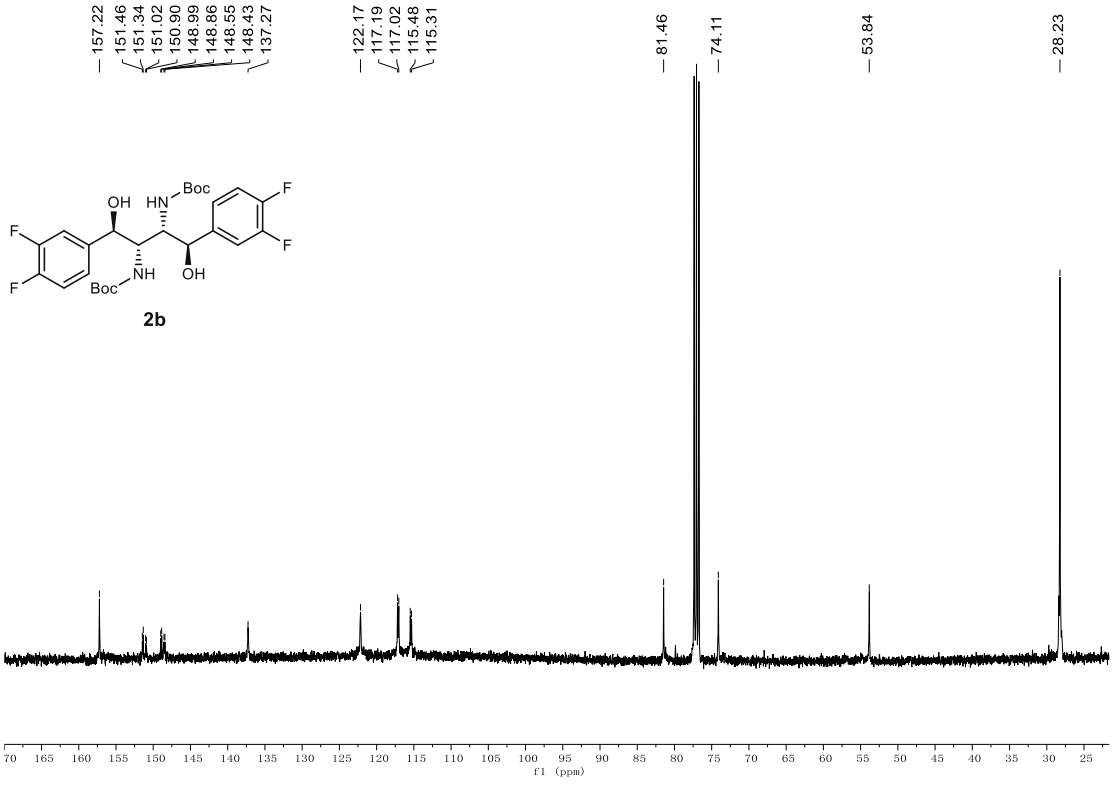
Supplementary Figure 74. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2a



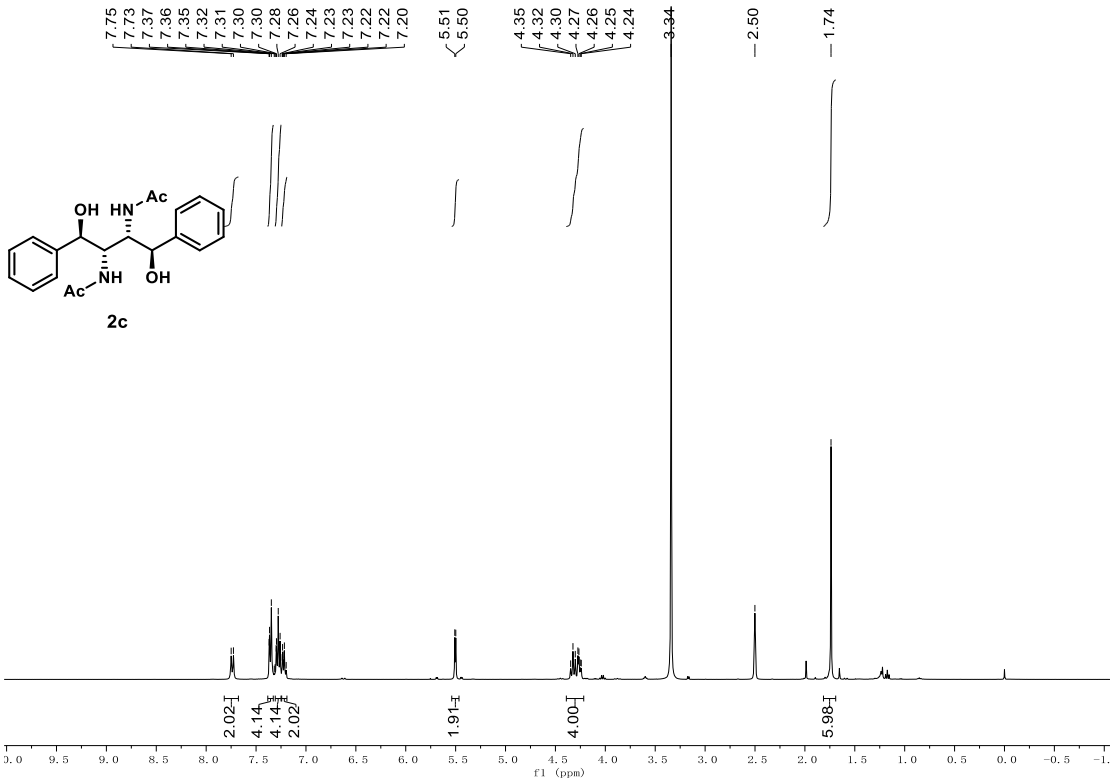
Supplementary Figure 75. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2a



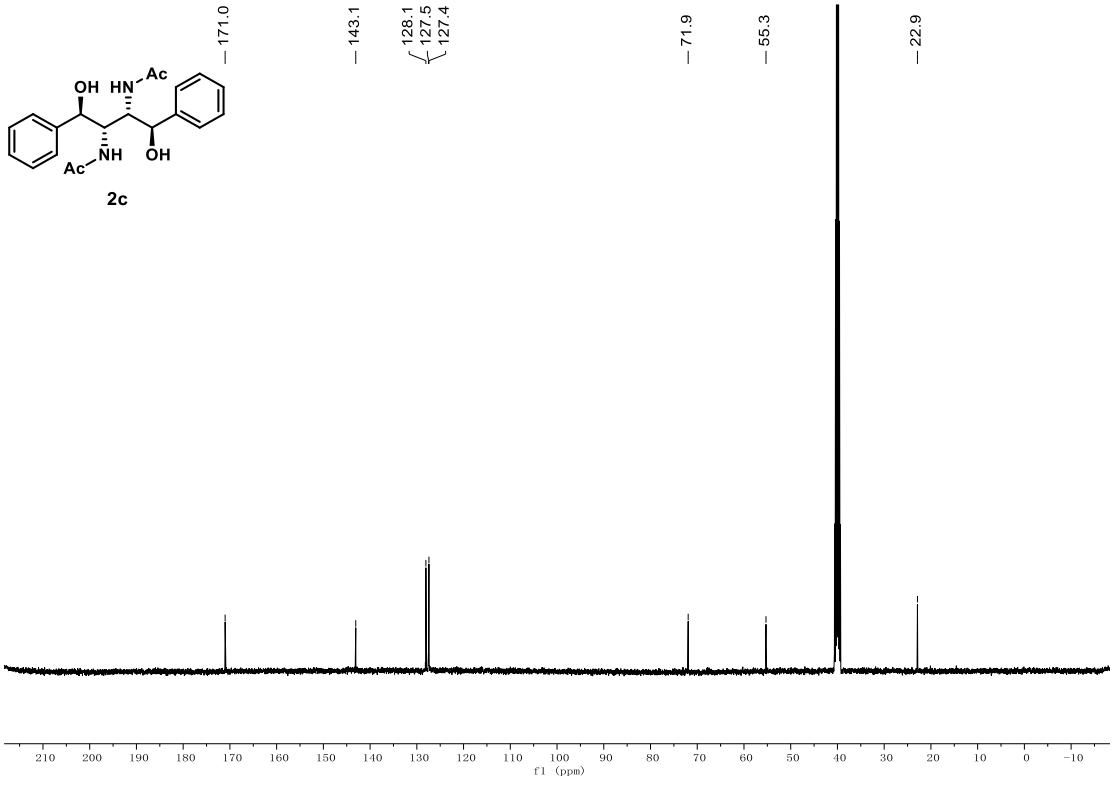
Supplementary Figure 76. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2b



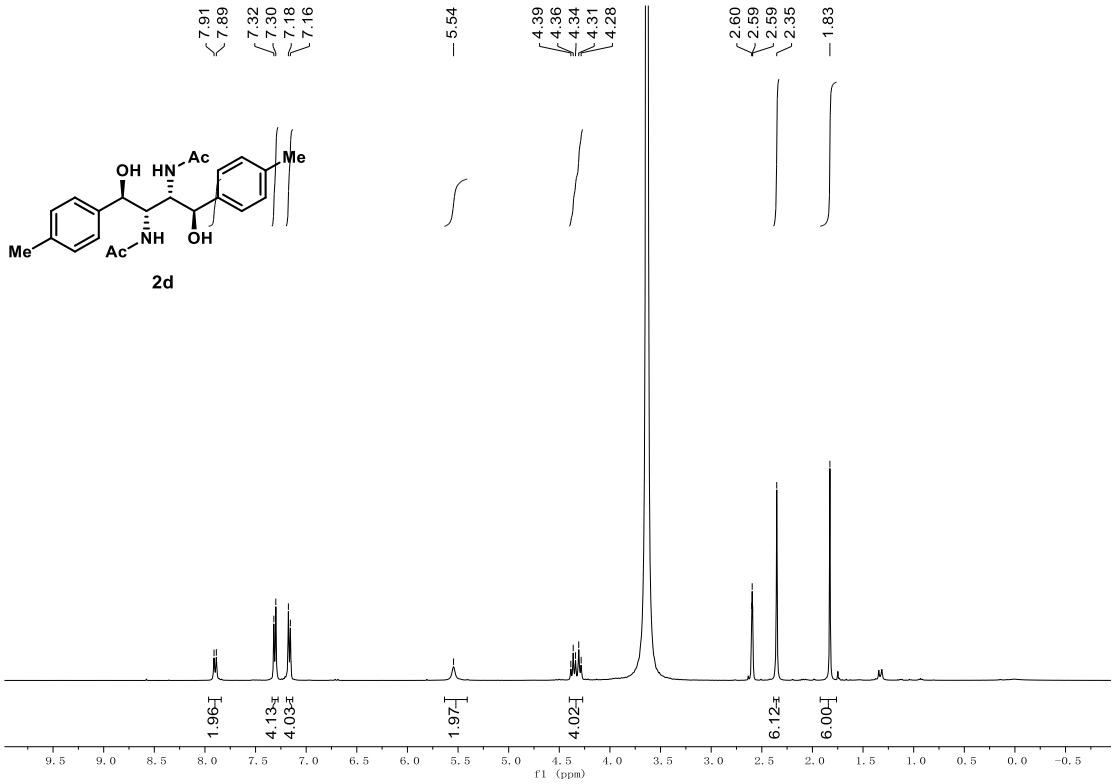
Supplementary Figure 77. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2b



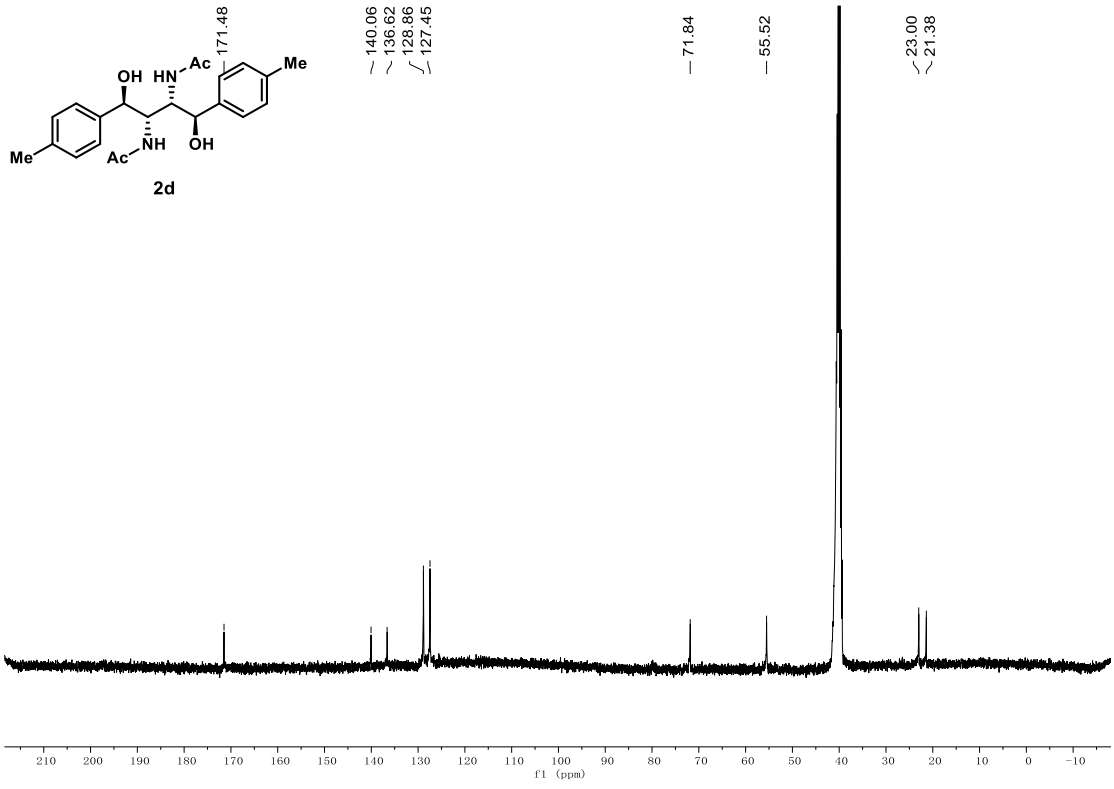
Supplementary Figure 78. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2c



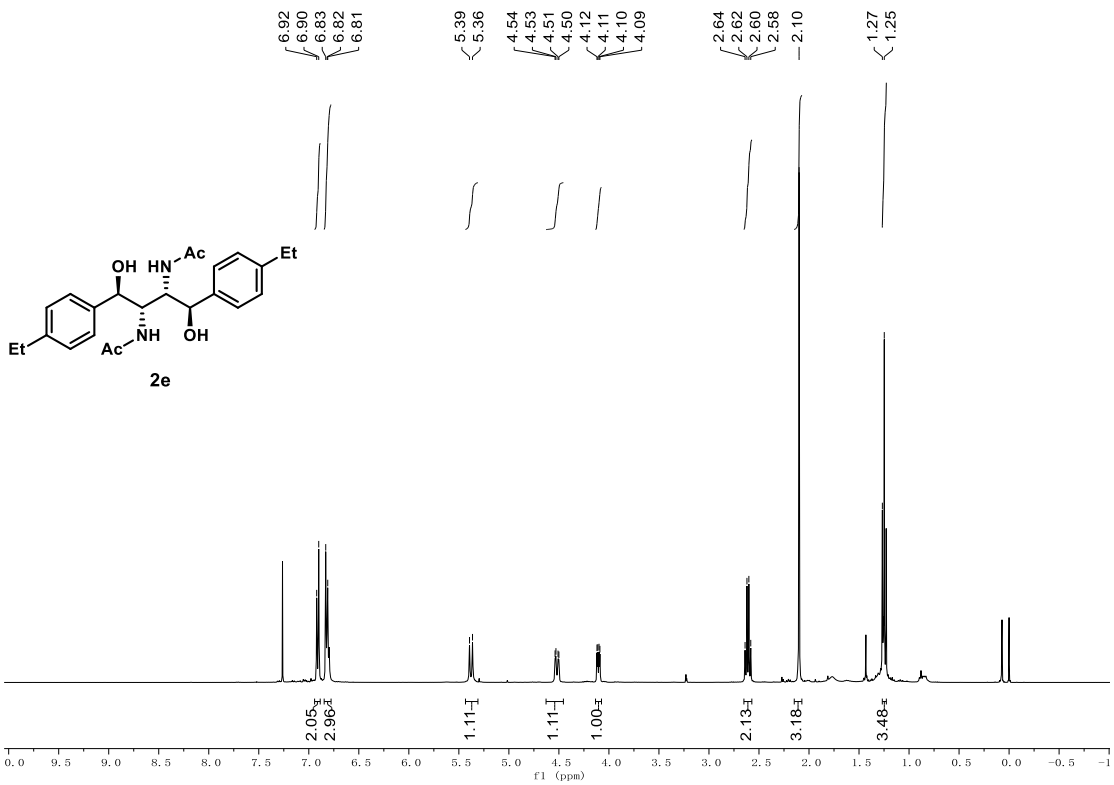
Supplementary Figure 79. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2c



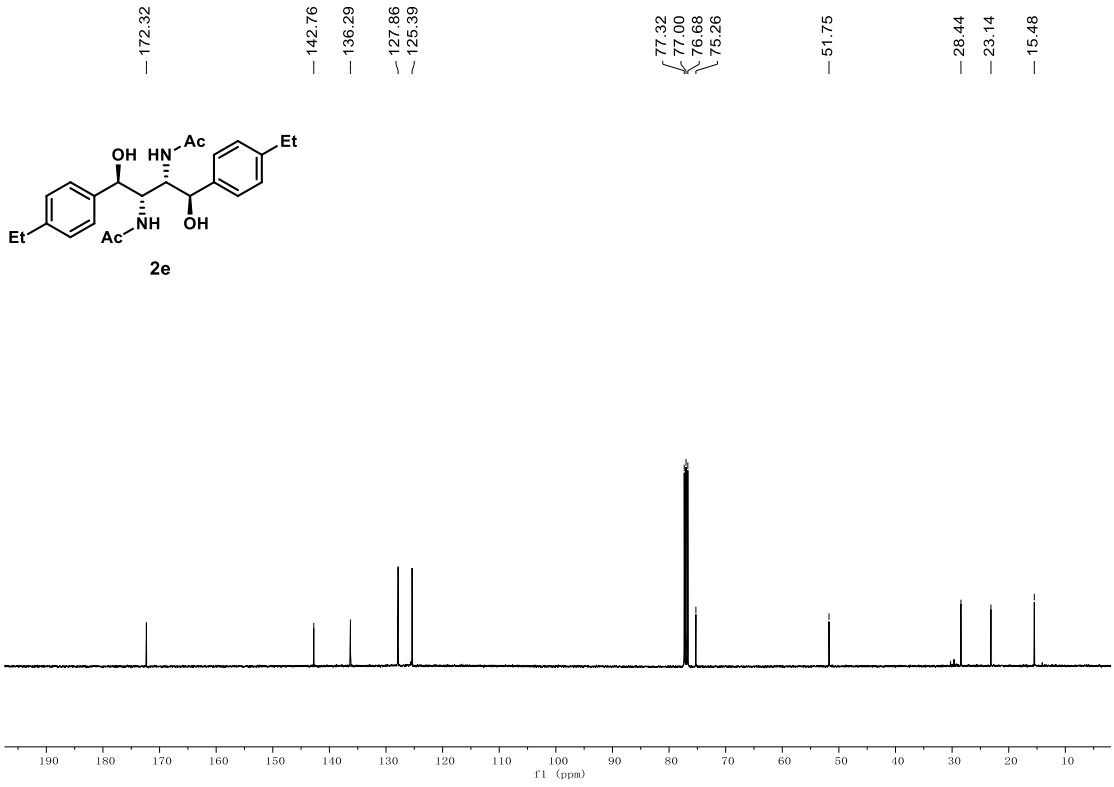
Supplementary Figure 80. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2d



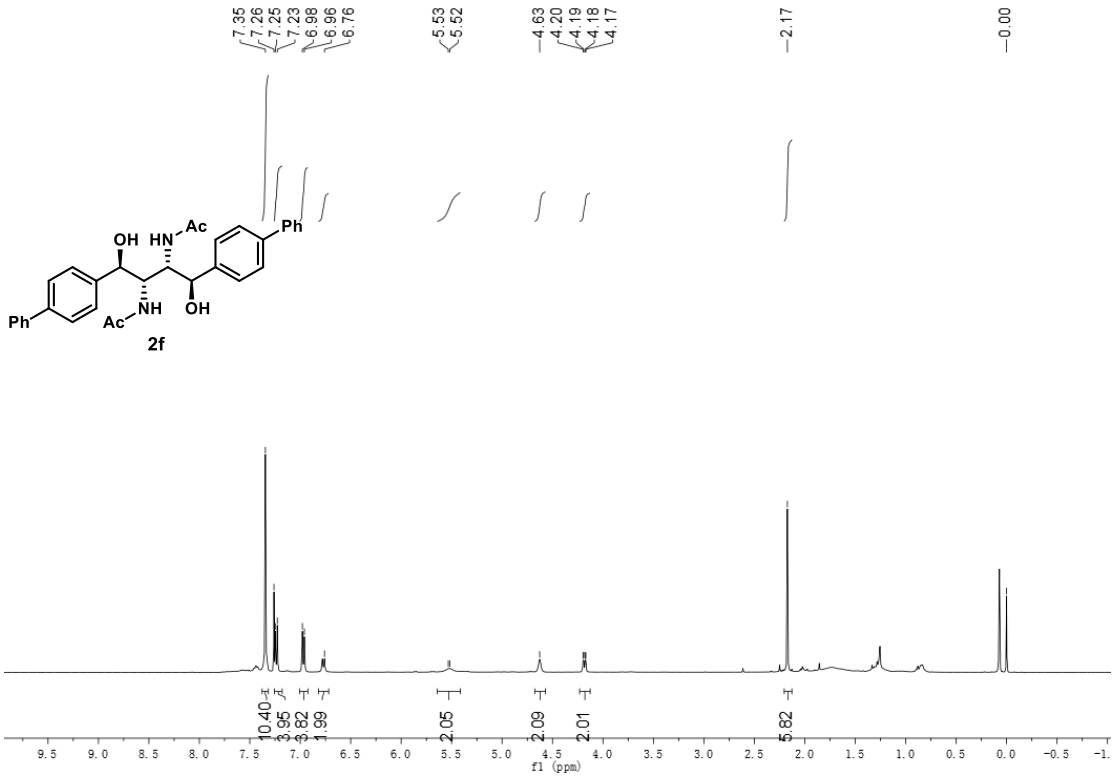
Supplementary Figure 81. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2d



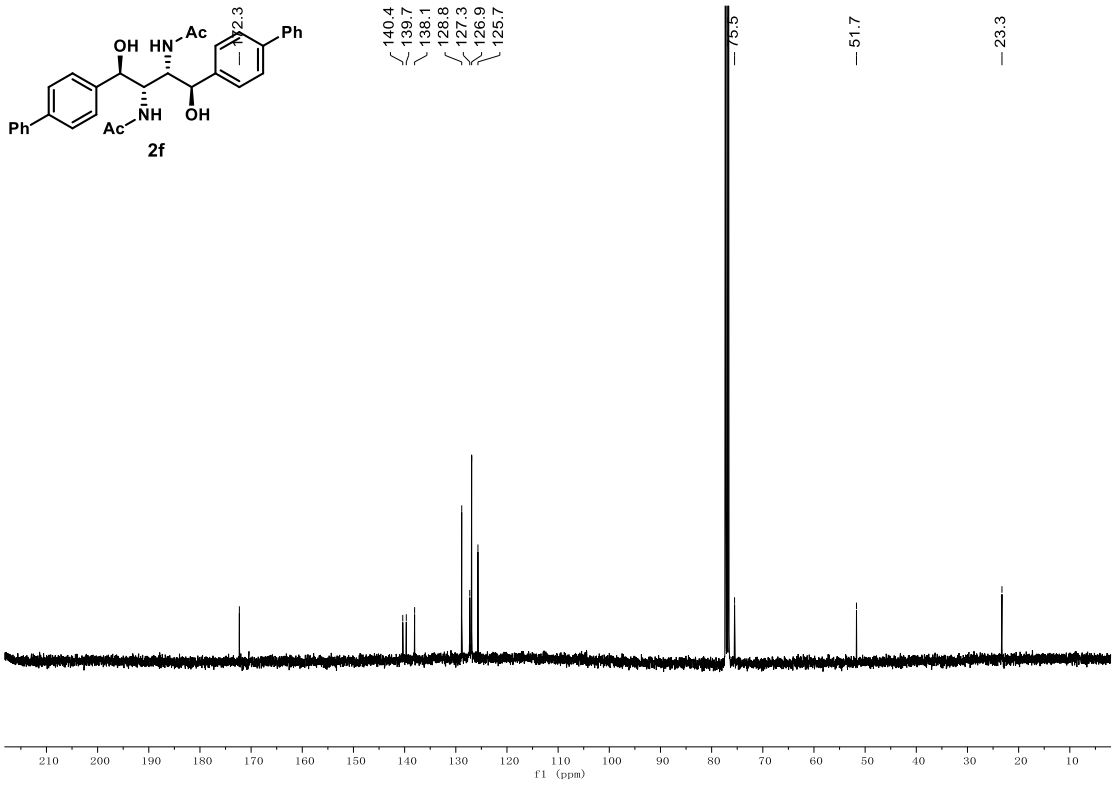
Supplementary Figure 82. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2e



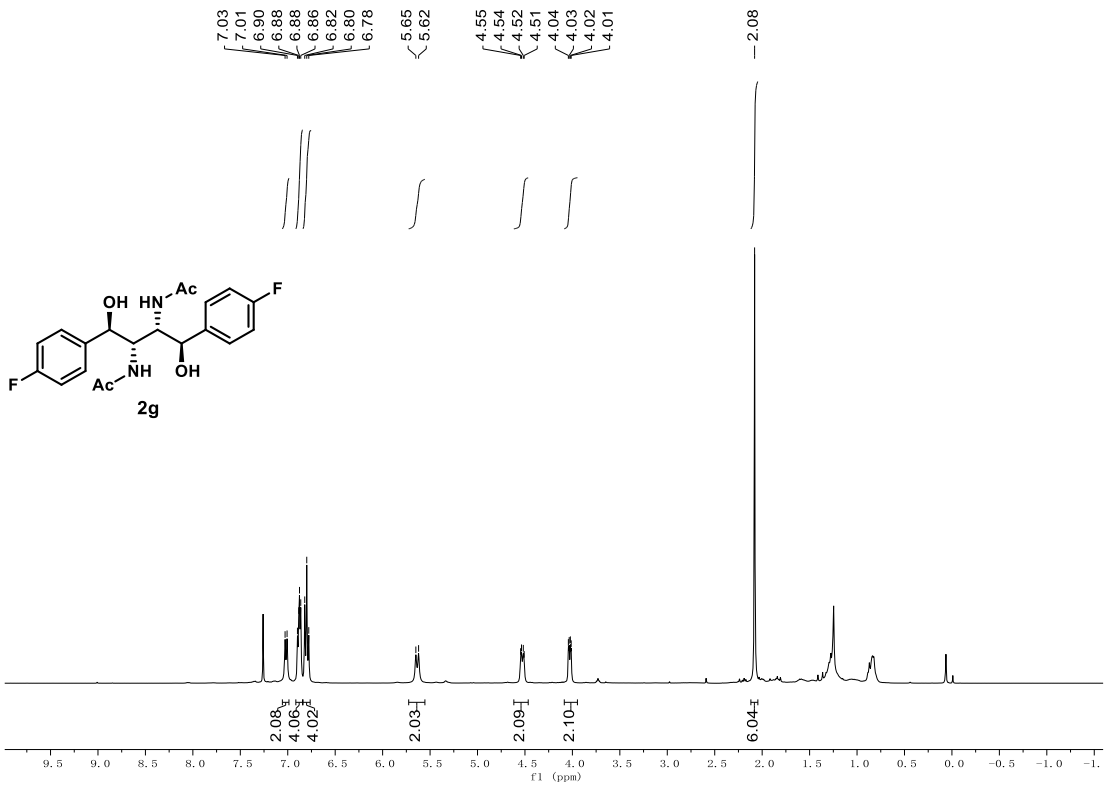
Supplementary Figure 83. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2e



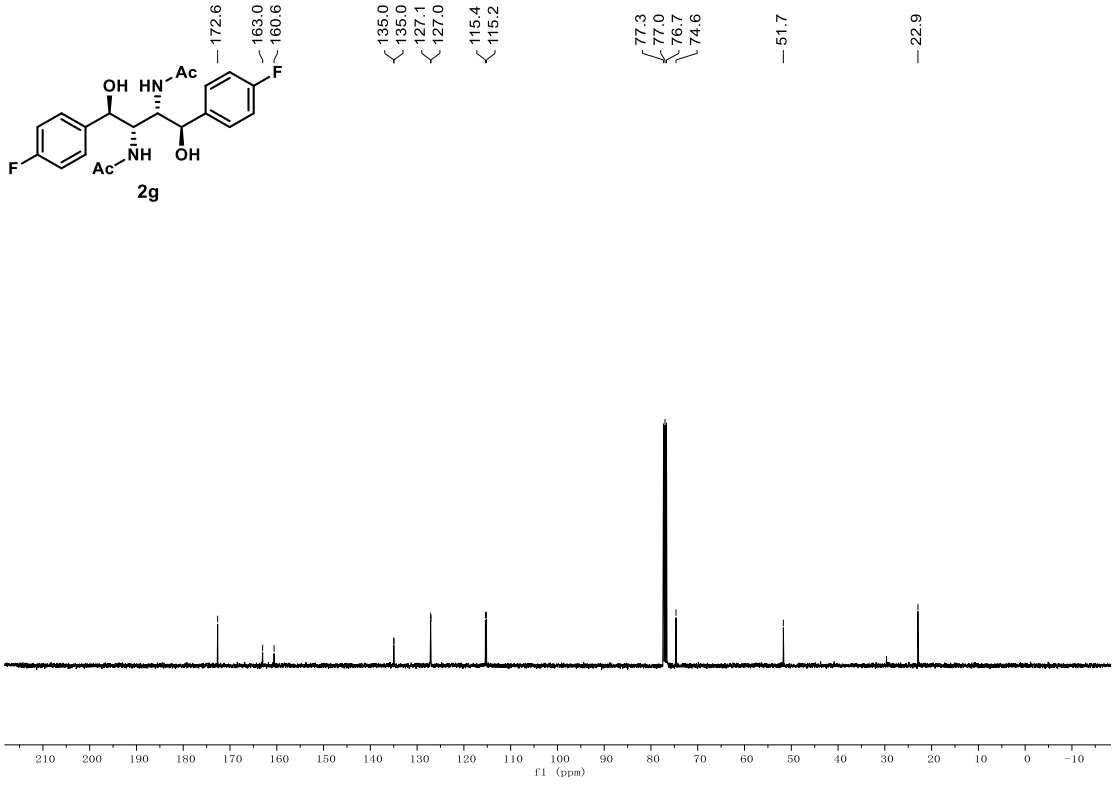
Supplementary Figure 84. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2f



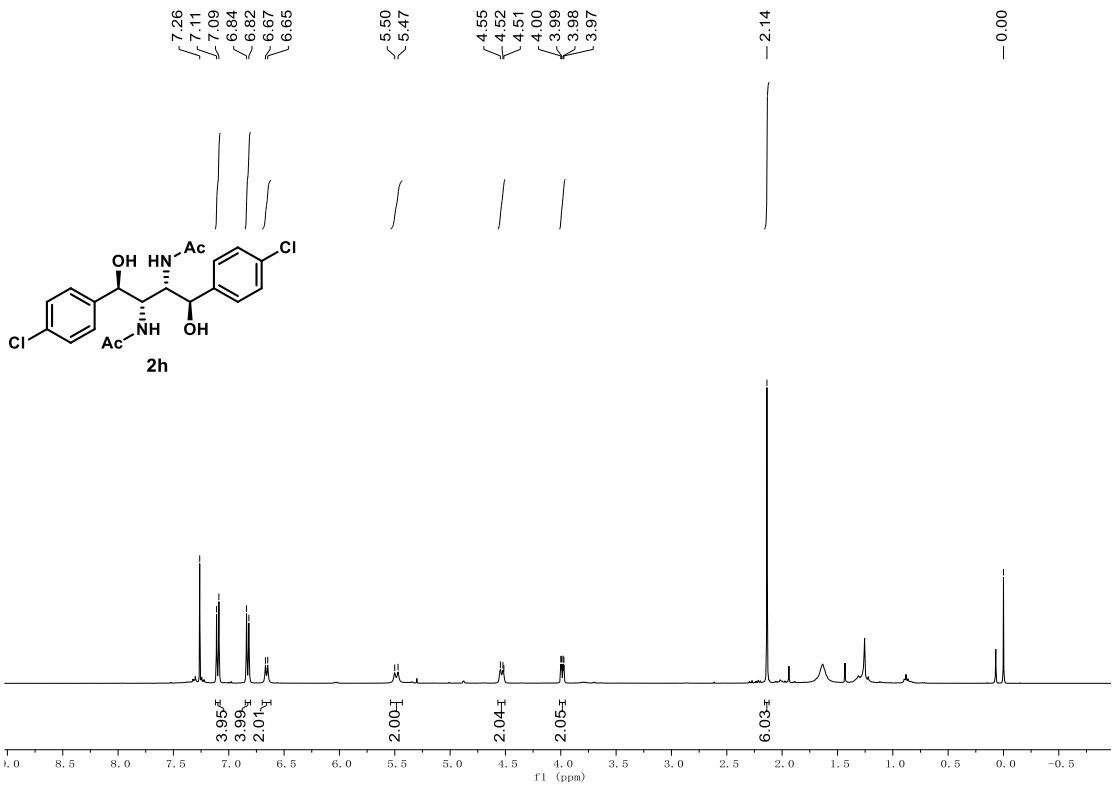
Supplementary Figure 85. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2f



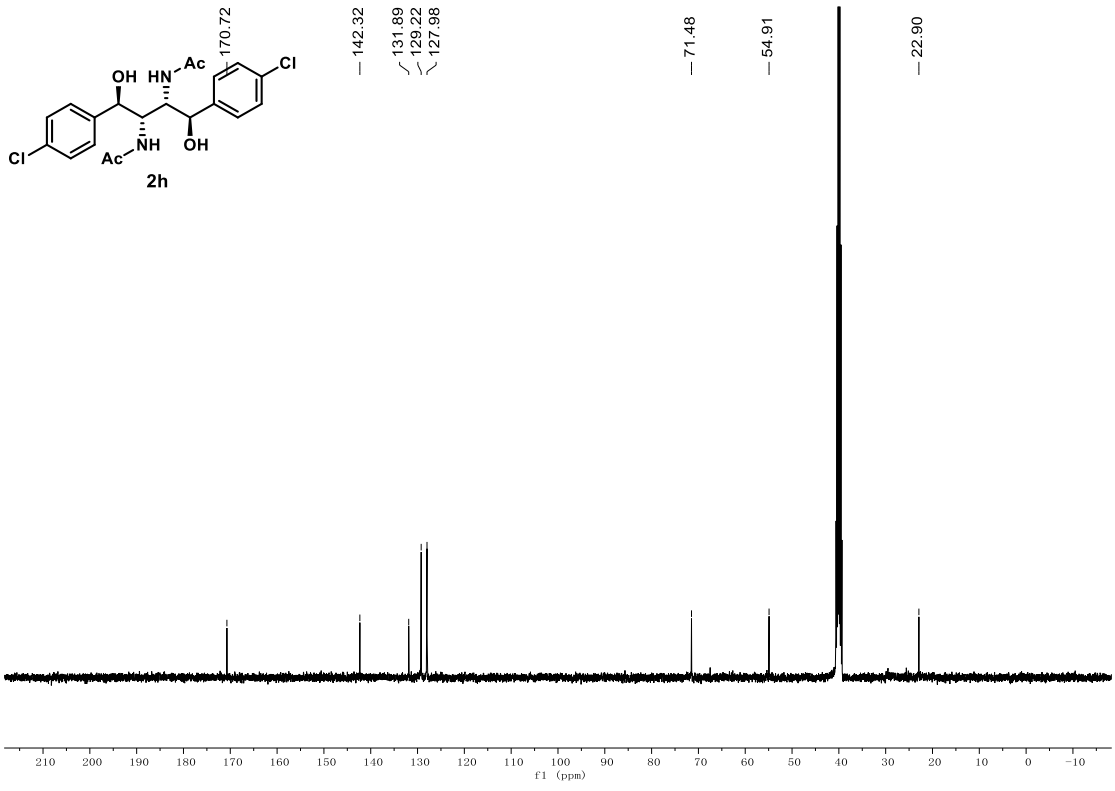
Supplementary Figure 86. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2g



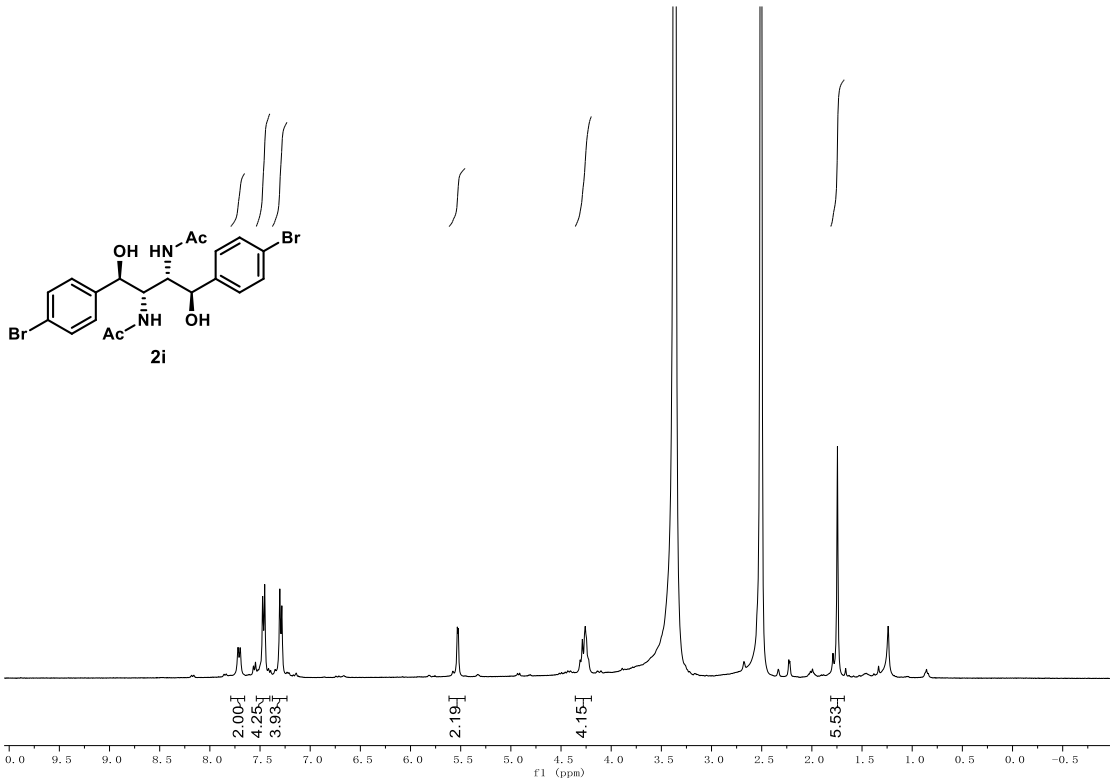
Supplementary Figure 87. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2g



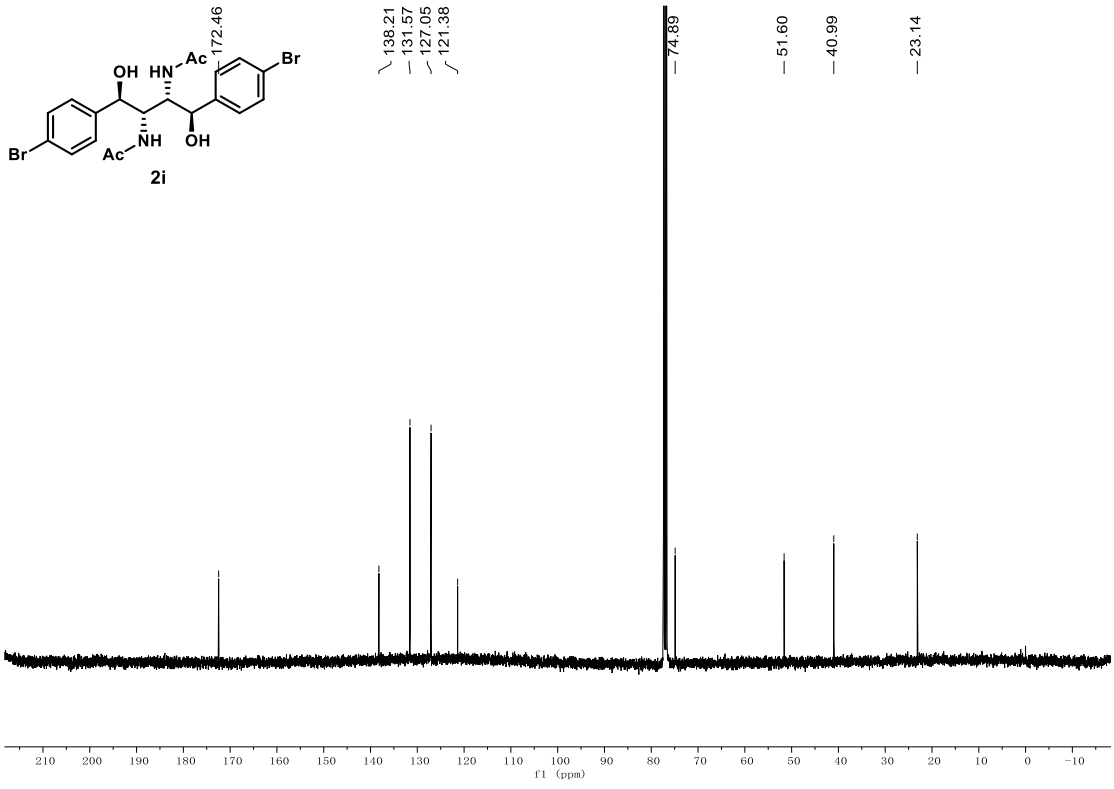
Supplementary Figure 88. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2h



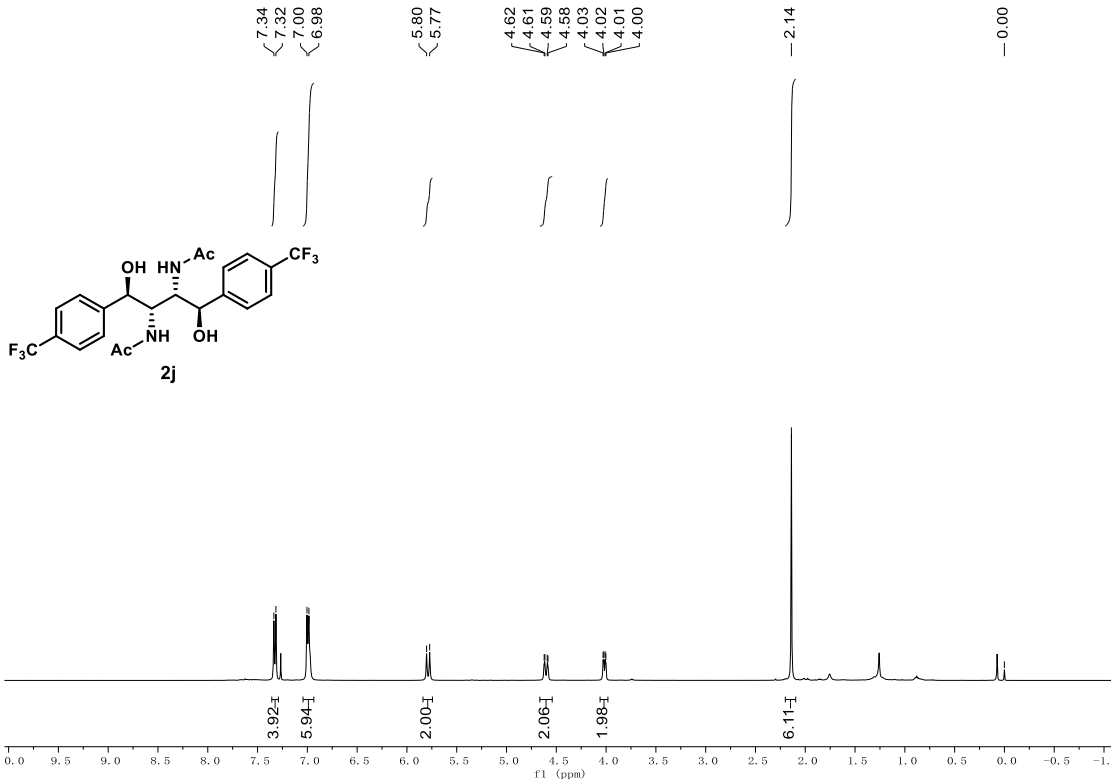
Supplementary Figure 89. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2h



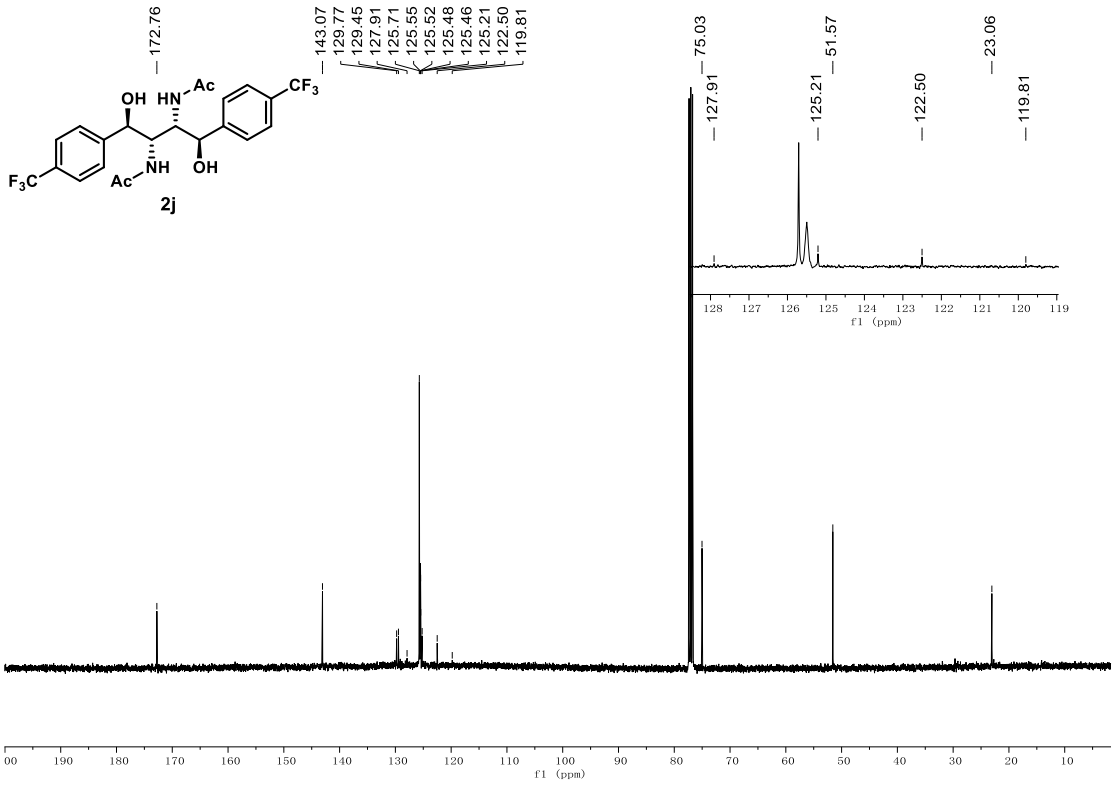
Supplementary Figure 90. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2i



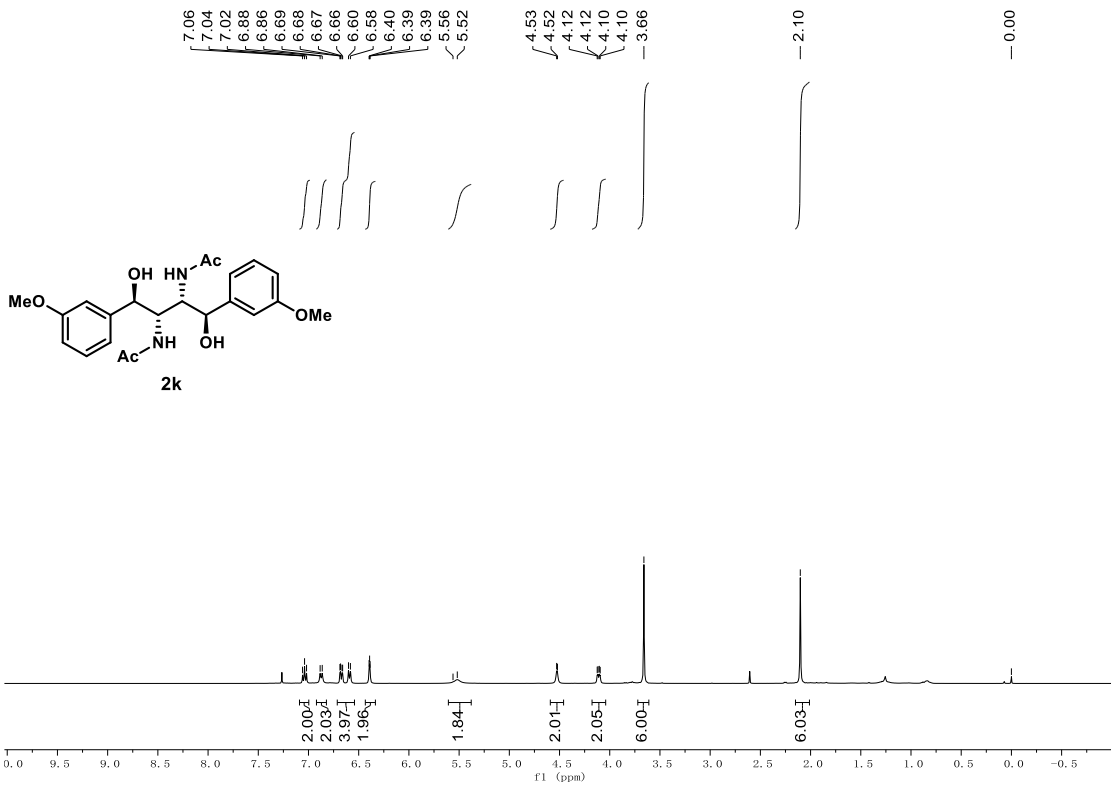
Supplementary Figure 91. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2i



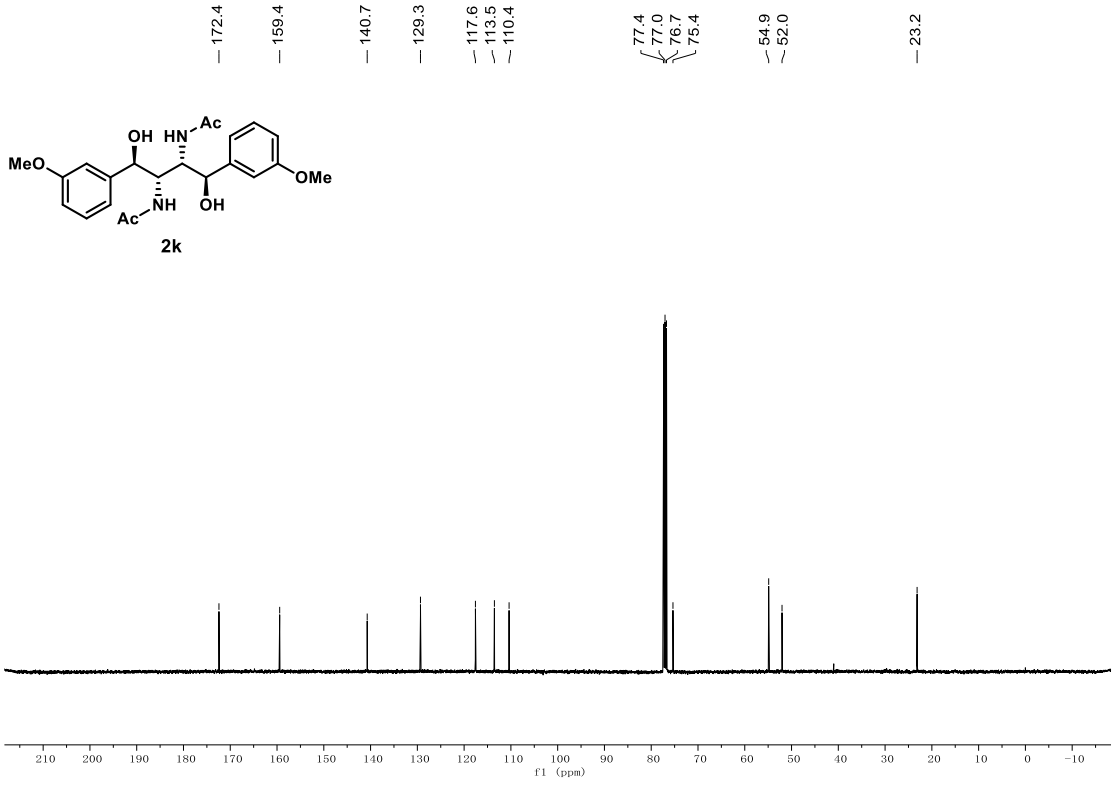
Supplementary Figure 92. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2j



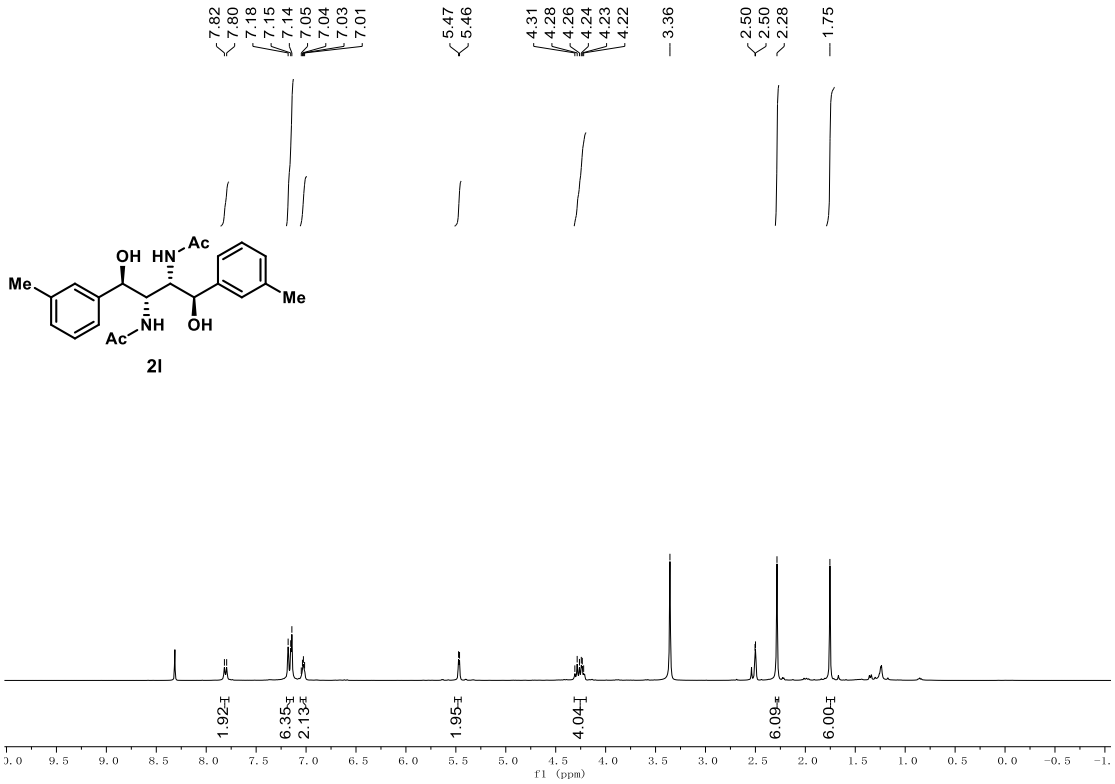
Supplementary Figure 93. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2j



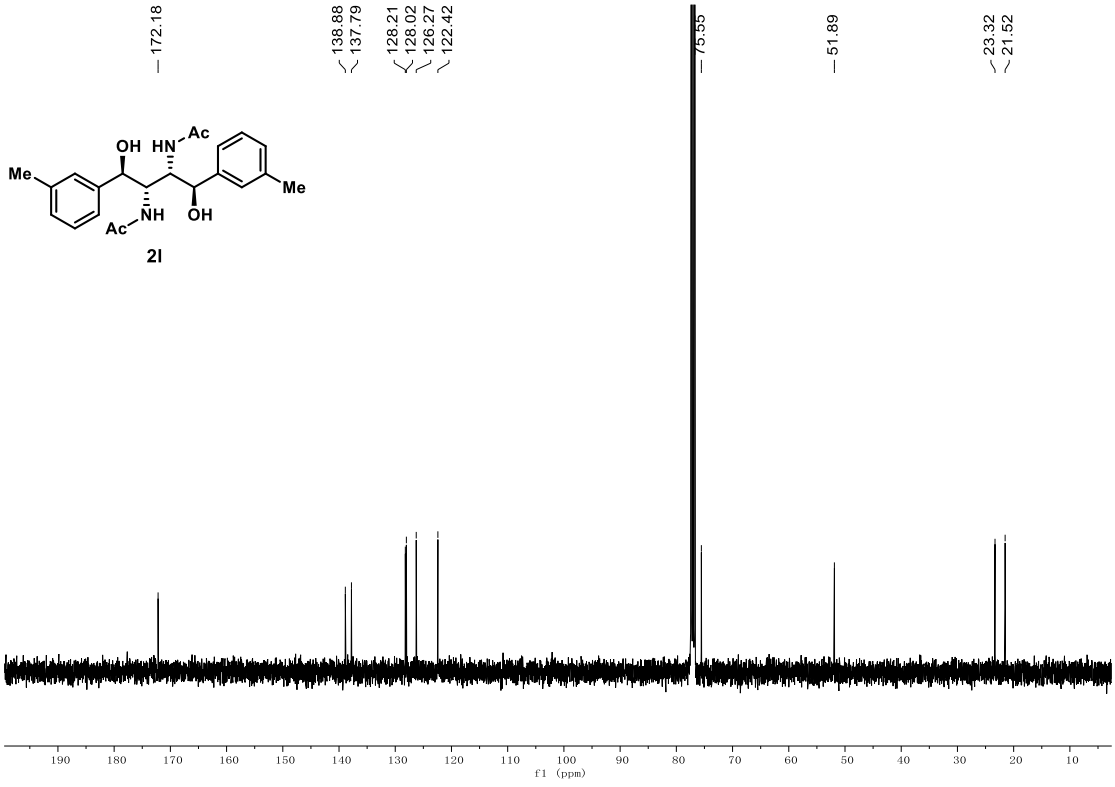
Supplementary Figure 94. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2k



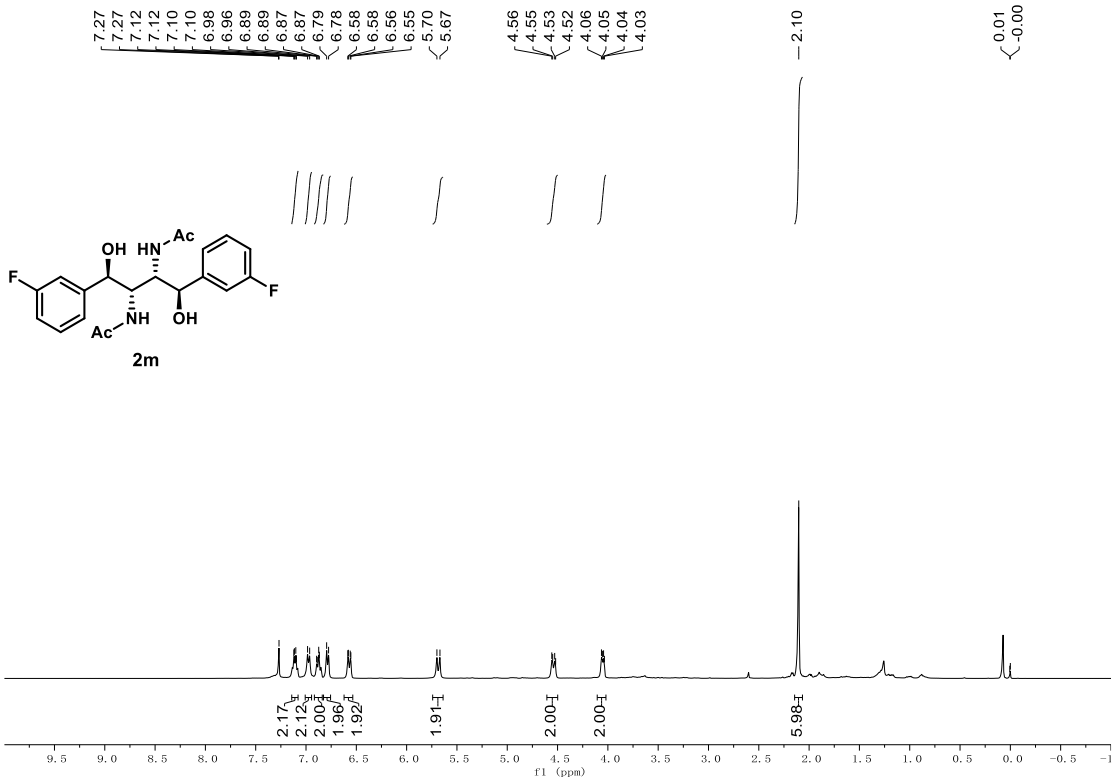
Supplementary Figure 95. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2k



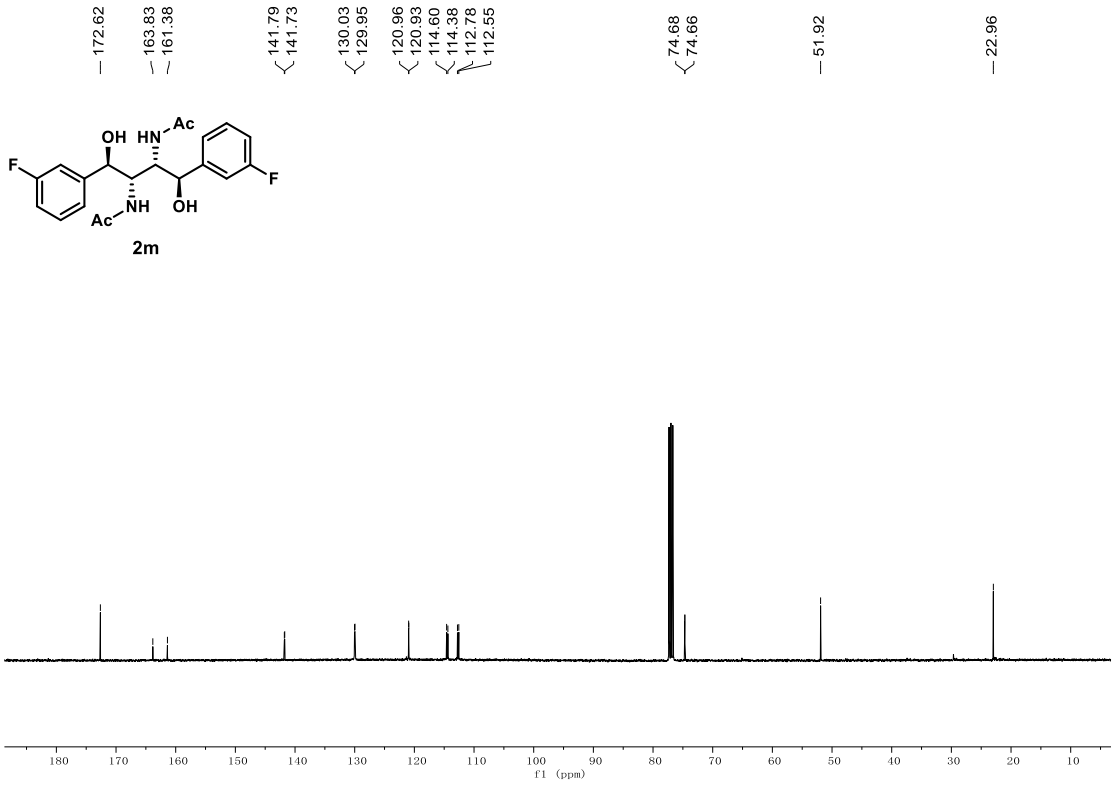
Supplementary Figure 96. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2l



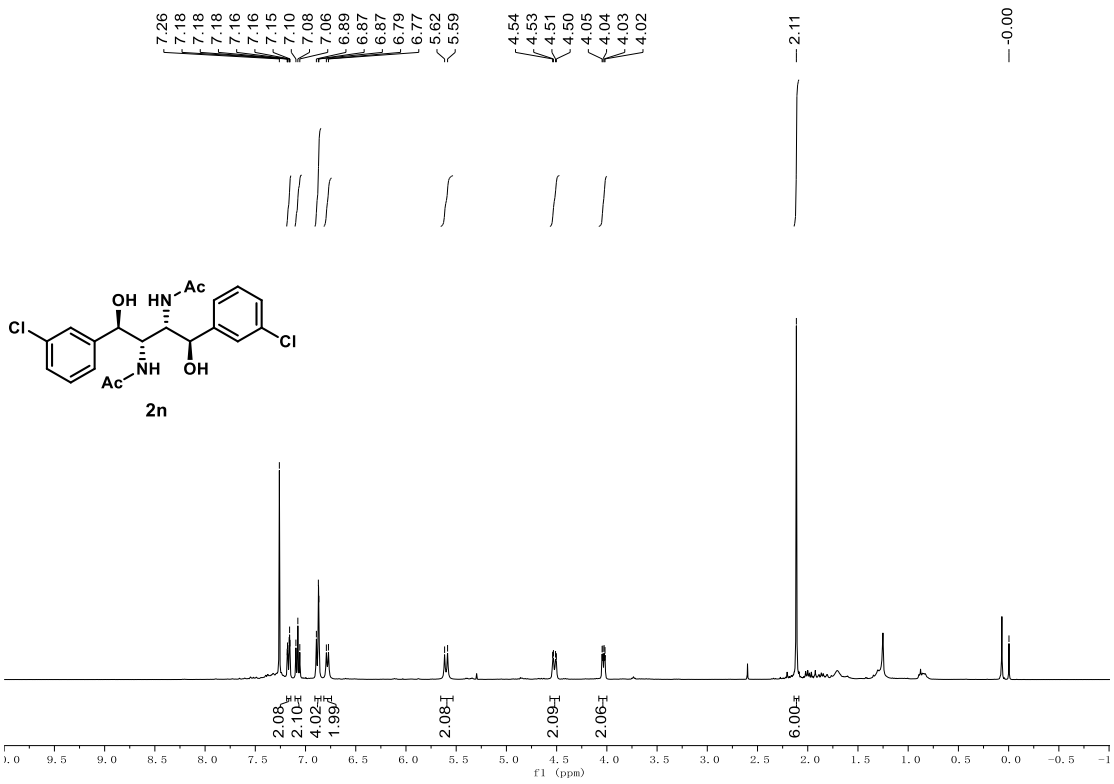
Supplementary Figure 97. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2l



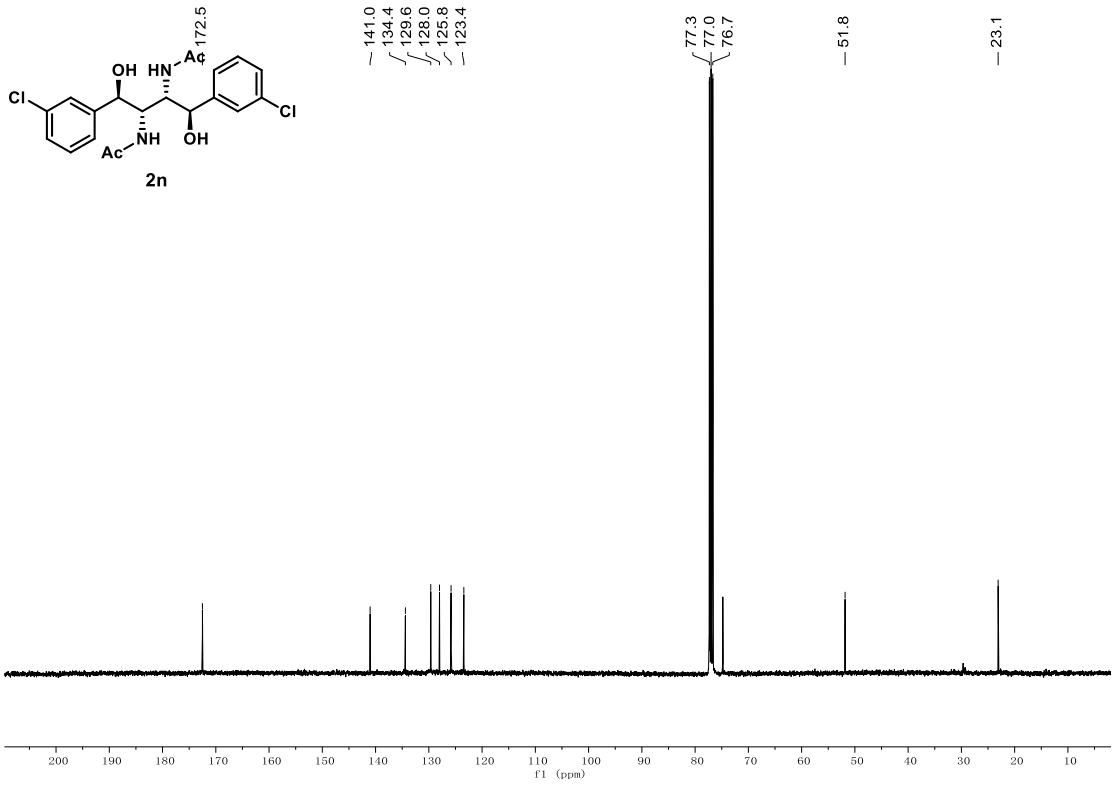
Supplementary Figure 98. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2m



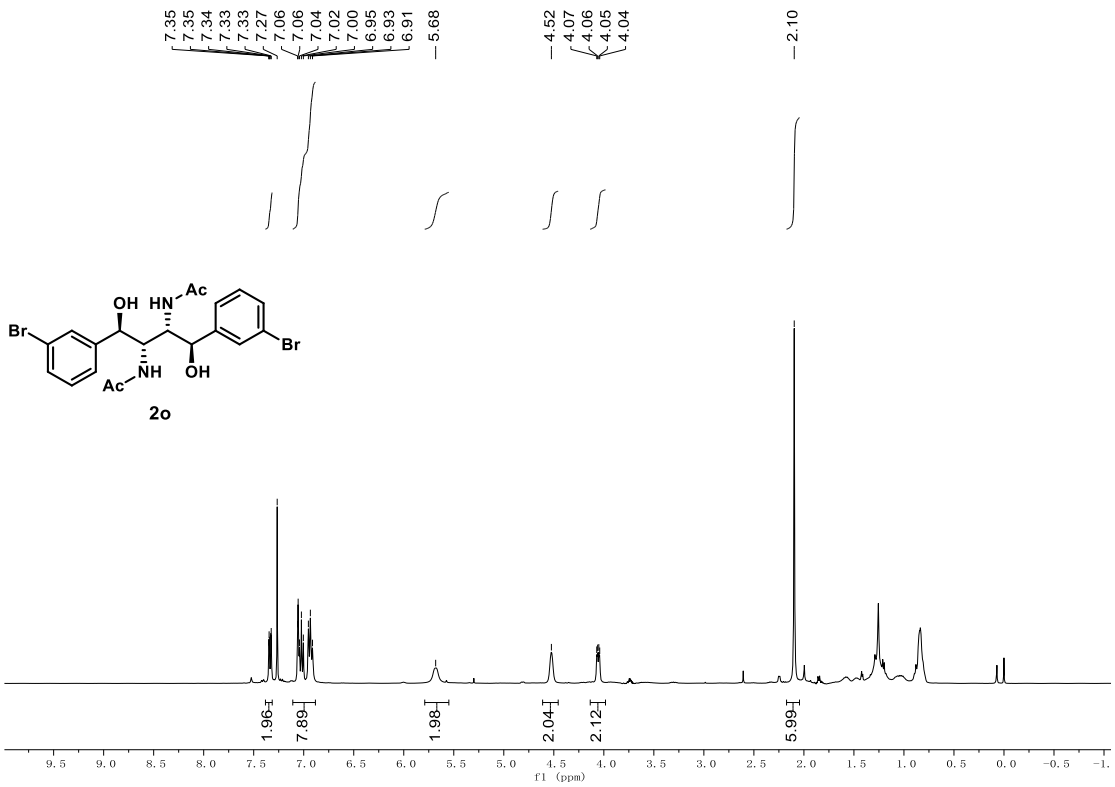
Supplementary Figure 99. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2m



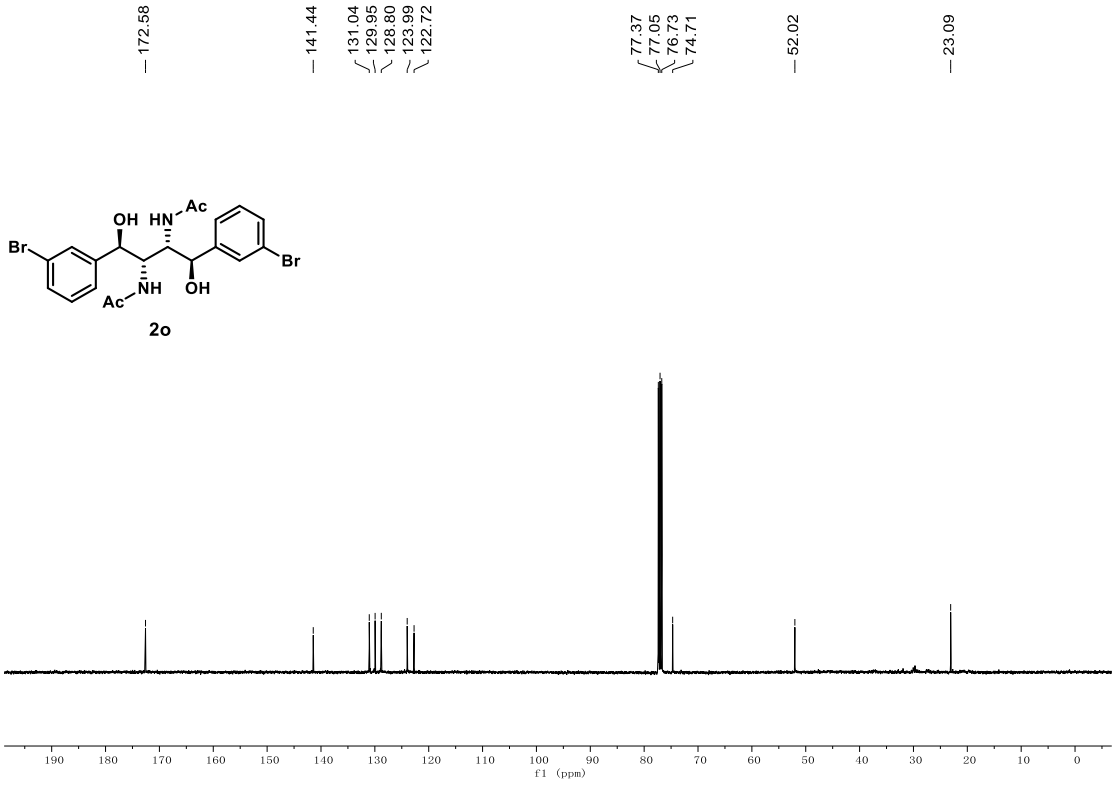
Supplementary Figure 100. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2n



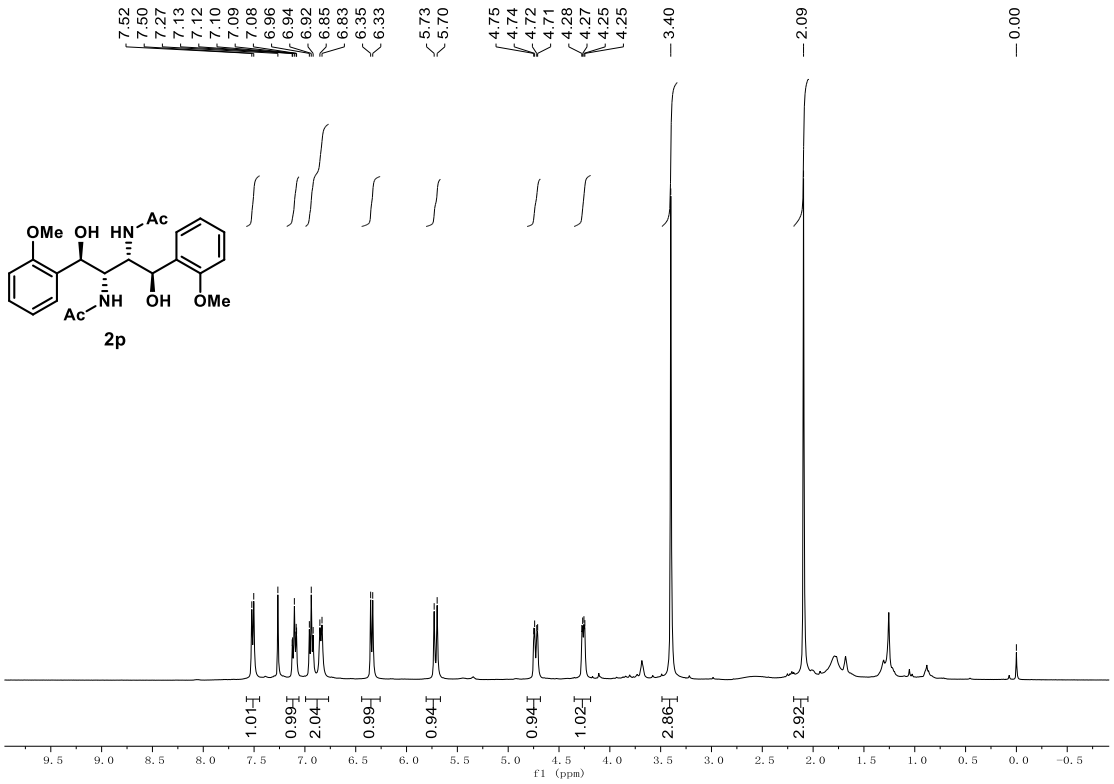
Supplementary Figure 101. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2n



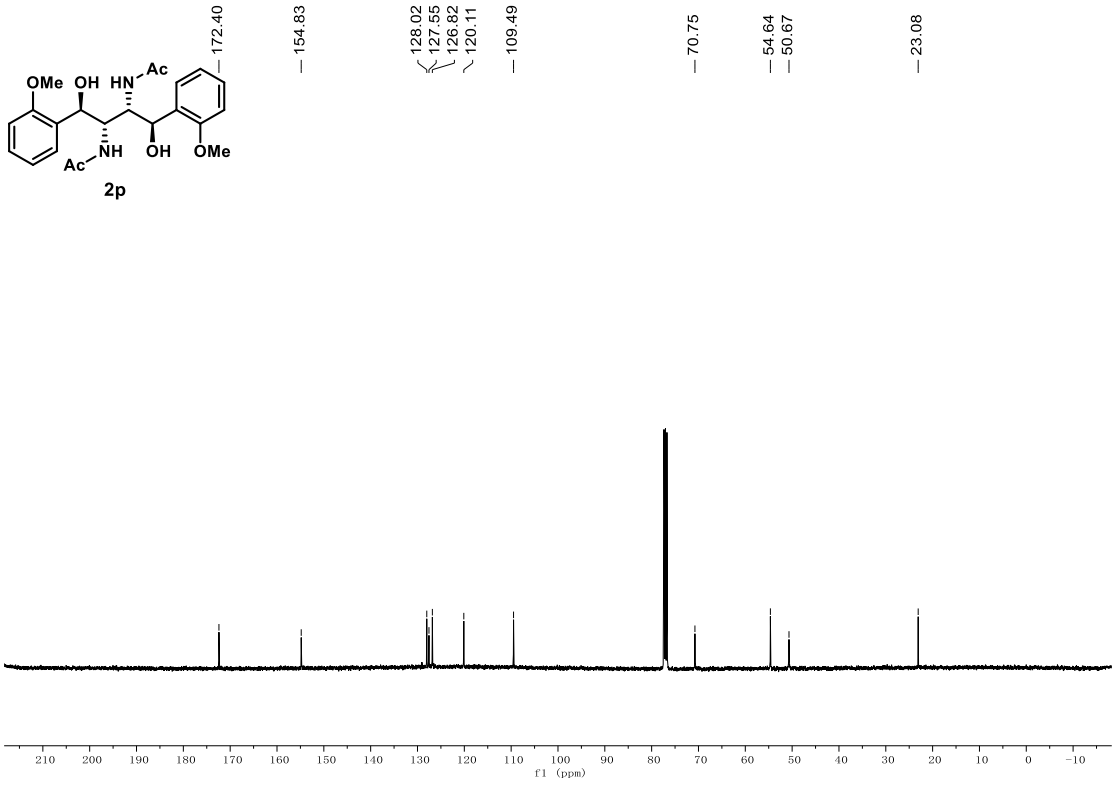
Supplementary Figure 102. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2o



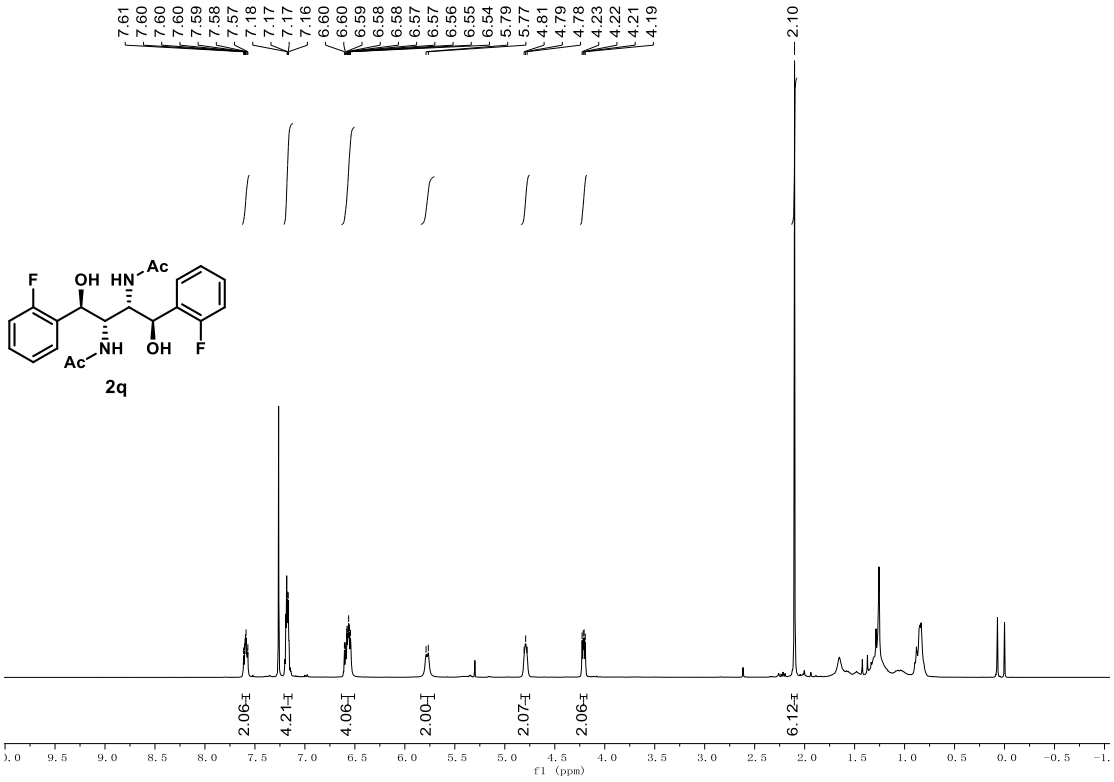
Supplementary Figure 103. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2o



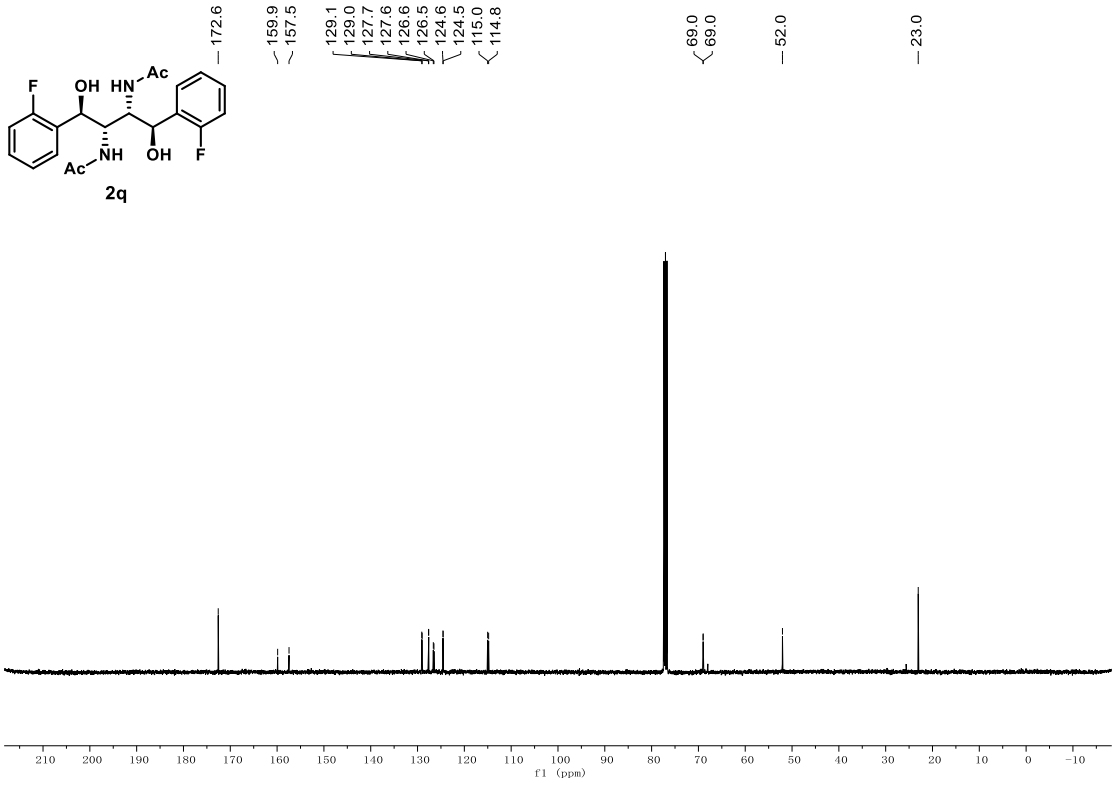
Supplementary Figure 104. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2p



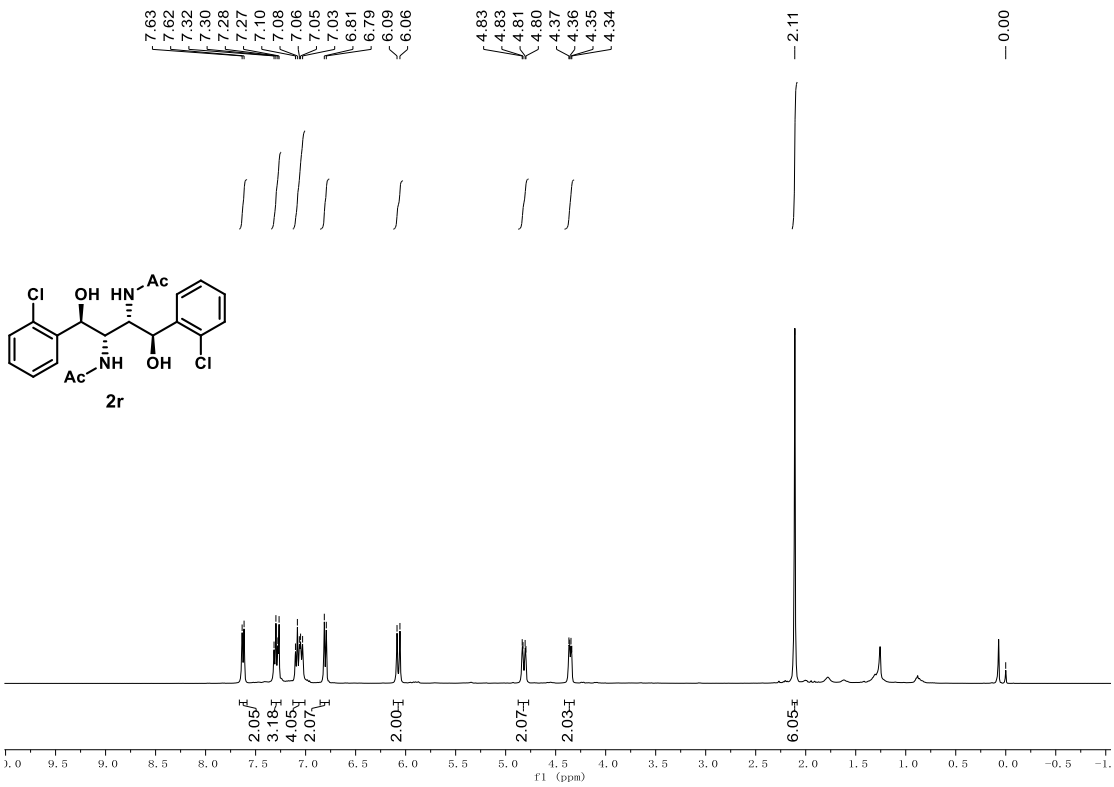
Supplementary Figure 105. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2p



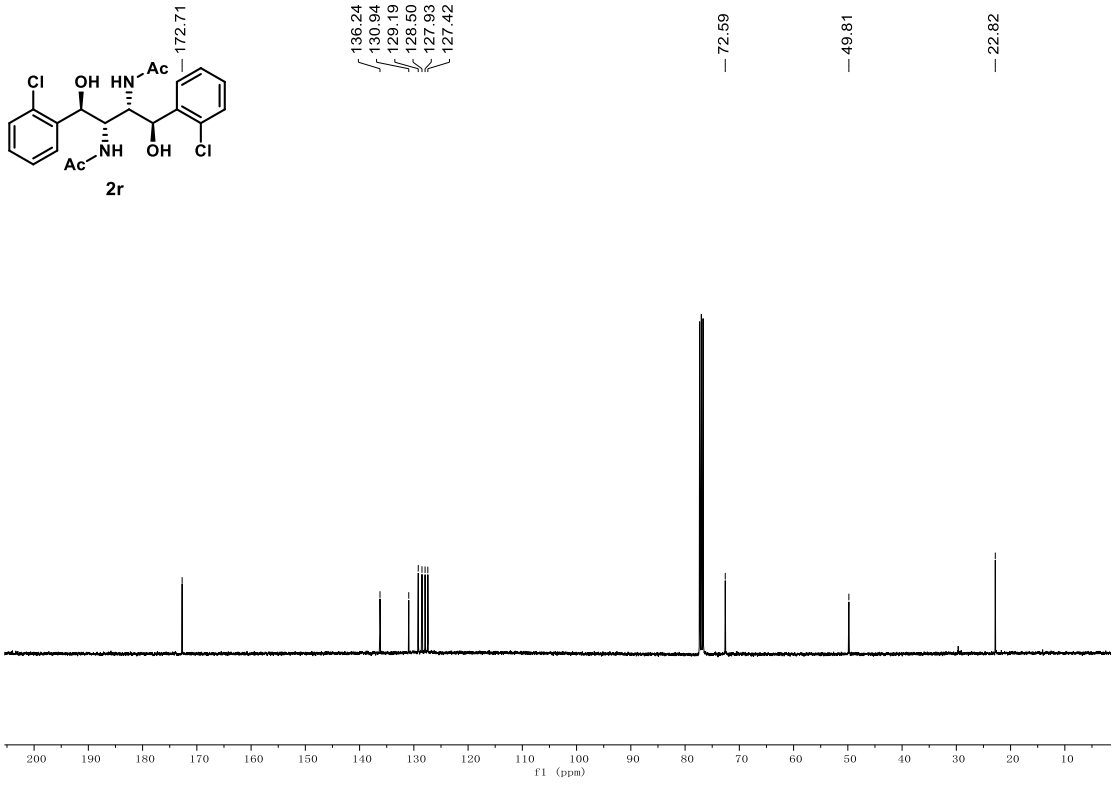
Supplementary Figure 106. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2q



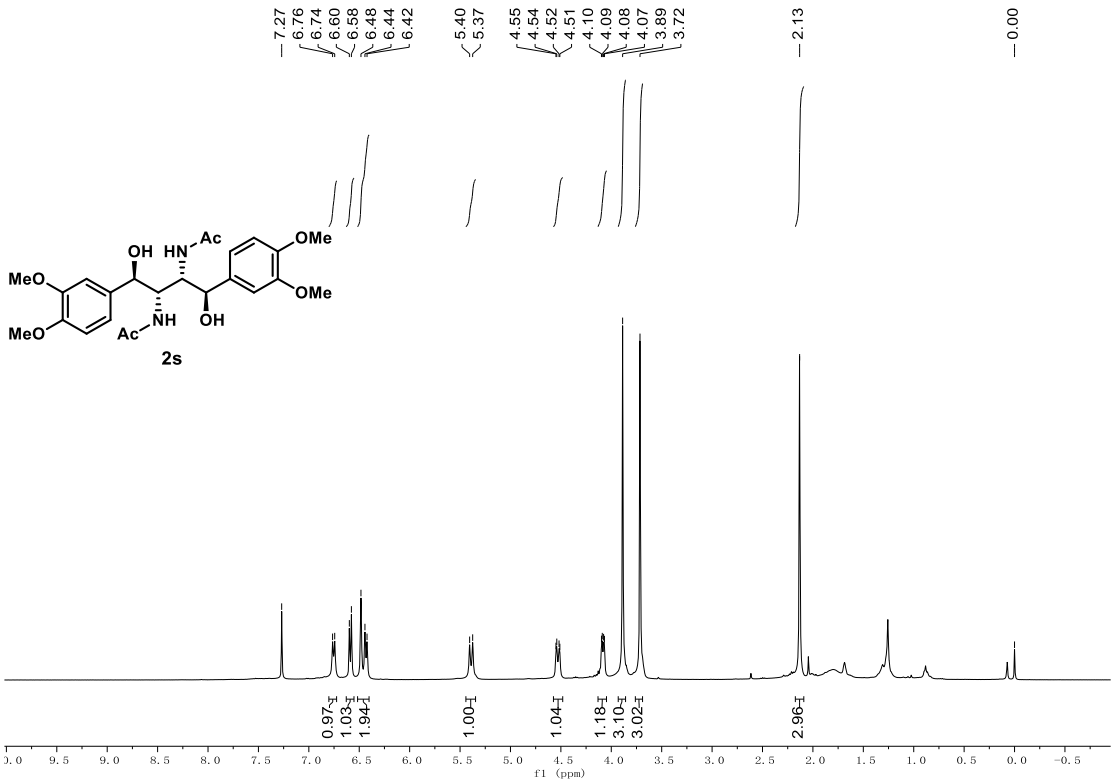
Supplementary Figure 107. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2q



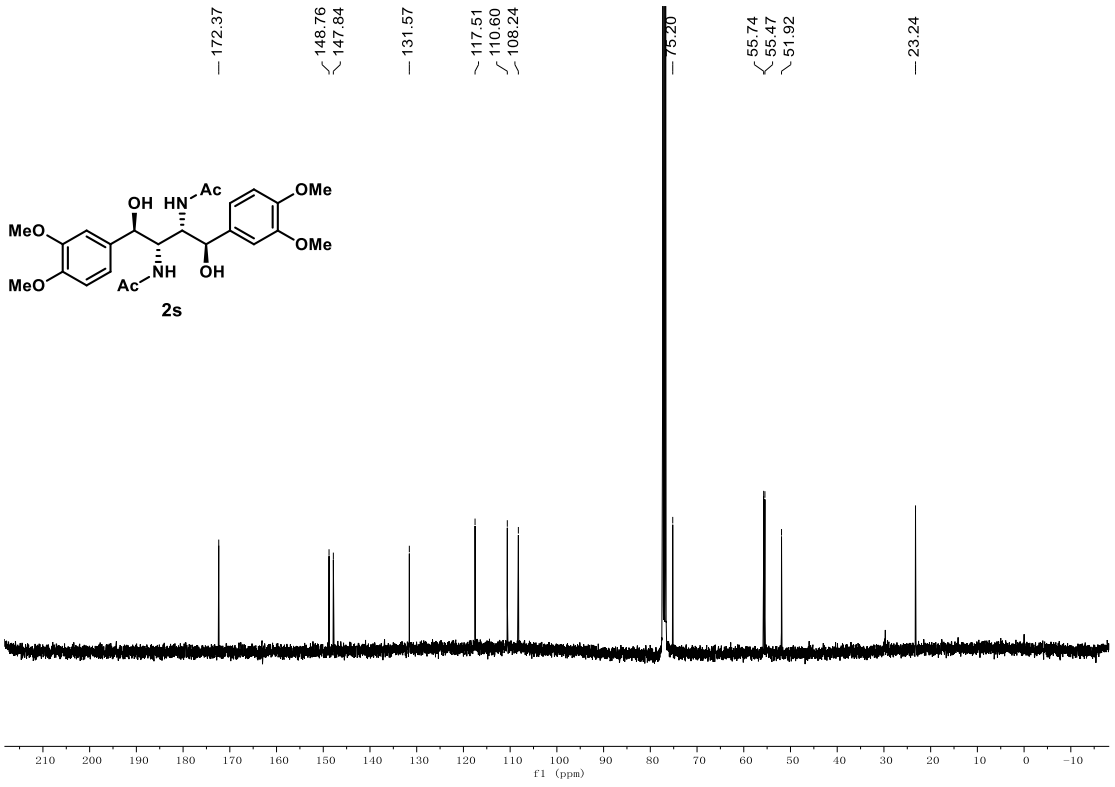
Supplementary Figure 108. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2r



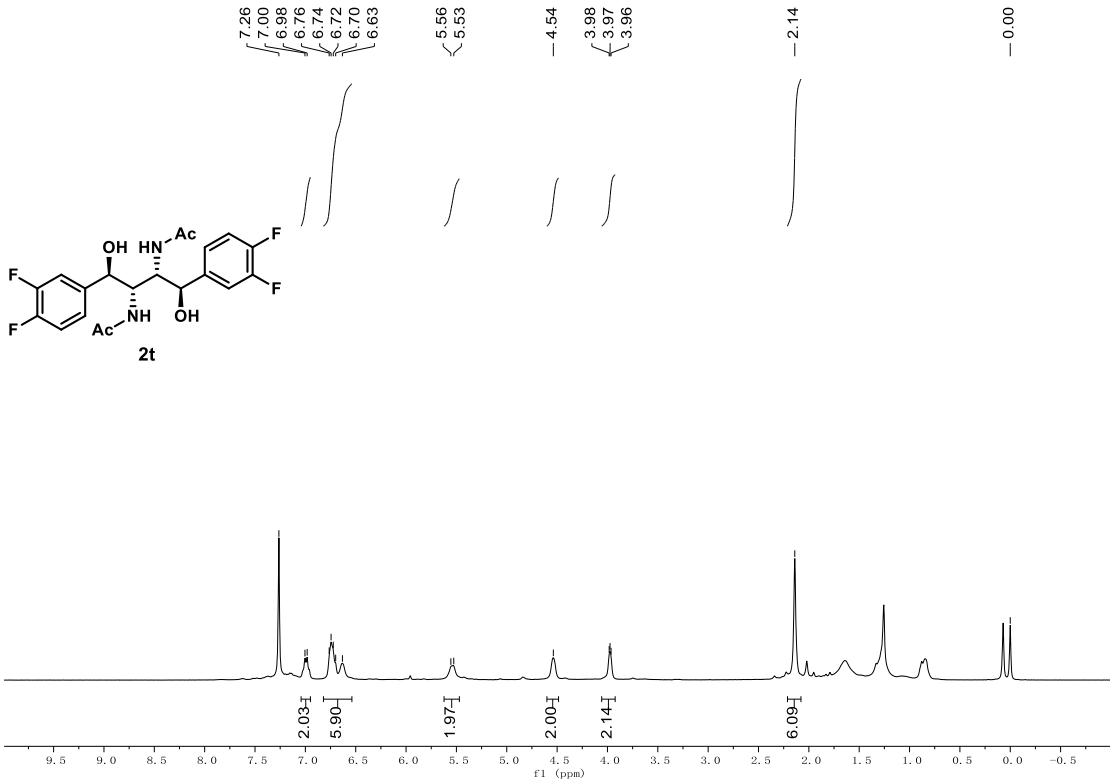
Supplementary Figure 109. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2r



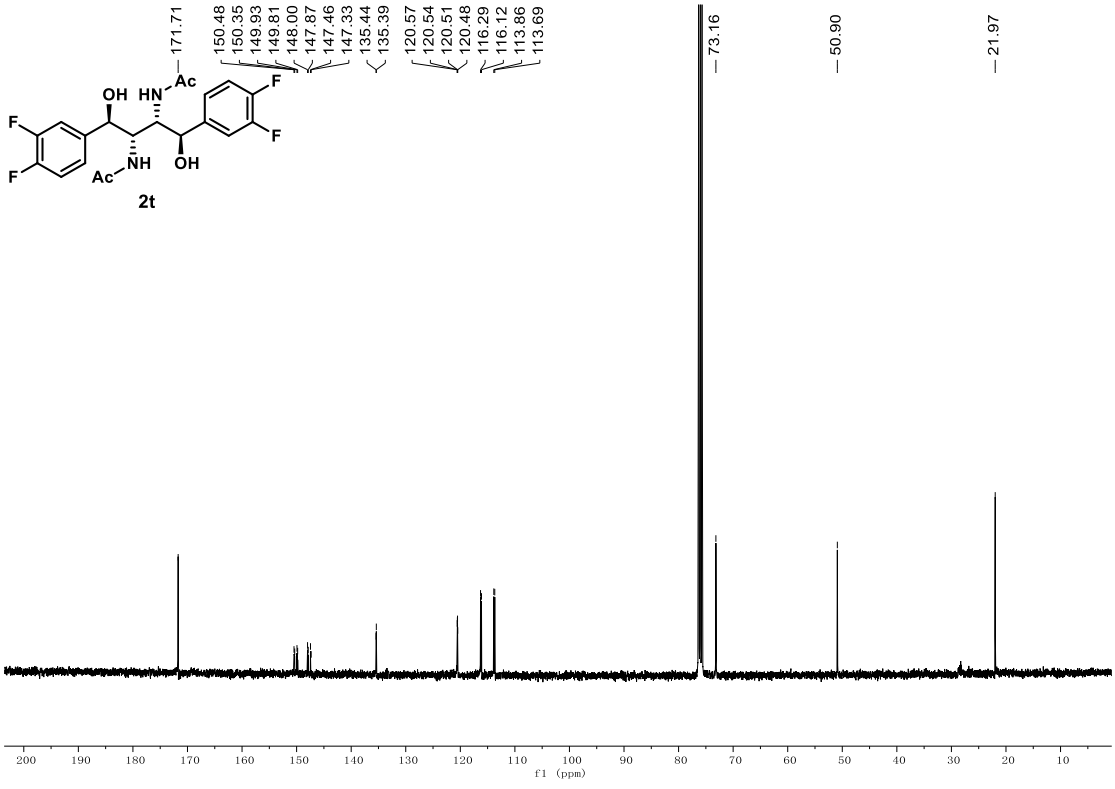
Supplementary Figure 110. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2s



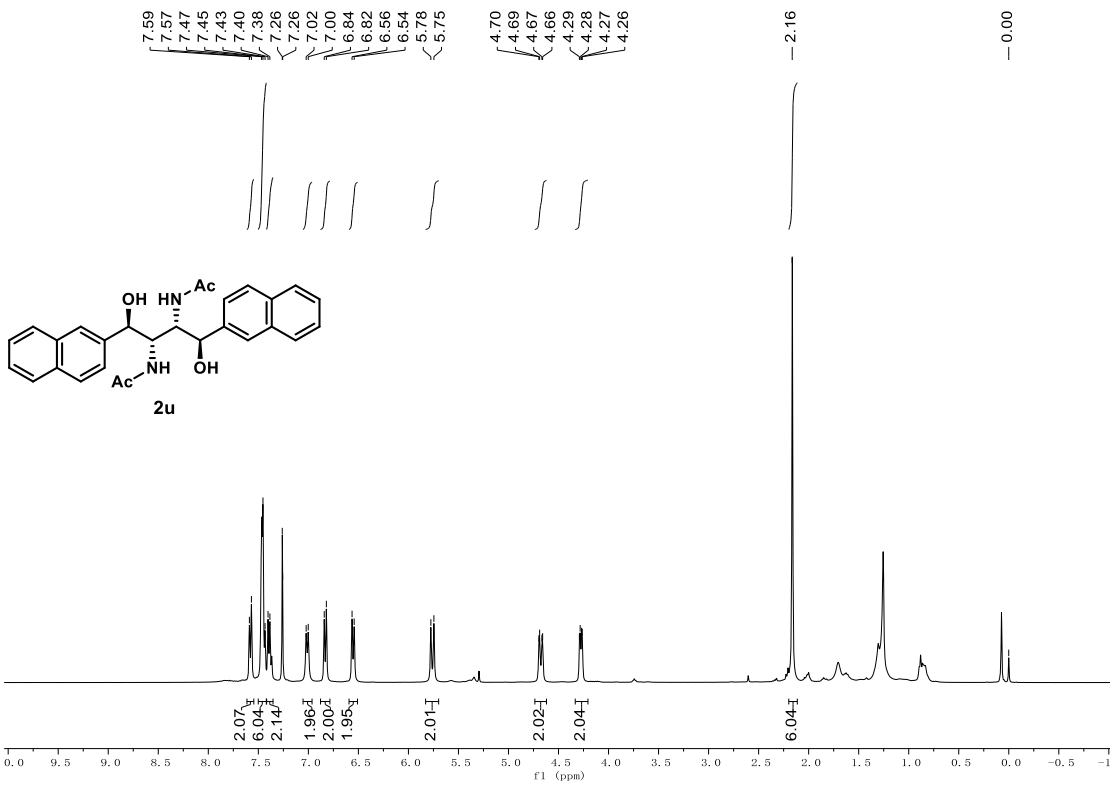
Supplementary Figure 111. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2s



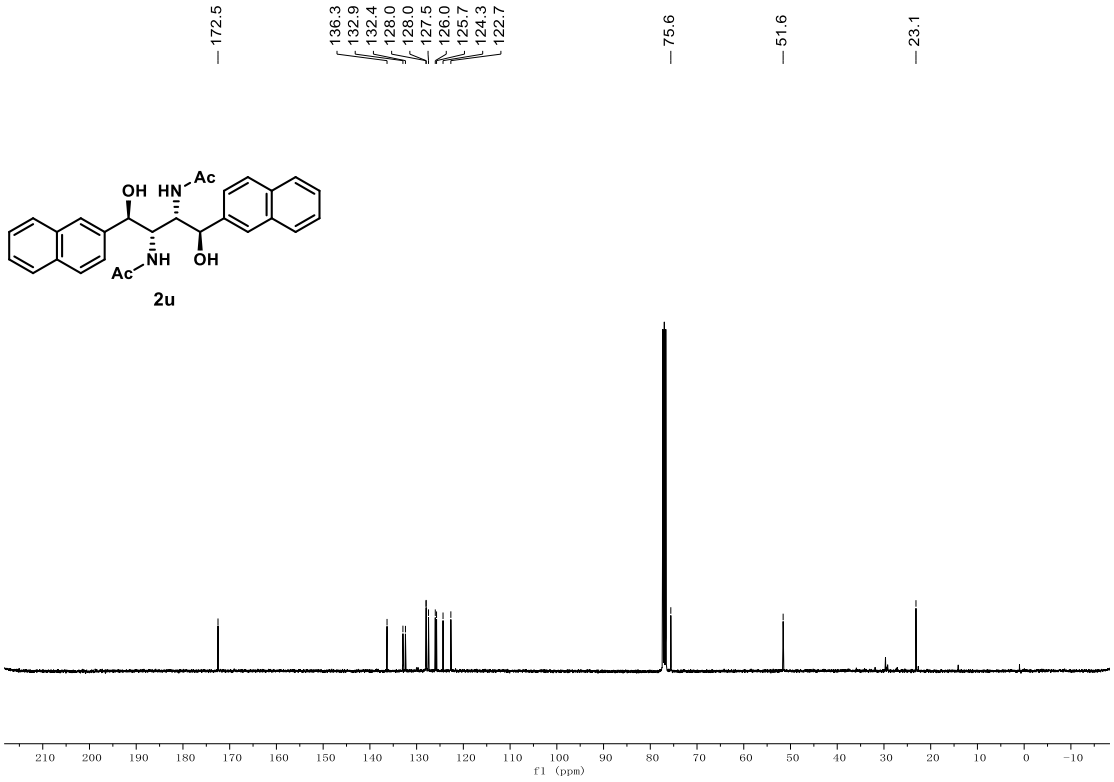
Supplementary Figure 112. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2t



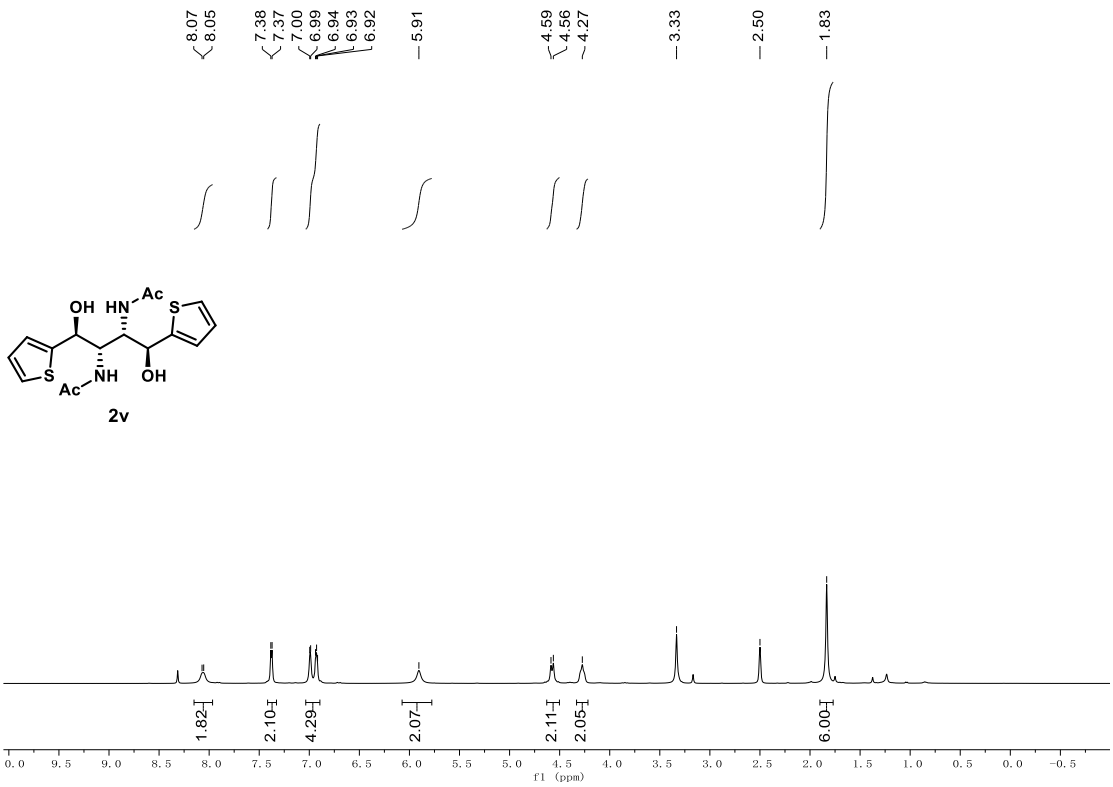
Supplementary Figure 113. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2t



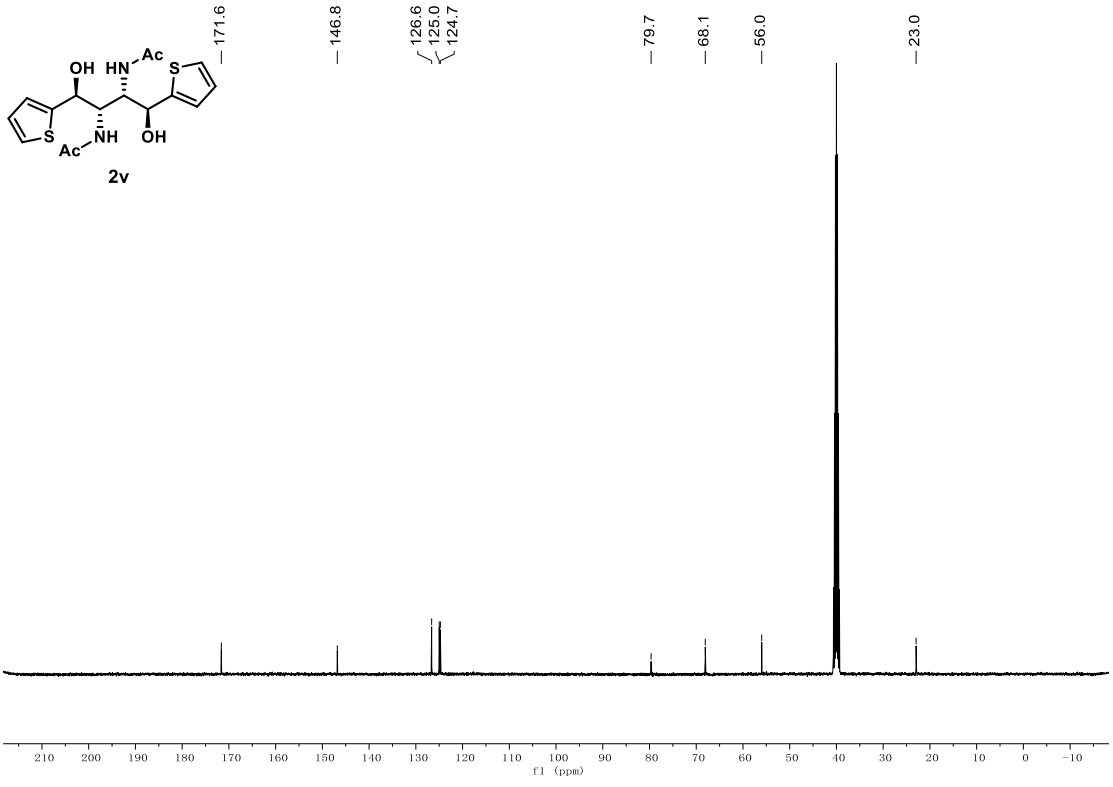
Supplementary Figure 114. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2u



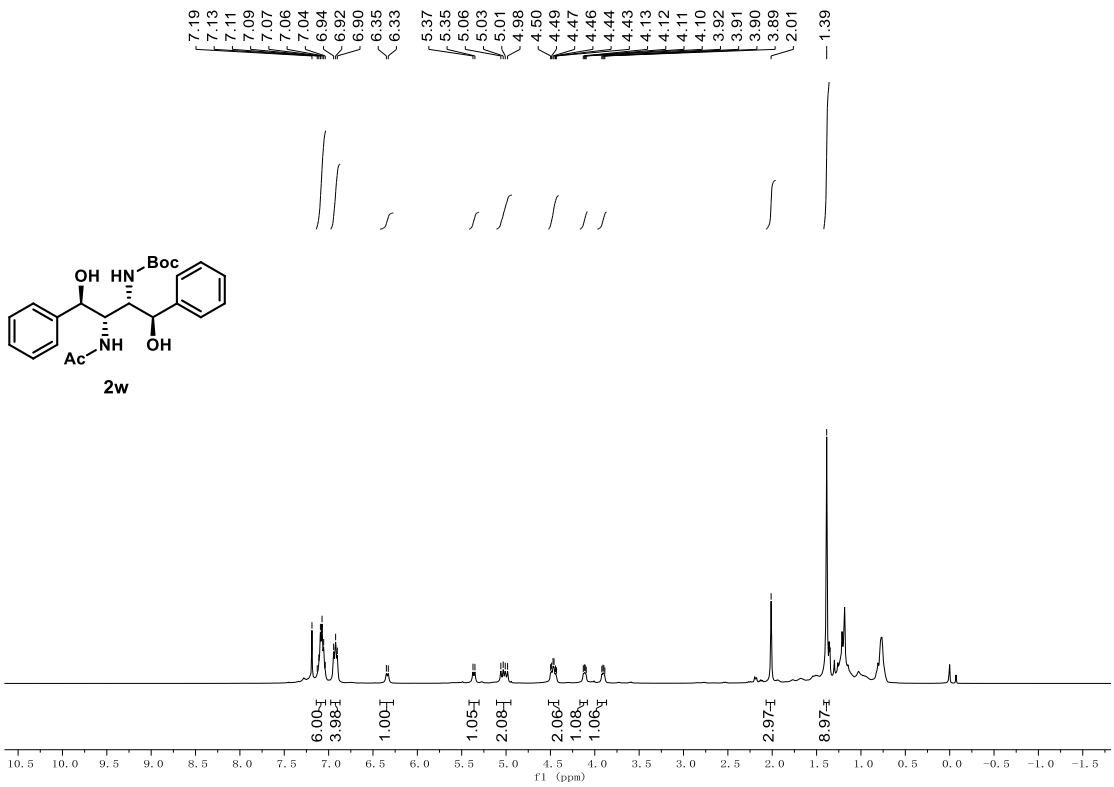
Supplementary Figure 115. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2u



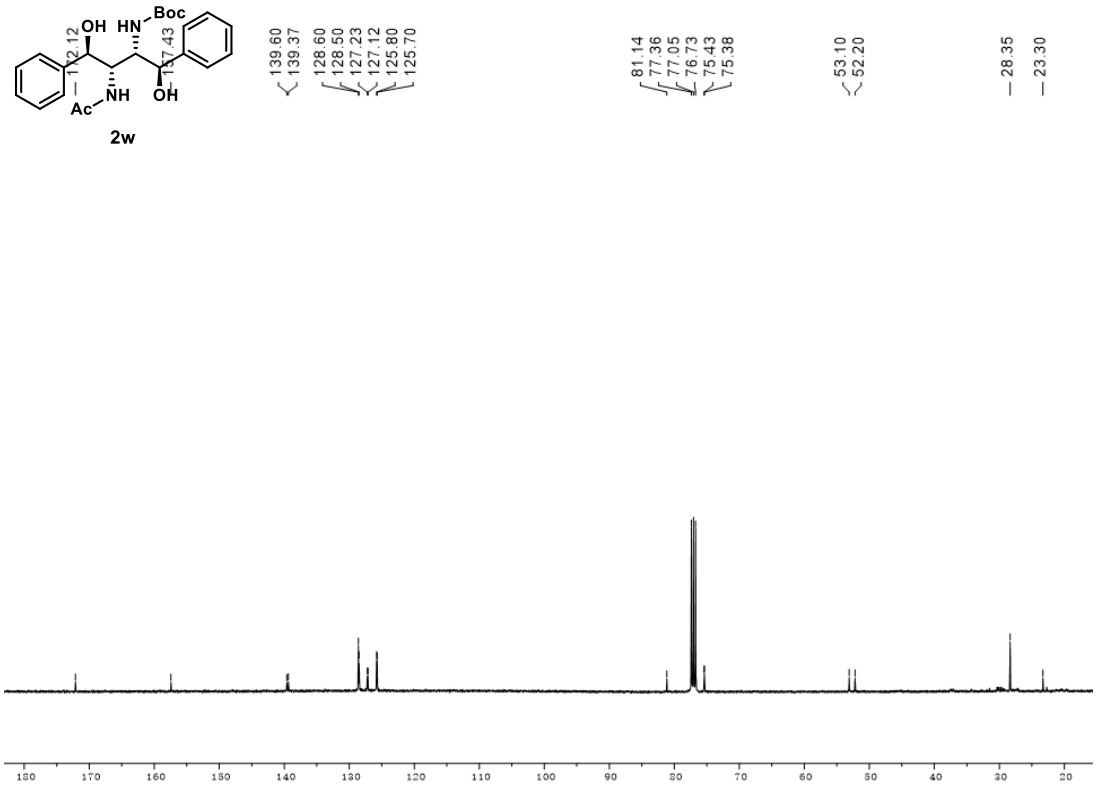
Supplementary Figure 116. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2v



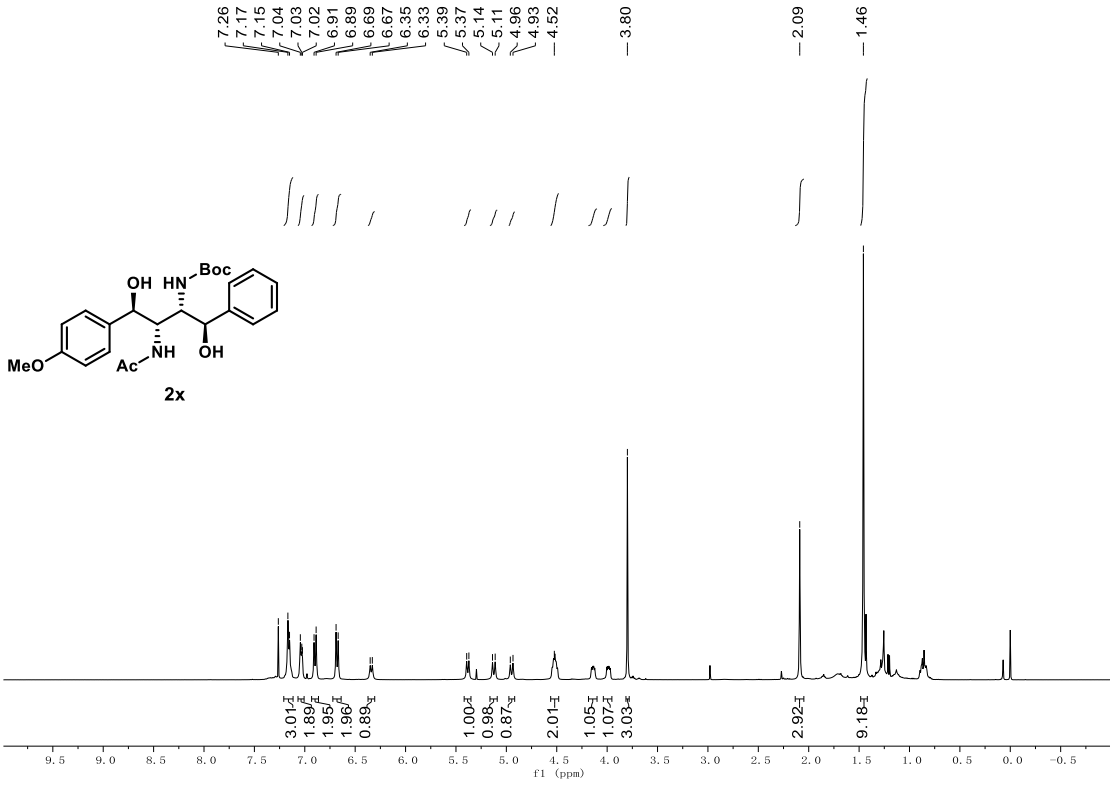
Supplementary Figure 117. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2v



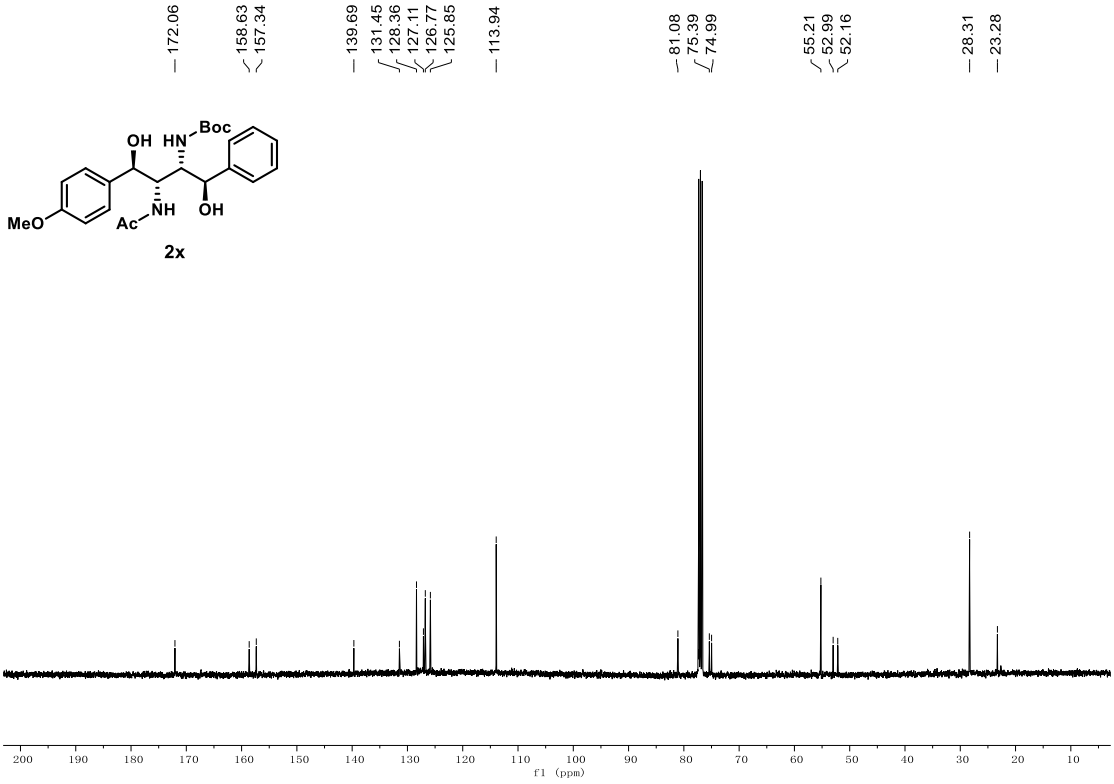
Supplementary Figure 118. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2w



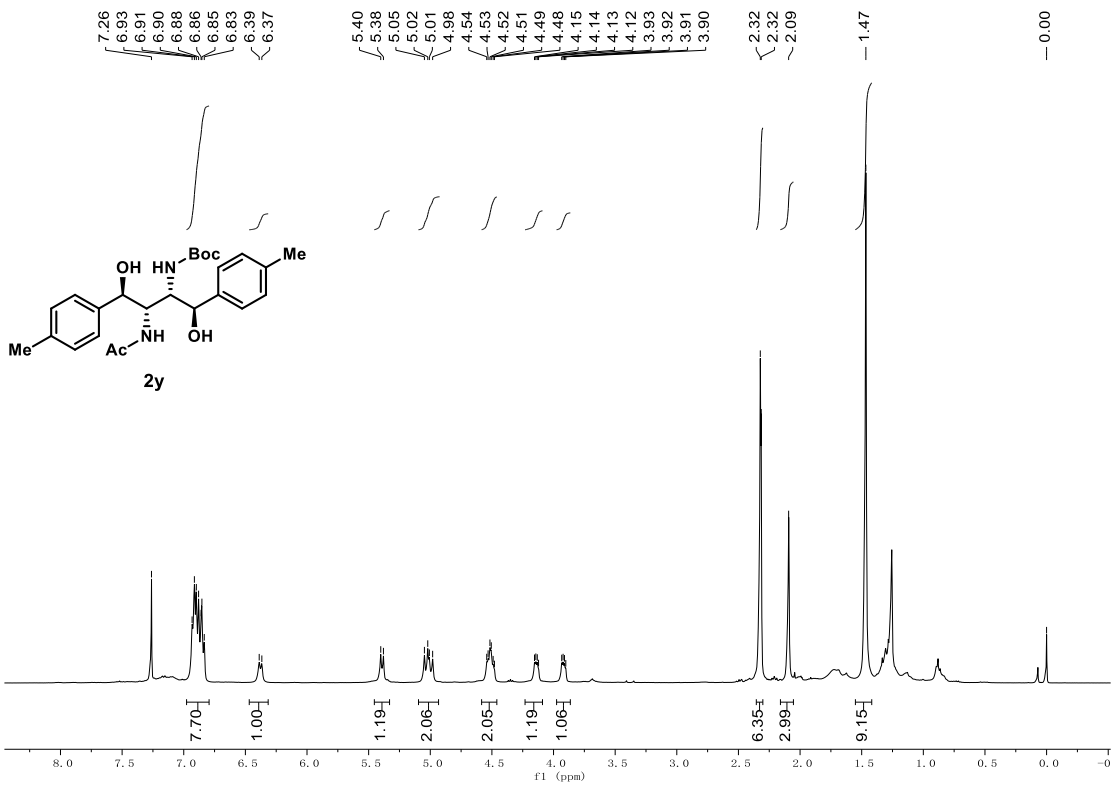
Supplementary Figure 119. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2w



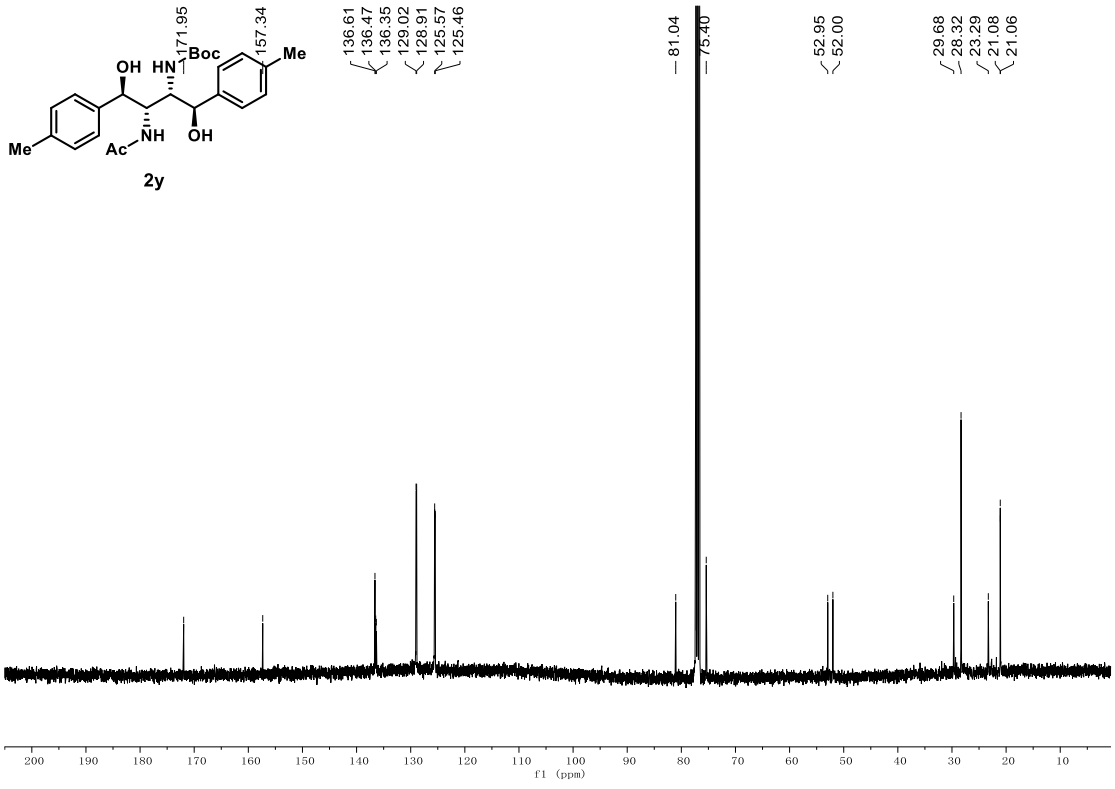
Supplementary Figure 120. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2x



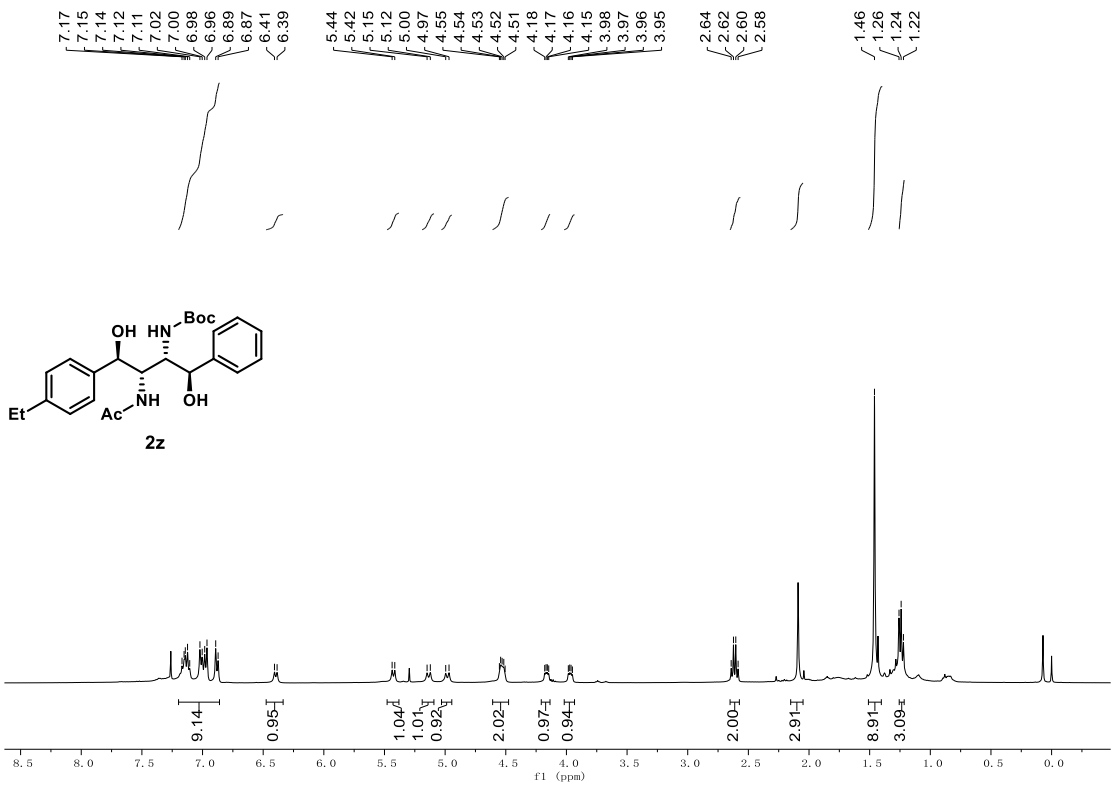
Supplementary Figure 121. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2x



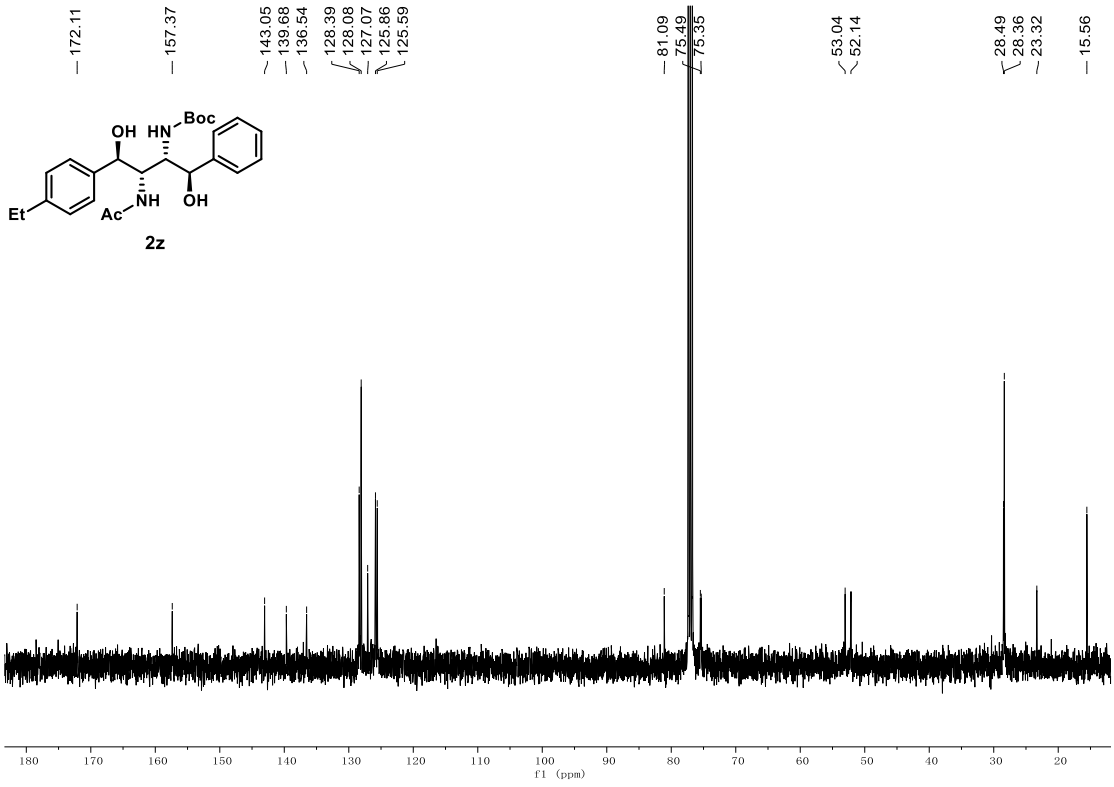
Supplementary Figure 122. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2y



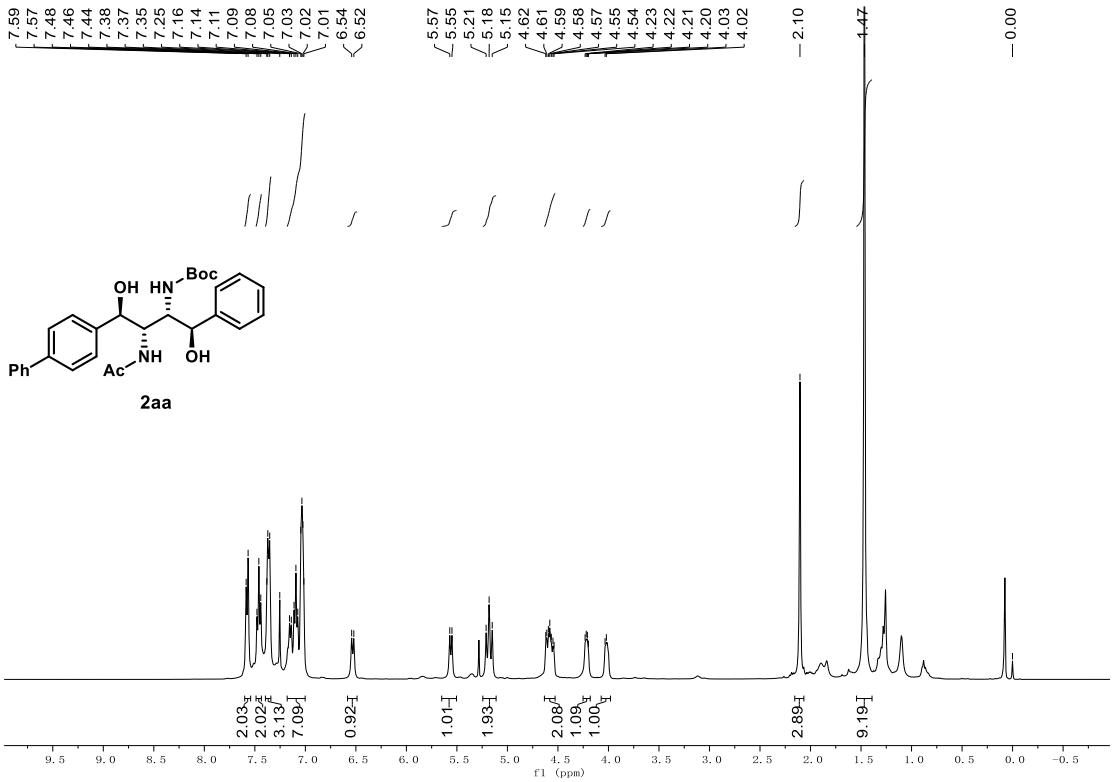
Supplementary Figure 123. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2y



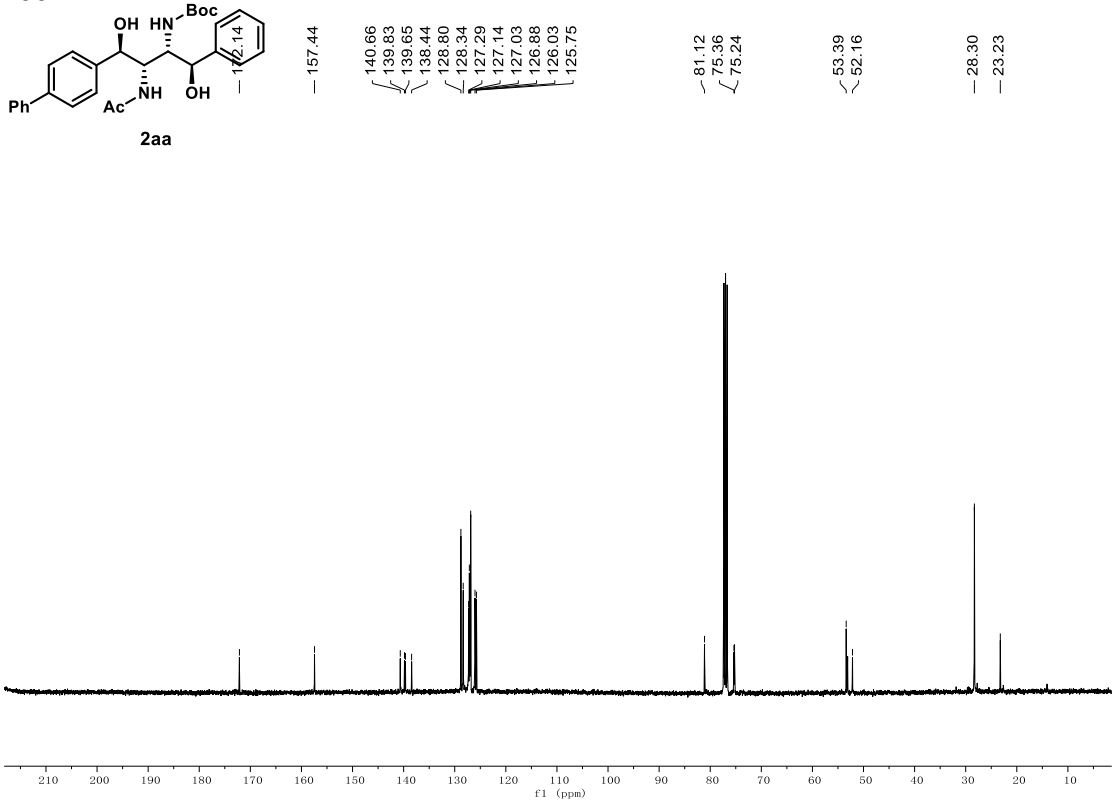
Supplementary Figure 124. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2z



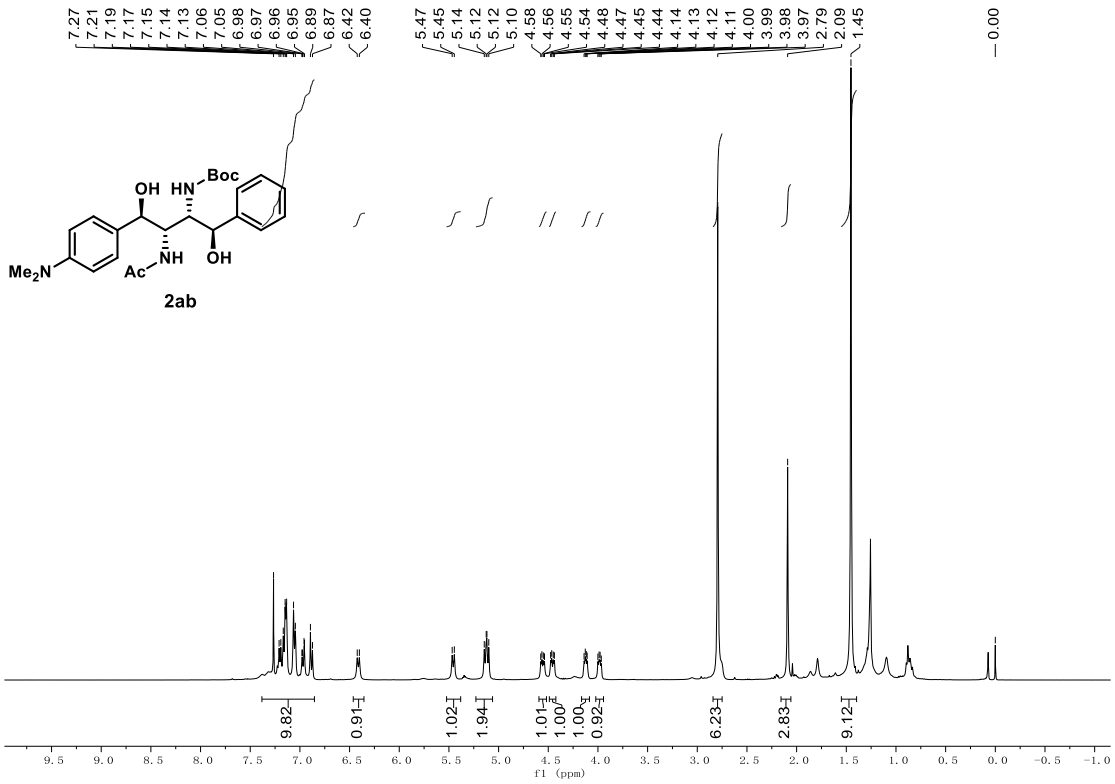
Supplementary Figure 125. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2z



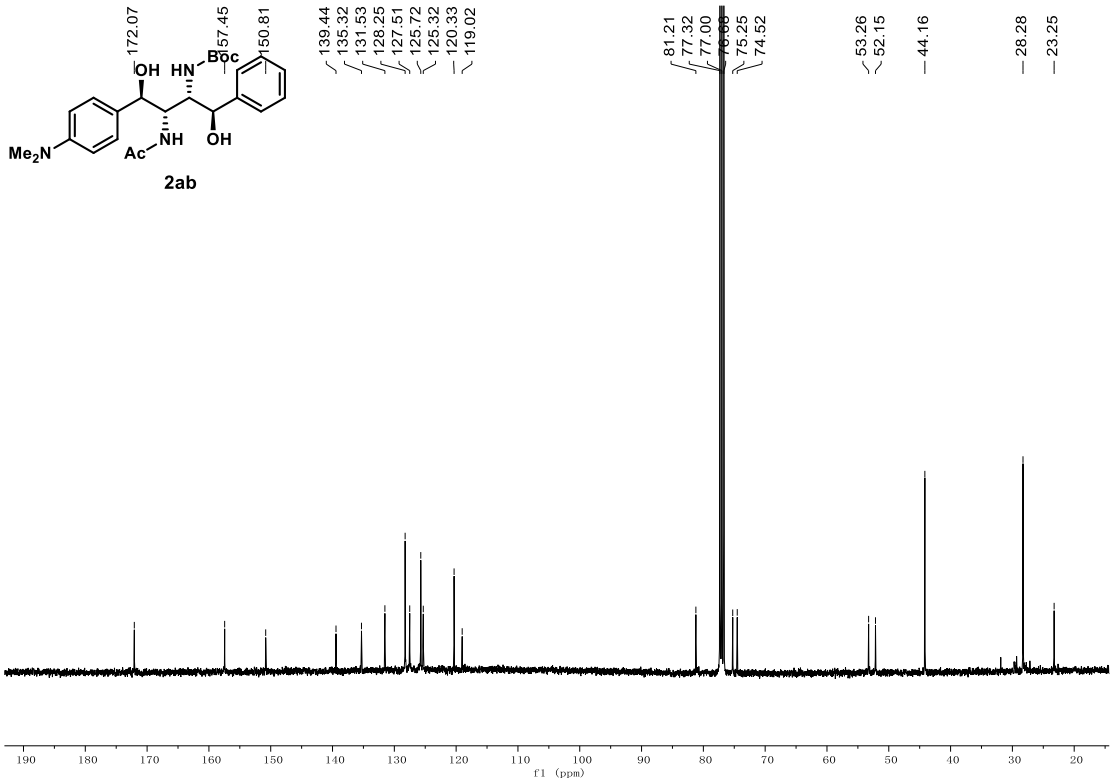
Supplementary Figure 126. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2aa



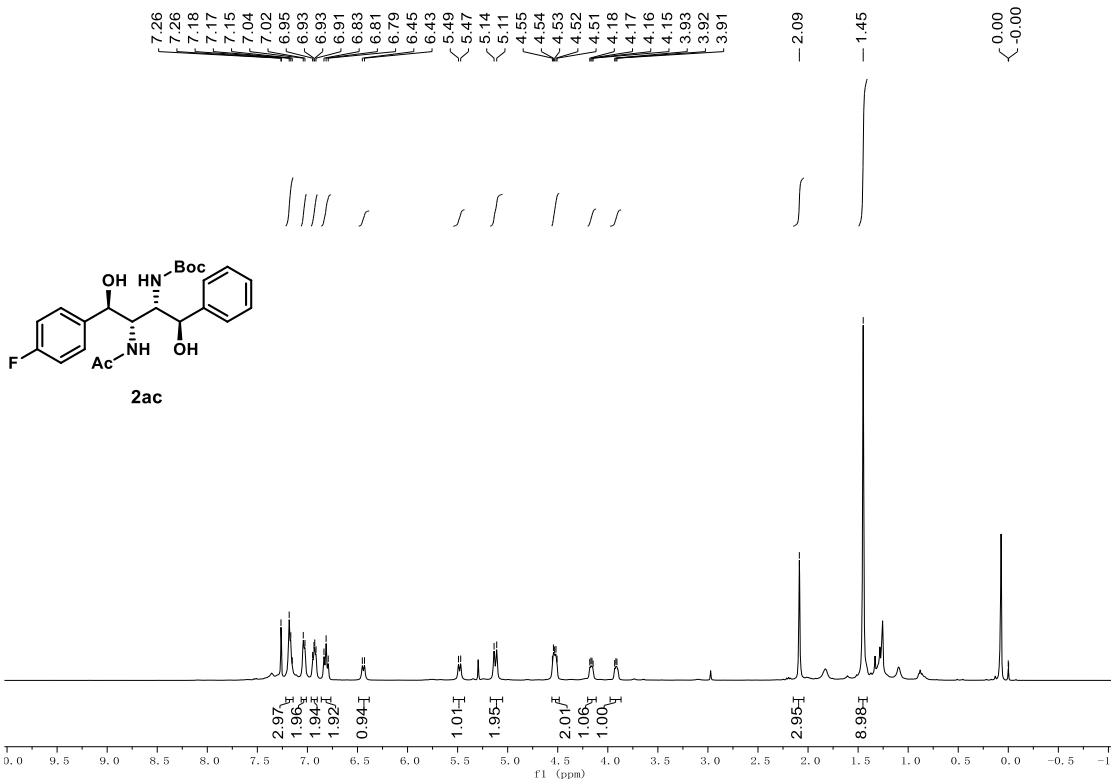
Supplementary Figure 127. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2aa



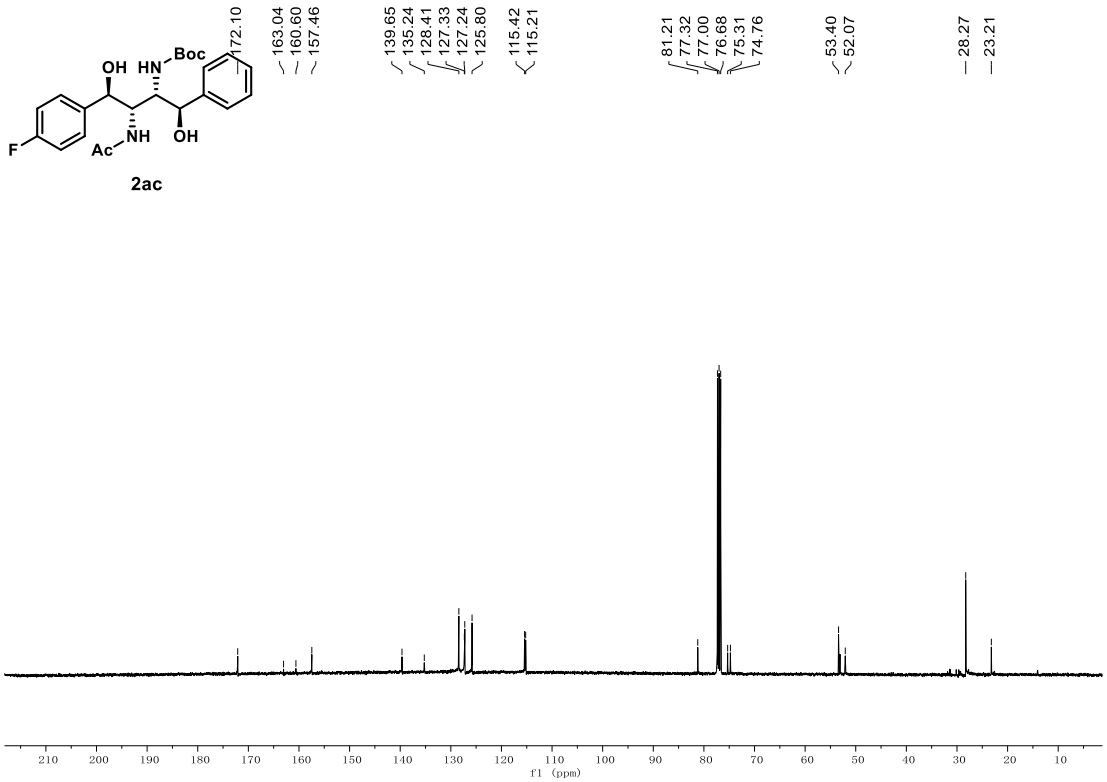
Supplementary Figure 128. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ab



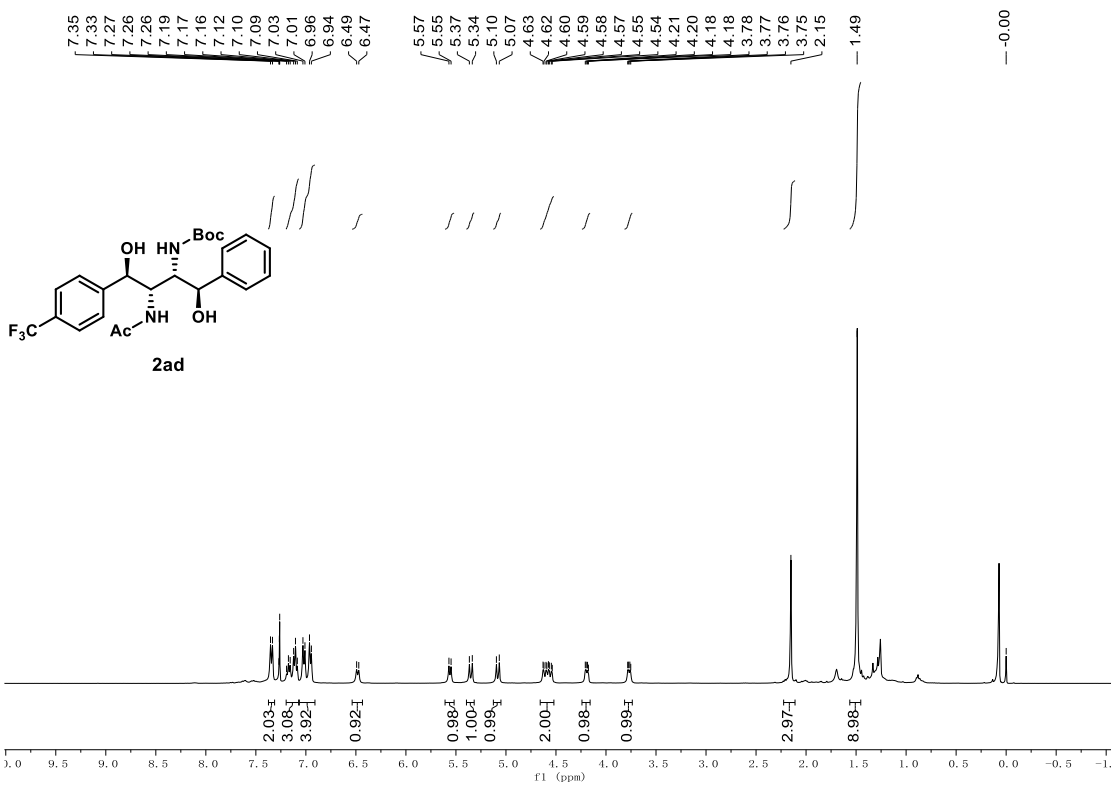
Supplementary Figure 129. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ab



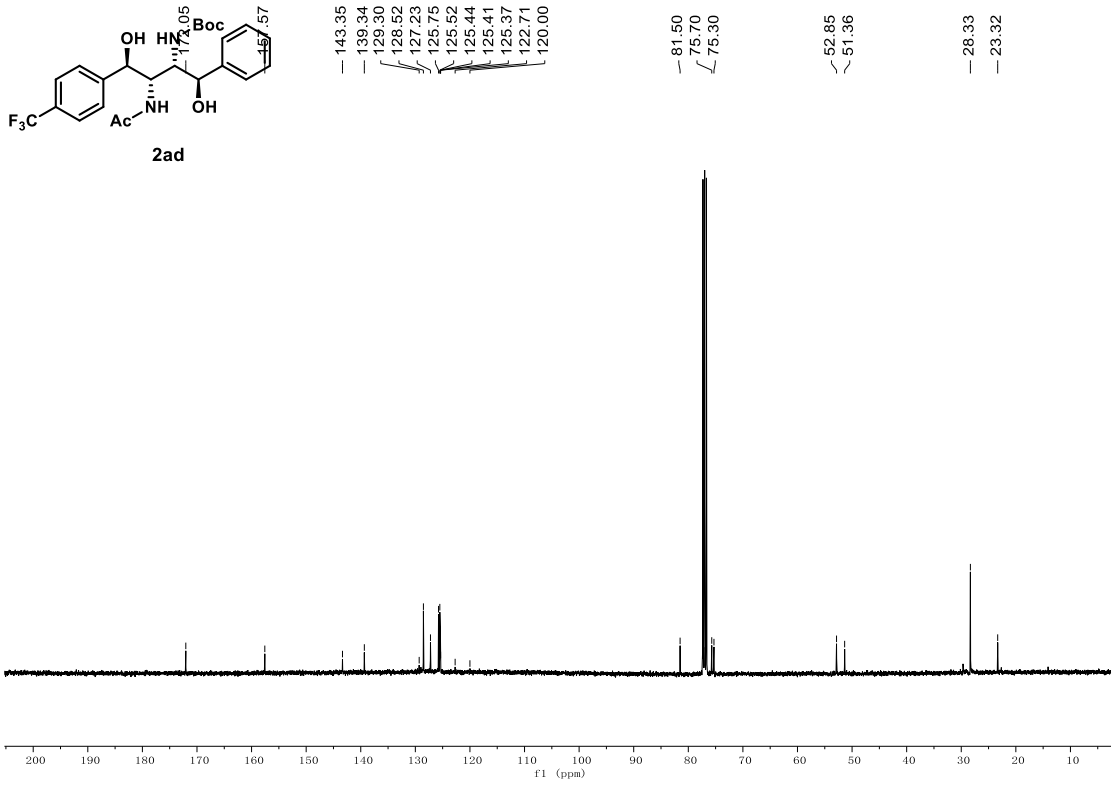
Supplementary Figure 130. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ac



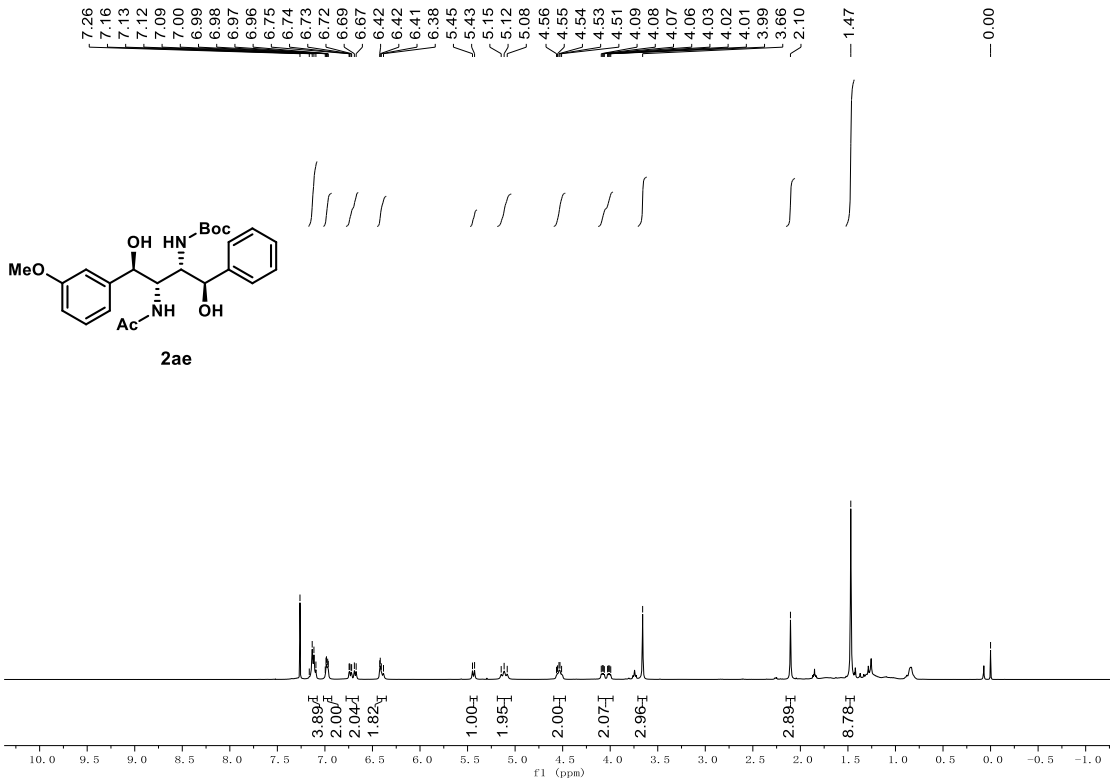
Supplementary Figure 131. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ac



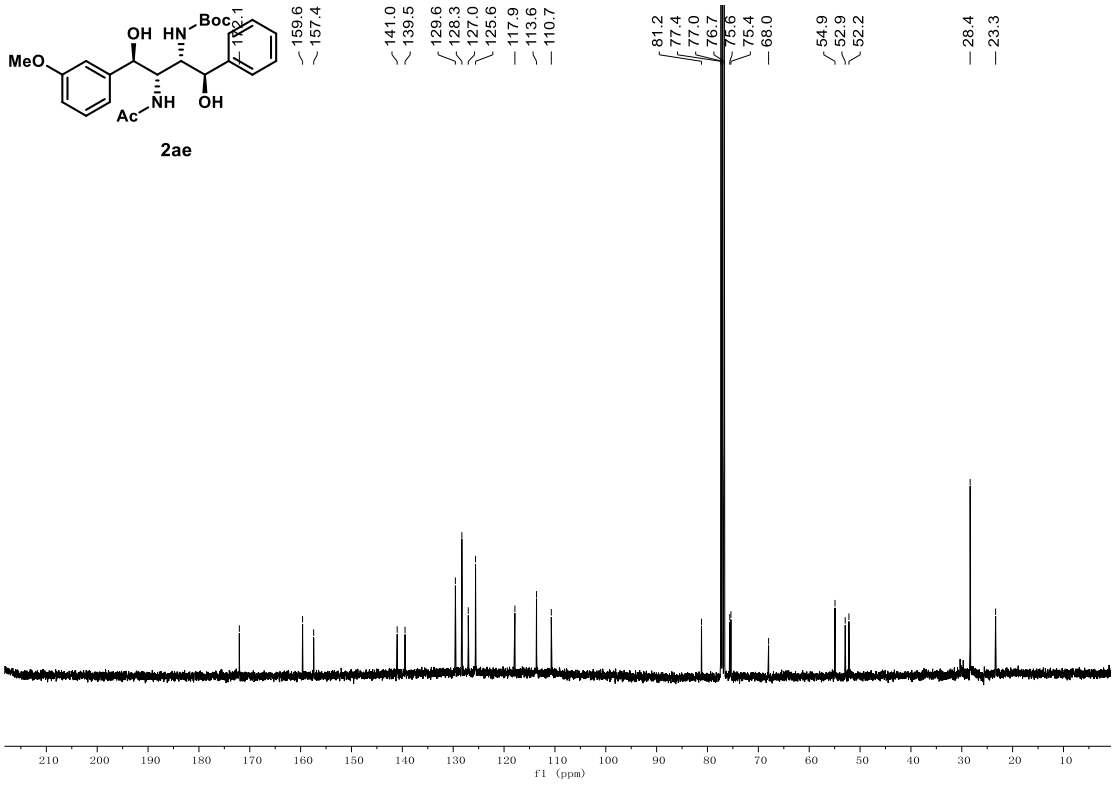
Supplementary Figure 132. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ad



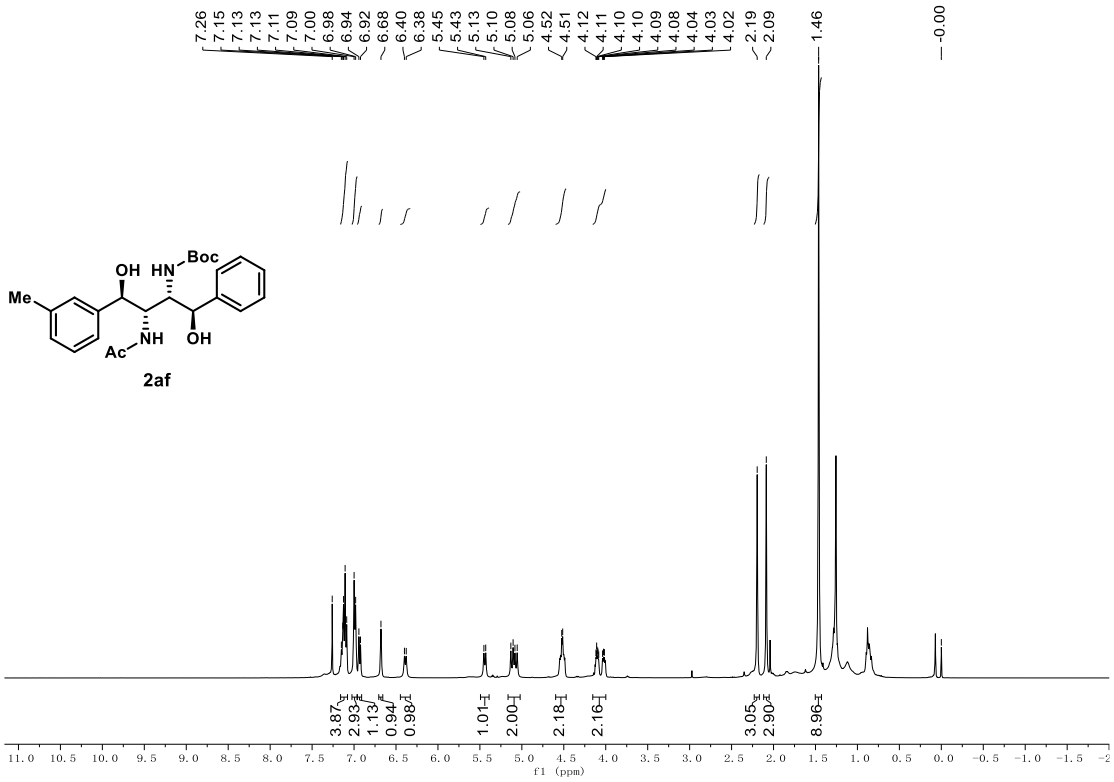
Supplementary Figure 133. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ad



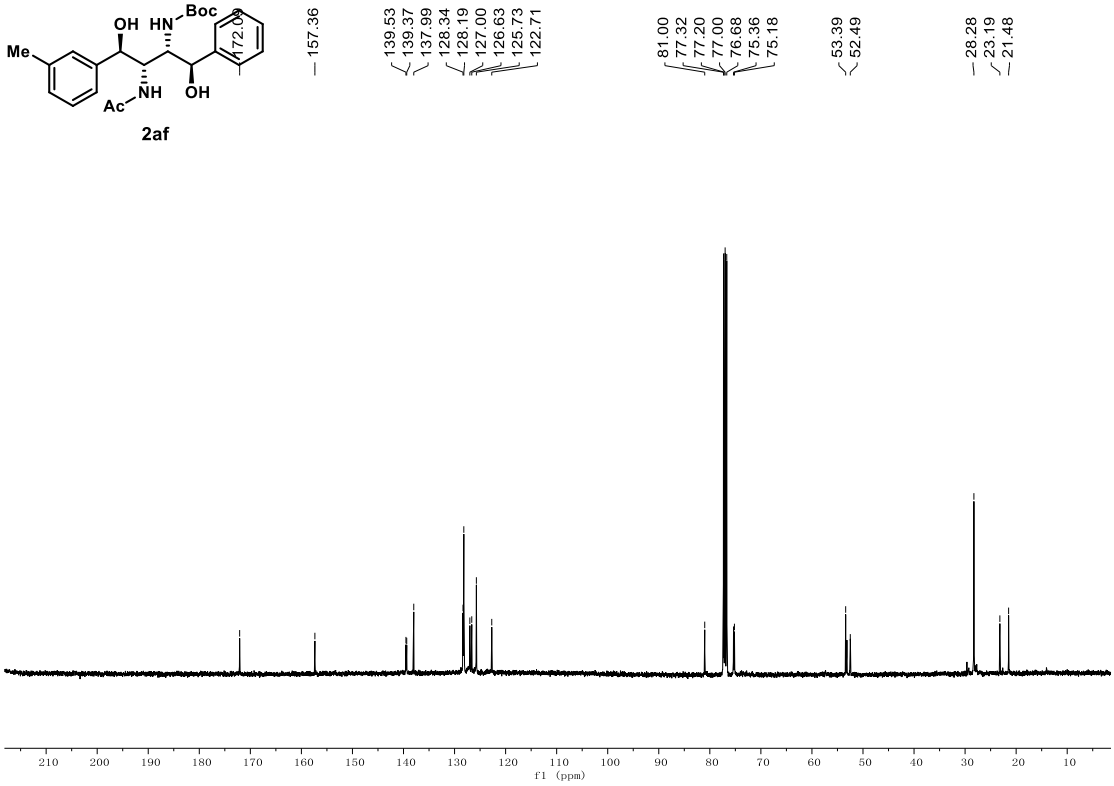
Supplementary Figure 134. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ae



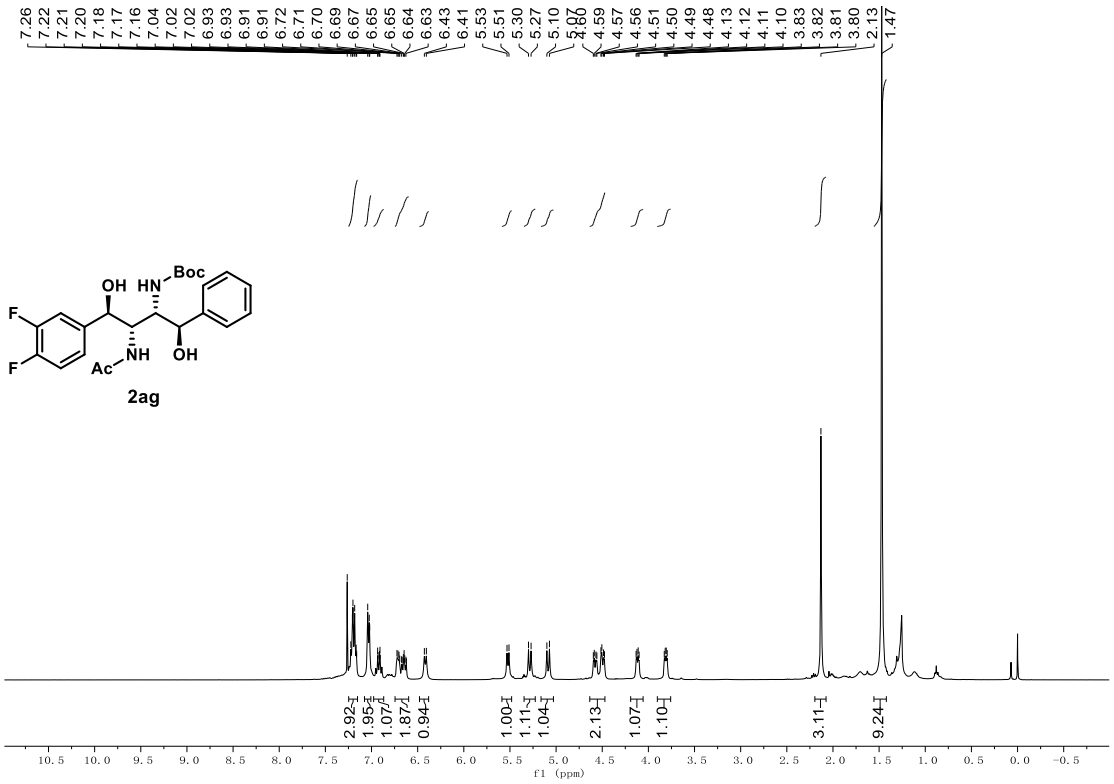
Supplementary Figure 135. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ae



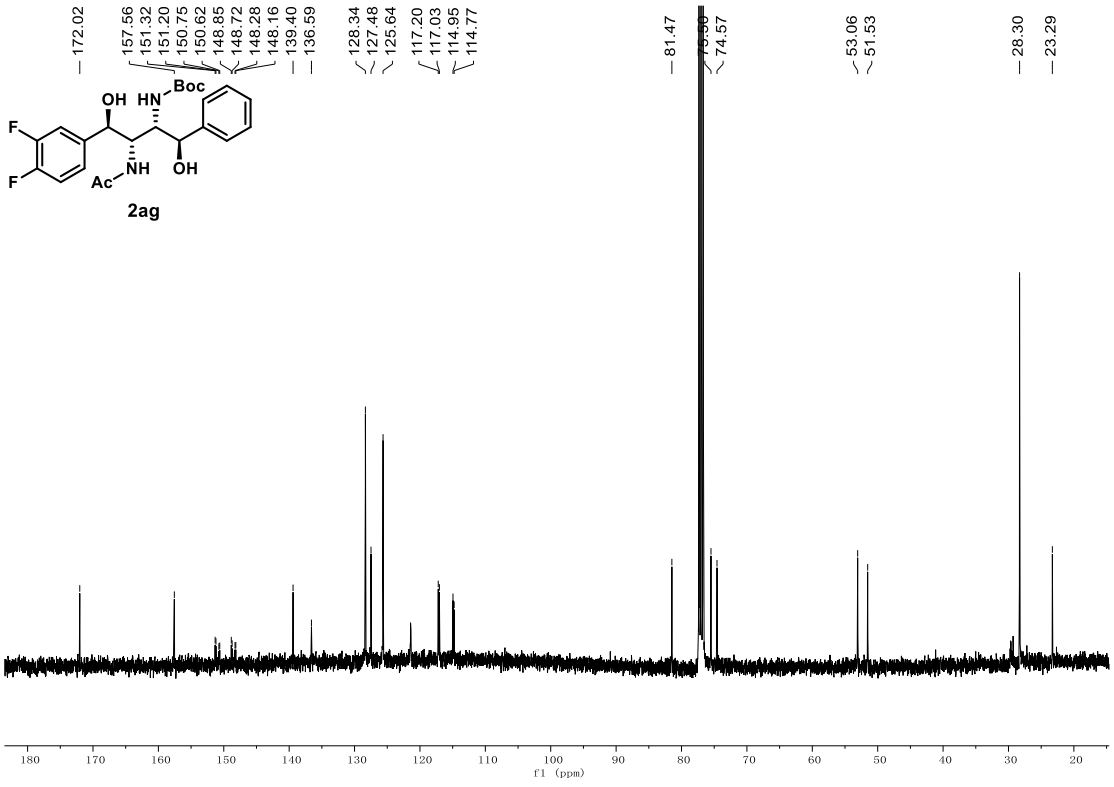
Supplementary Figure 136. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2af



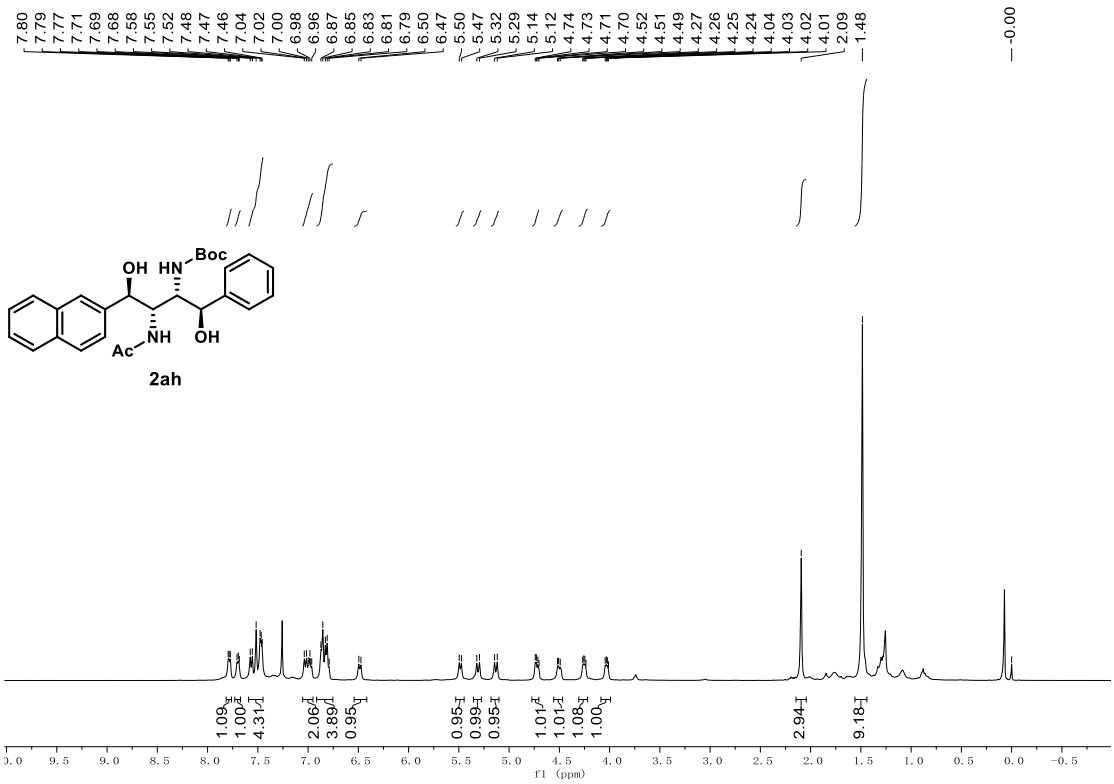
Supplementary Figure 137. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2af



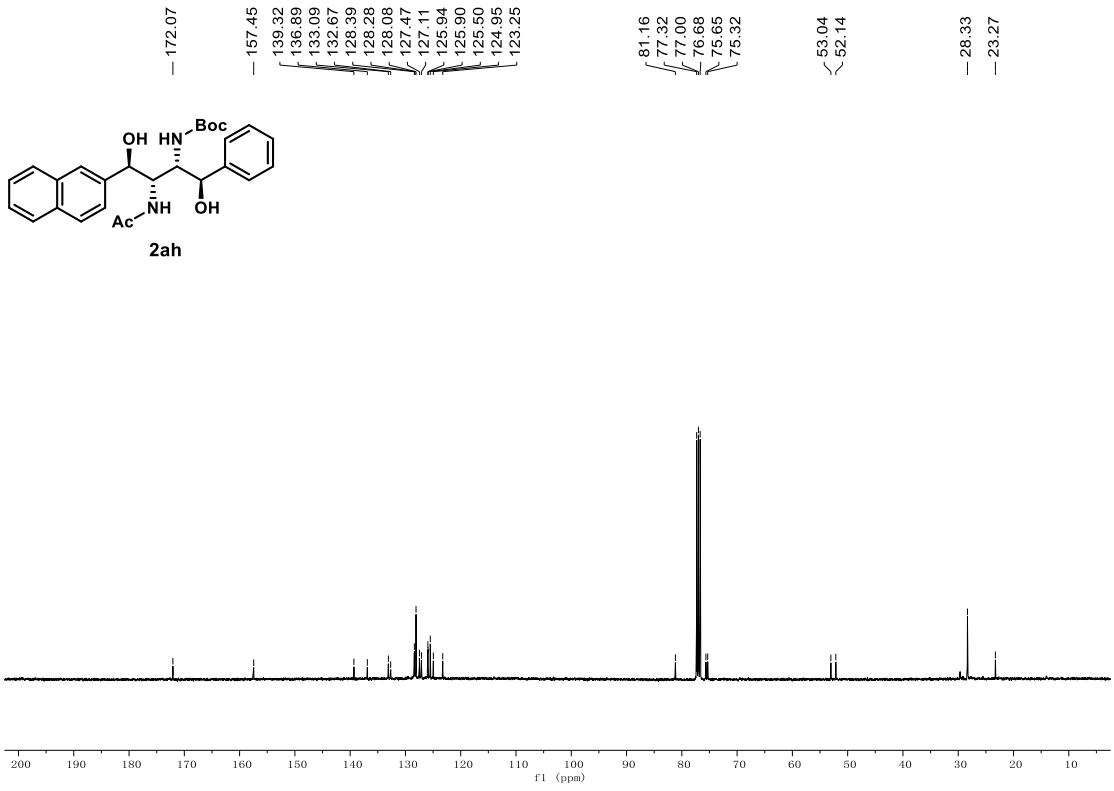
Supplementary Figure 138. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ag



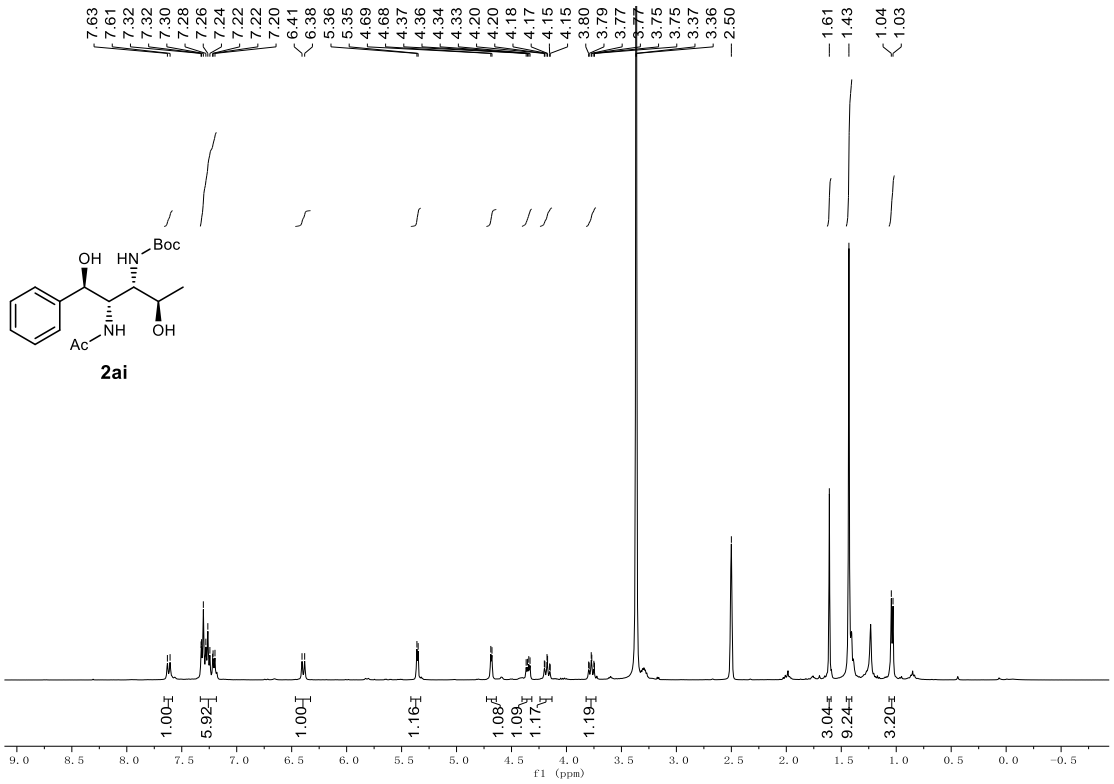
Supplementary Figure 139. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ag



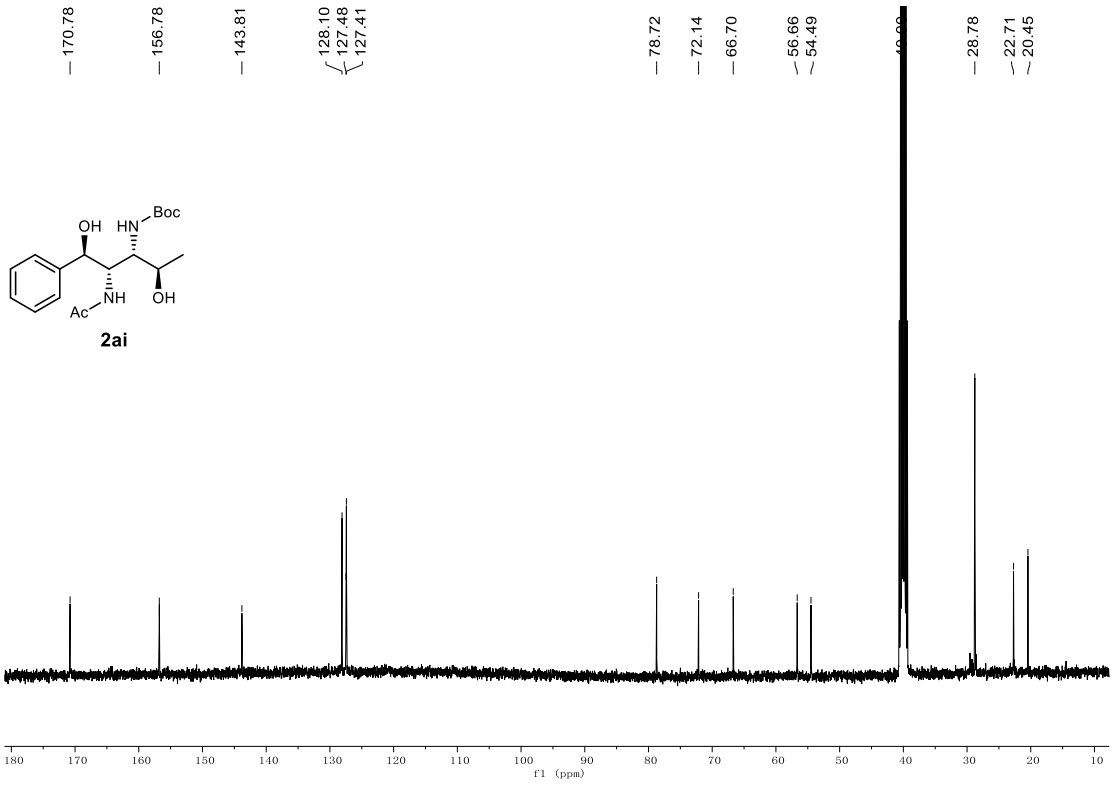
^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ah



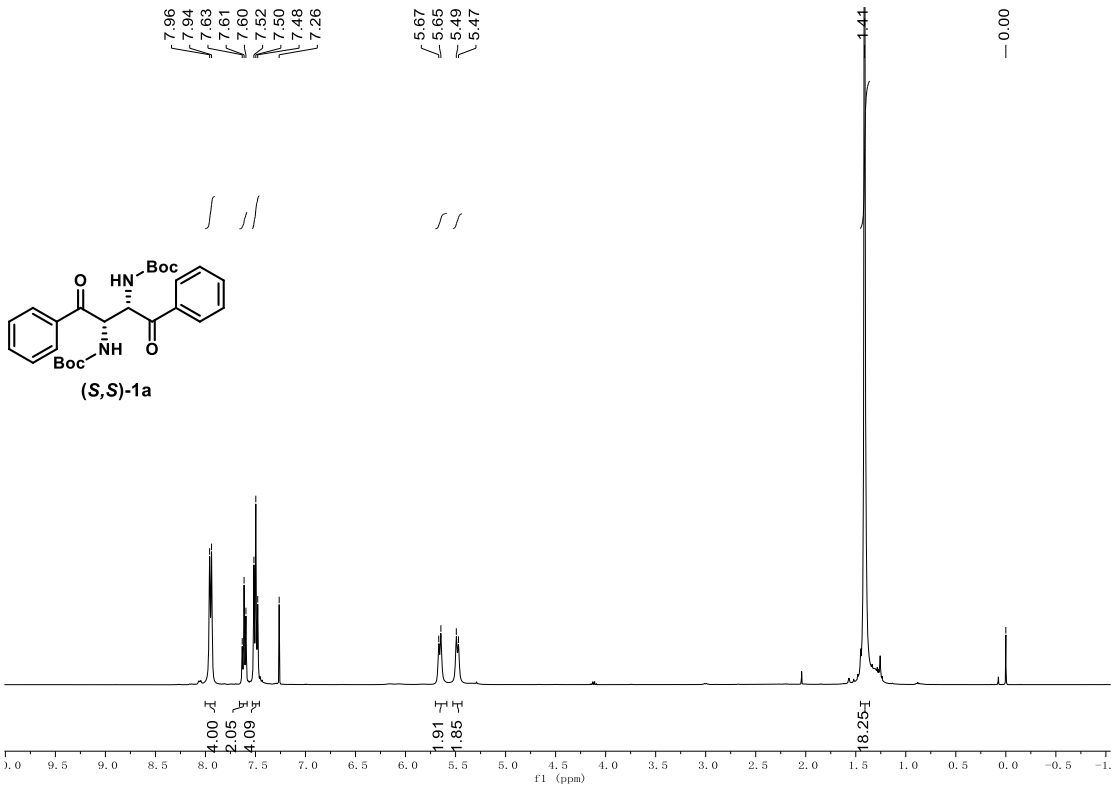
Supplementary Figure 140. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ah



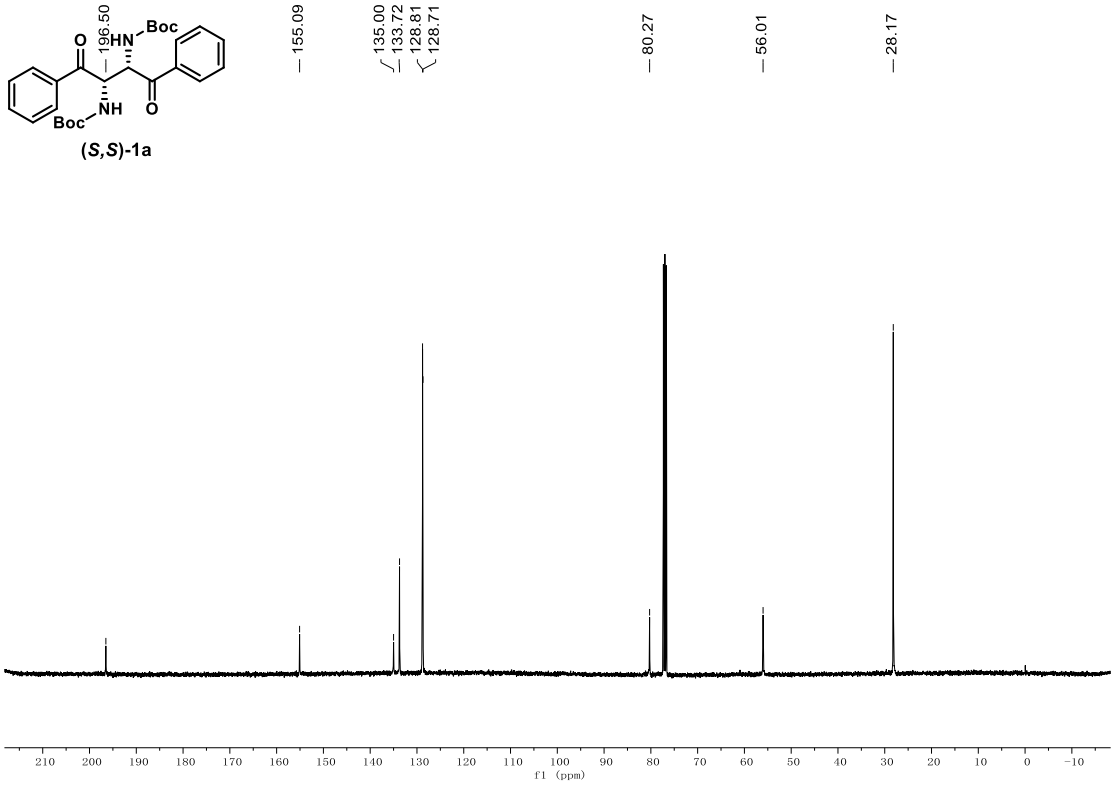
^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 2ai



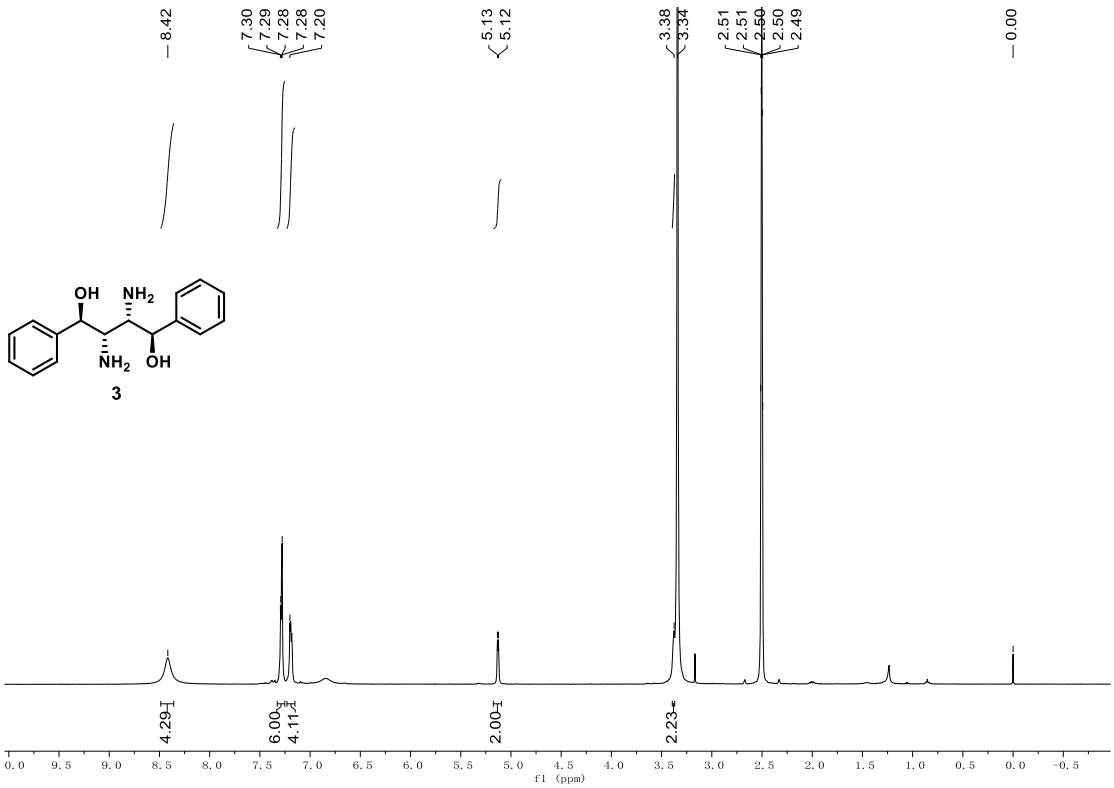
Supplementary Figure 141. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 2ai



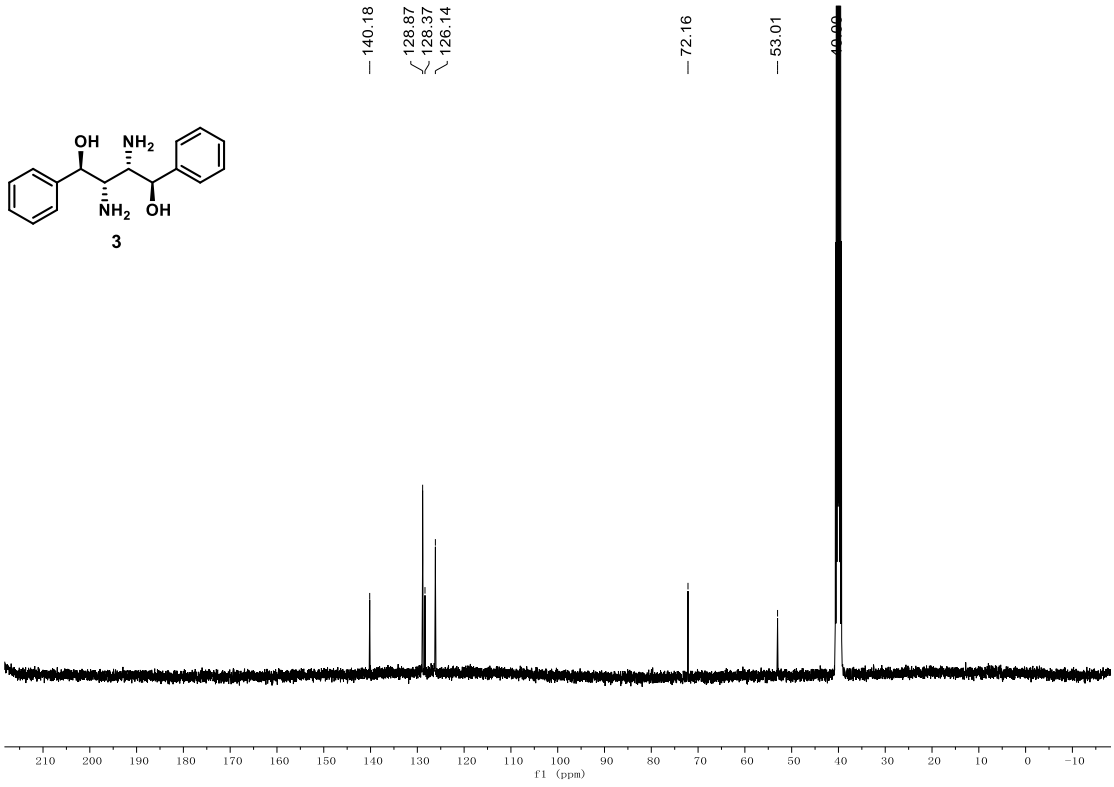
Supplementary Figure 142. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound (*S, S*)-1a



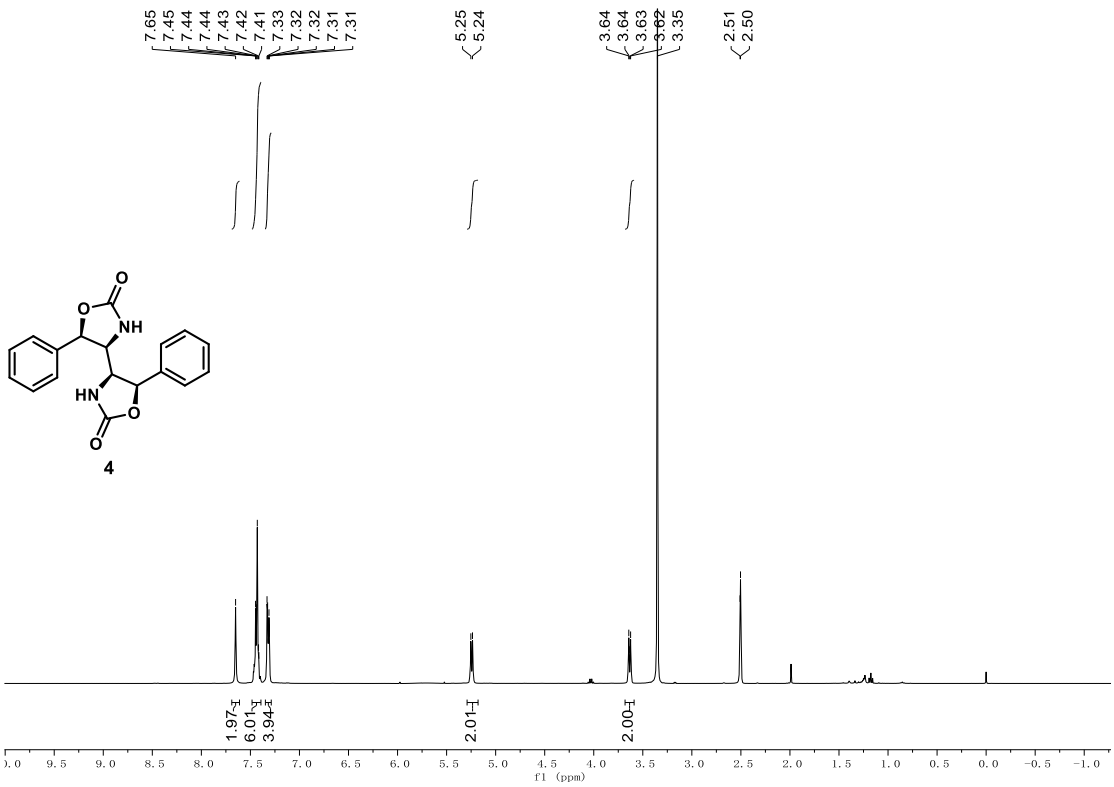
Supplementary Figure 143. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound (*S, S*)-1a



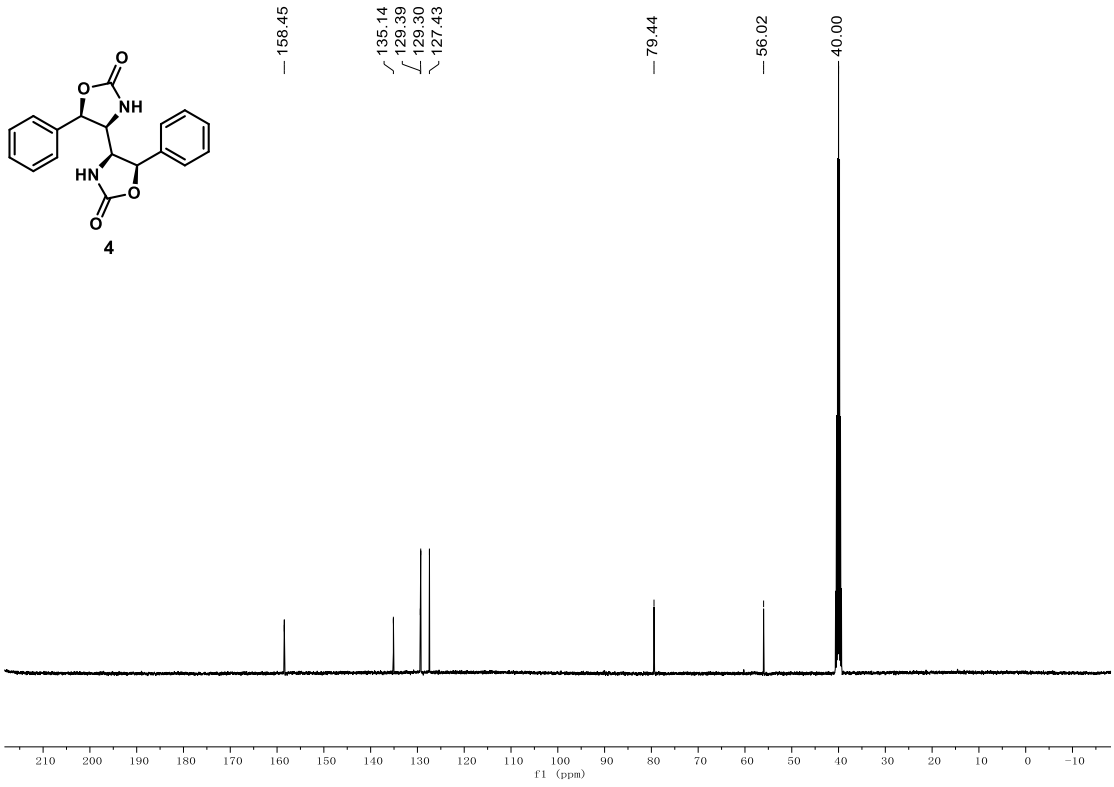
Supplementary Figure 144. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 3



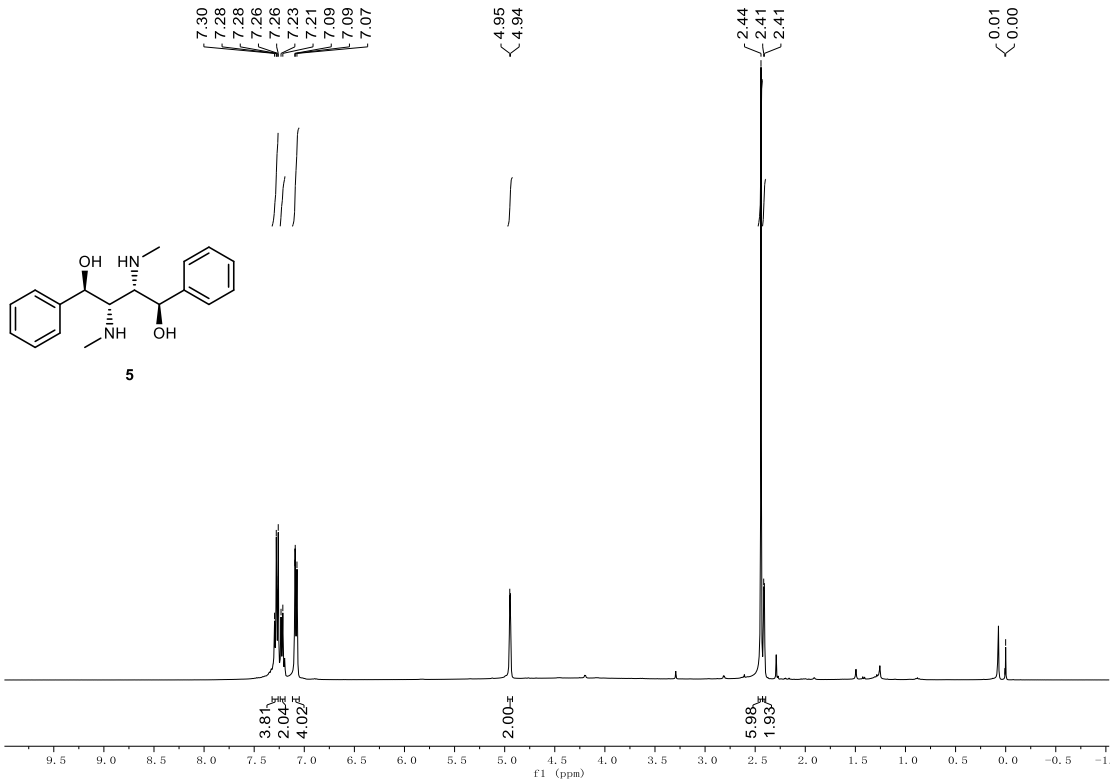
Supplementary Figure 145. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 3



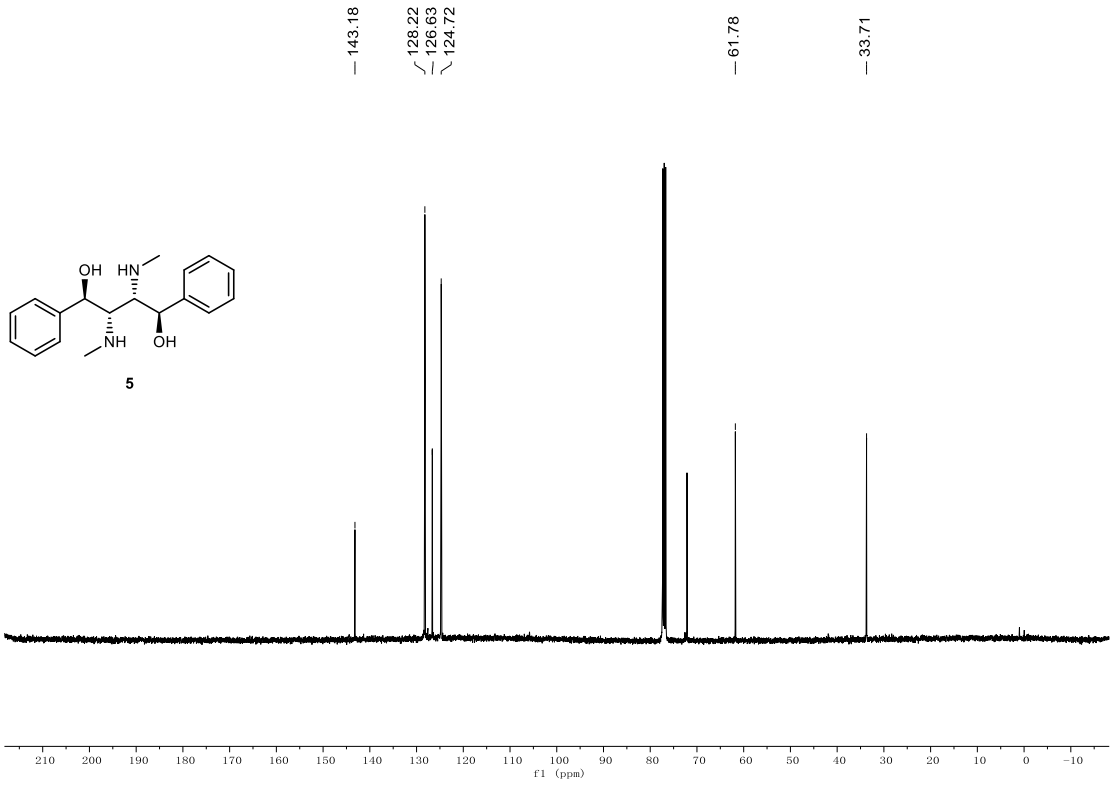
Supplementary Figure 146. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 4



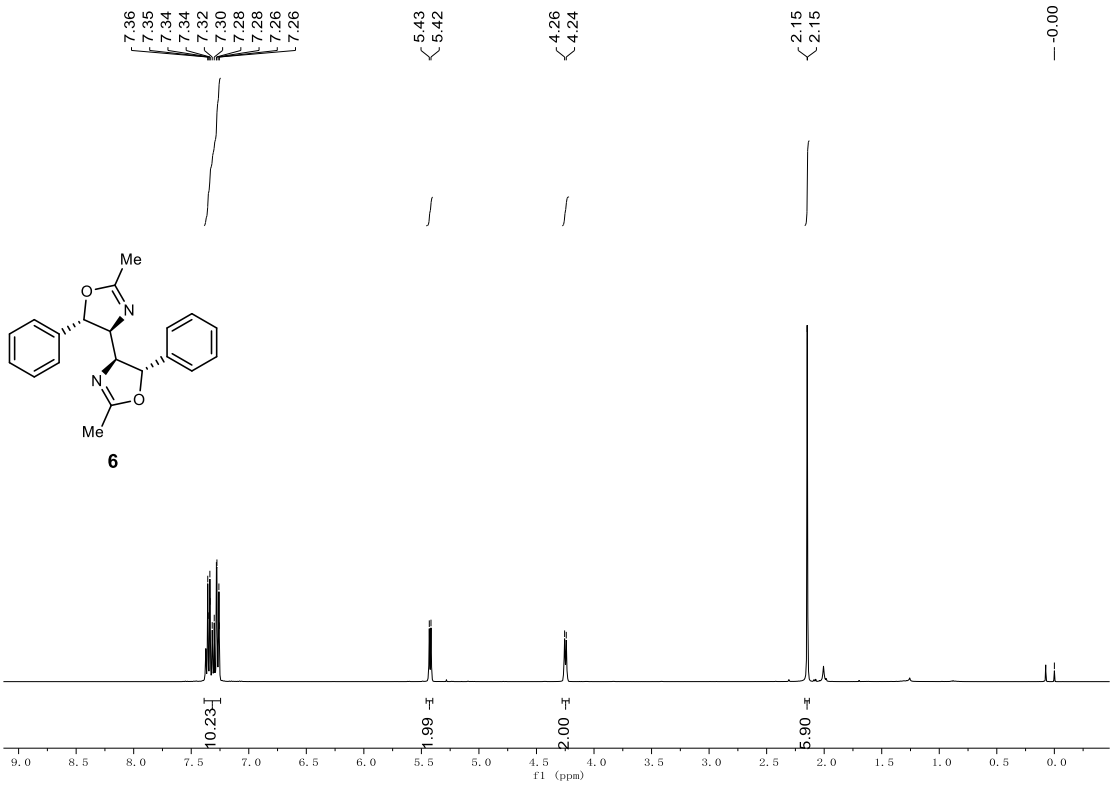
Supplementary Figure 147. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 4



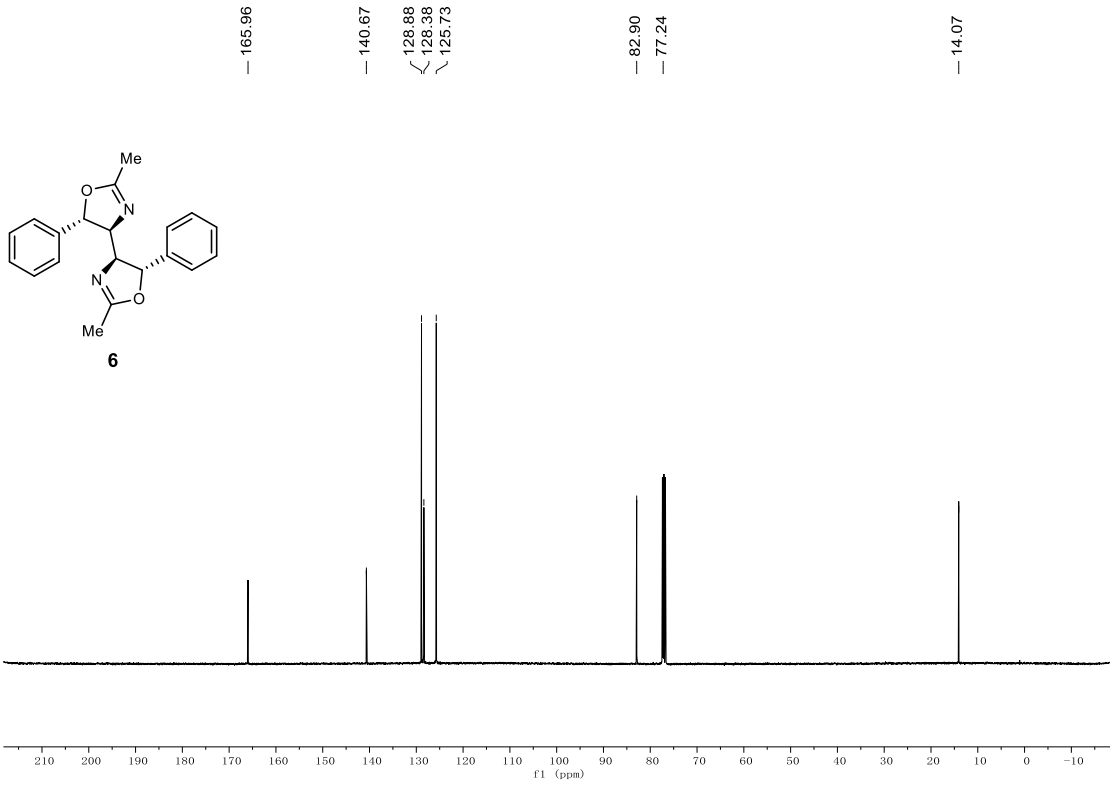
Supplementary Figure 148. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 5



Supplementary Figure 149. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 5

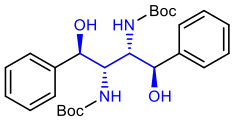


Supplementary Figure 150. ^{1}H NMR (400 MHz, $CDCl_3$) spectra for compound 6

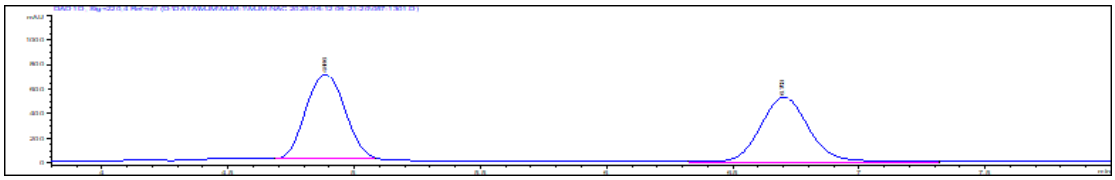


Supplementary Figure 151. ^{13}C NMR (100 MHz, $CDCl_3$) spectra for compound 6

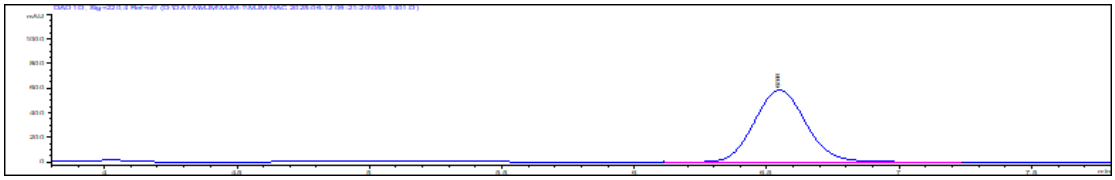
10. HPLC Data



Condition hexane: *i*-PrOH = 85:15,
Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

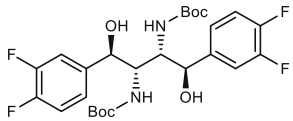


#	Time	Area	Height	Width	Area%	Symmetry
1	4.886	7411.9	683.5	0.1807	50.374	0.976
2	6.703	7301.9	531.1	0.2291	49.626	0.88

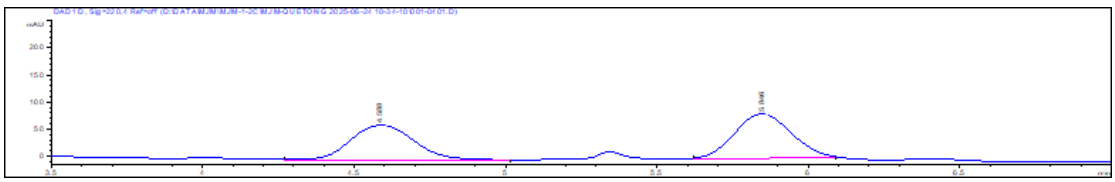


#	Time	Area	Height	Width	Area%	Symmetry
1	6.548	7961.8	585.1	0.2107	100.000	0.923

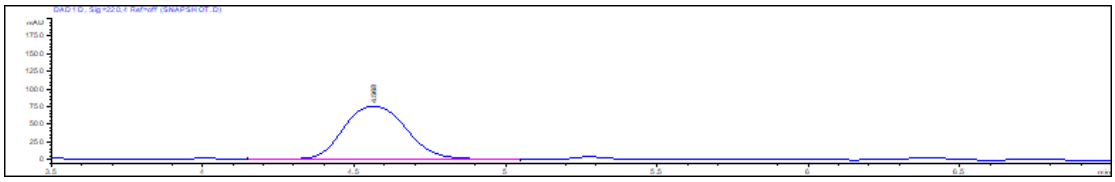
Supplementary Figure 152. HPLC spectra for compound 2a



Condition hexane: *i*-PrOH = 85:15,
Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



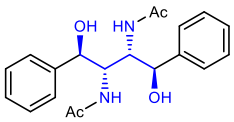
#	Time	Area	Height	Width	Area%	Symmetry
1	4.588	981.4	65	0.2517	47.942	0.889
2	5.846	1065.6	81.8	0.2172	52.058	0.92



#	Time	Area	Height	Width	Area%	Symmetry

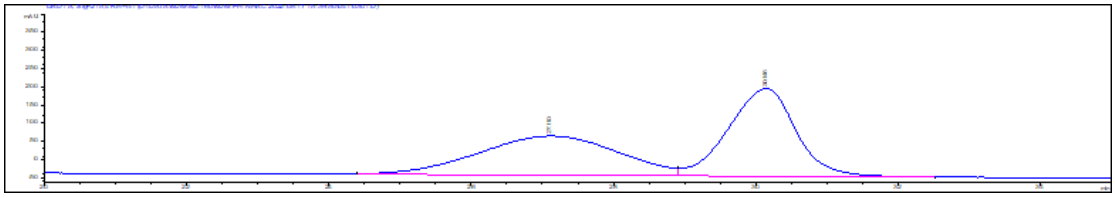
1	4.568	11207.2	761.7	0.2356	100.000	0.897

Supplementary Figure 154. HPLC spectra for compound 2b

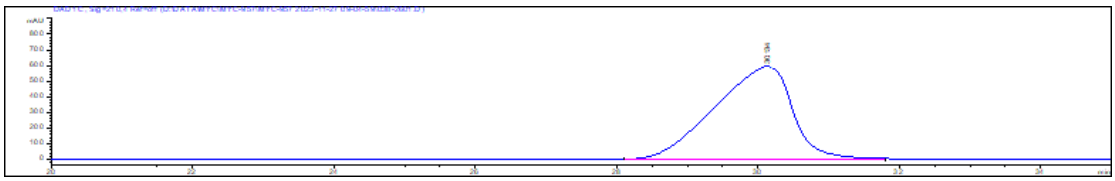


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

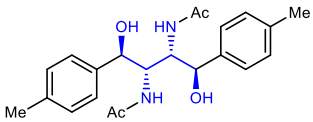


#	Time	Area	Height	Width	Area%	Symmetry
1	27.11	14594.9	107	1.6038	47.915	1.036
2	30.146	15865.4	240.2	0.8805	52.085	1.224



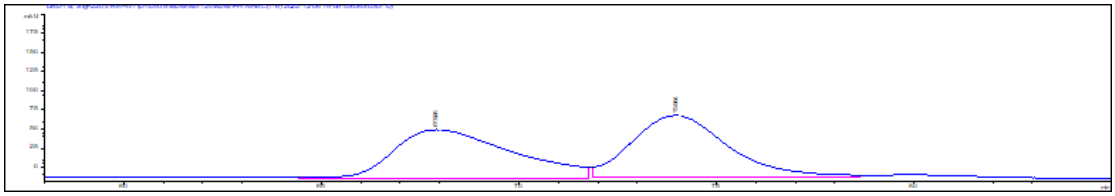
#	Time	Area	Height	Width	Area%	Symmetry
1	30.134	45372	592.7	1.2759	100.000	1.974

Supplementary Figure 153. HPLC spectra for compound 2c

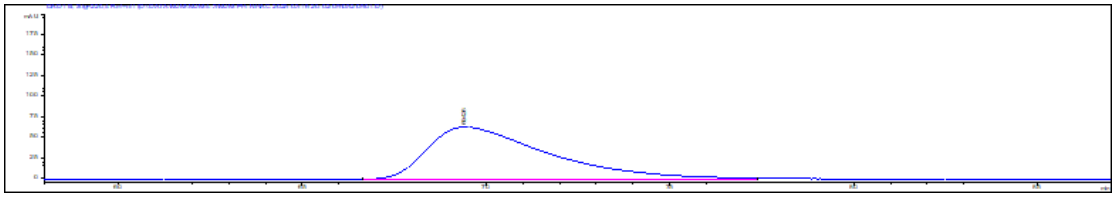


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

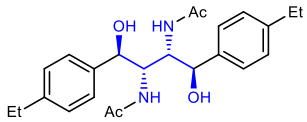


#	Time	Area	Height	Width	Area%	Symmetry
1	67.926	13212.2	63.4	3.4728	49.100	0.565
2	73.984	13696.4	80.4	2.8392	50.900	0.796



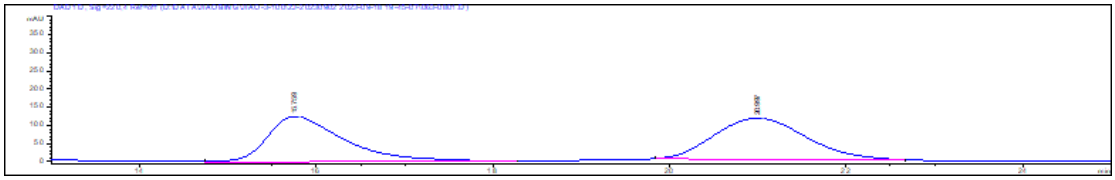
#	Time	Area	Height	Width	Area%	Symmetry
1	69.426	14883	64.1	3.8701	100.000	0.436

Supplementary Figure 155. HPLC spectra for compound 2d

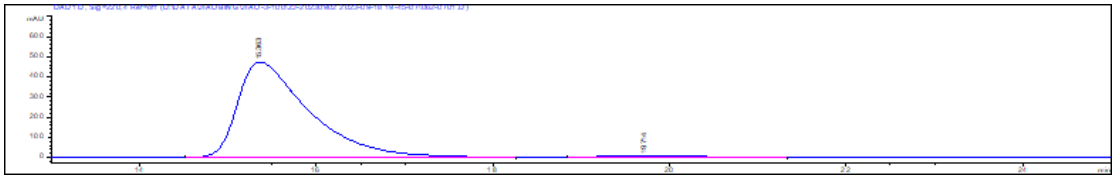


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

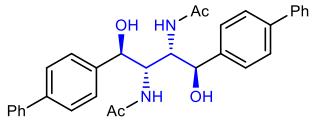


#	Time	Area	Height	Width	Area%	Symmetry
1	15.759	7572	124.8	1.0111	48.131	0.557
2	20.997	8159.9	113.4	1.1988	51.869	0.863



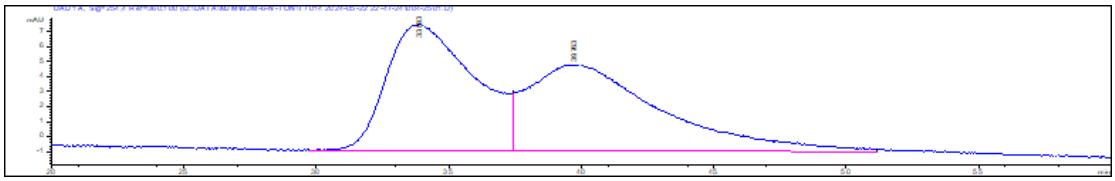
#	Time	Area	Height	Width	Area%	Symmetry
1	15.363	26556.2	472.4	0.8059	98.480	0.426
2	19.714	410	5.4	0.8899	1.520	0.605

Supplementary Figure 156. HPLC spectra for compound 2e

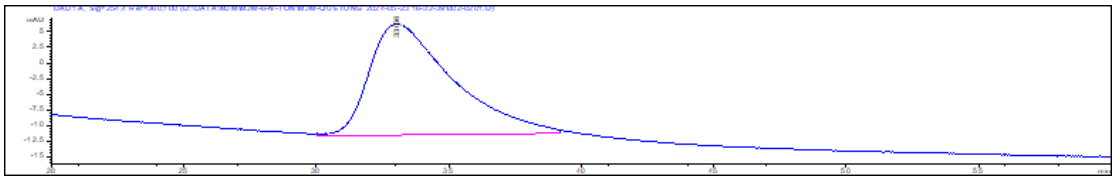


Condition hexane: *i*-PrOH = 90:10,

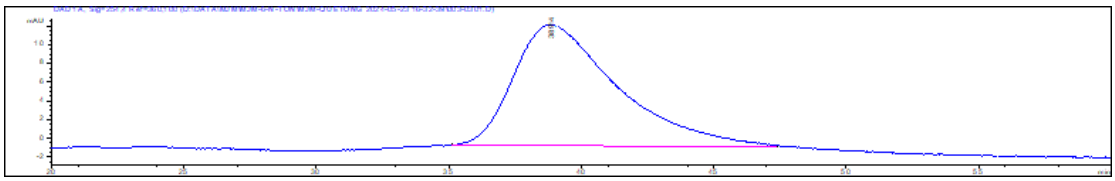
Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H



#	Time	Area	Height	Width	Area%	Symmetry
1	33.893	1879.5	8.4	3.7136	49.817	0.629
2	39.763	1893.3	5.7	5.5491	50.183	0.628

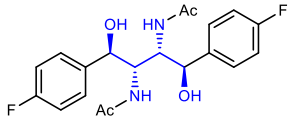


#	Time	Area	Height	Width	Area%	Symmetry
1	33.056	3906.2	17.8	3.6496	100.000	0.498



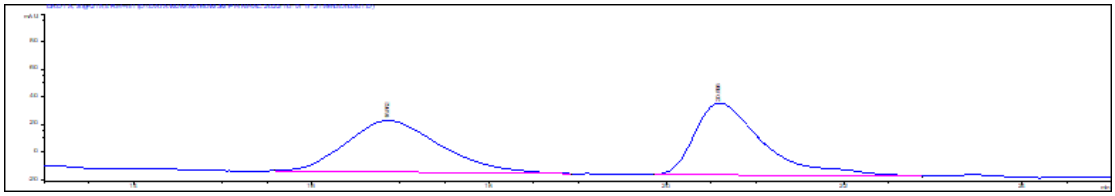
#	Time	Area	Height	Width	Area%	Symmetry
1	38.914	3521.1	12.9	4.5619	100.000	0.575

Supplementary Figure 157. HPLC spectra for compound 2f

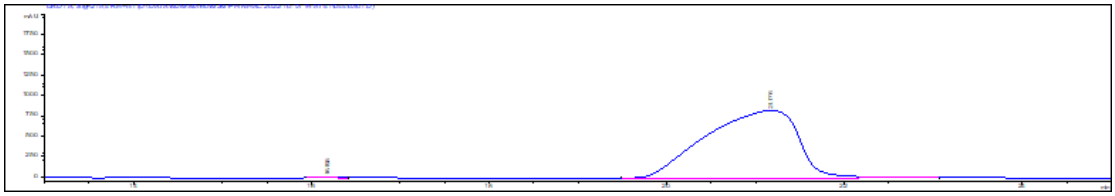


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

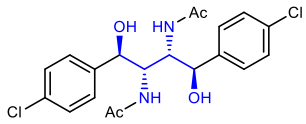


#	Time	Area	Height	Width	Area%	Symmetry
1	16.862	2737.1	37.1	0.8656	49.703	0.769
2	20.595	2769.8	52	0.6275	50.297	0.552



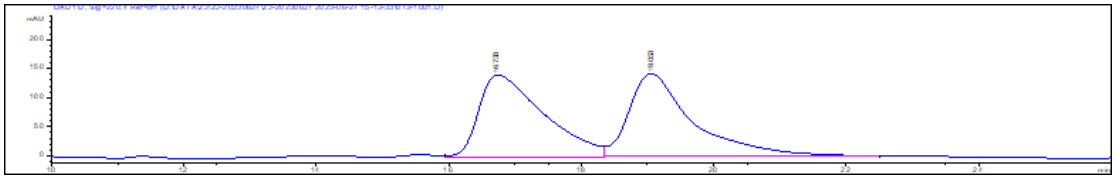
#	Time	Area	Height	Width	Area%	Symmetry
1	16.193	153.4	7.9	0.3253	0.256	0.67
2	21.176	59855.9	827.8	0.8576	99.744	2.274

Supplementary Figure 158. HPLC spectra for compound 2g



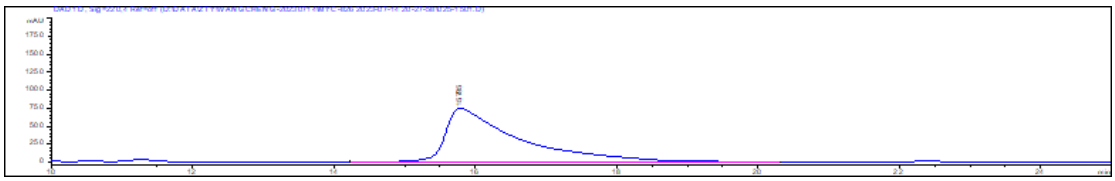
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



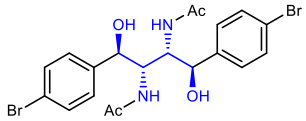
#	Time	Area	Height	Width	Area%	Symmetry

1	16.733	9561.2	140.9	0.9314	50.183	0.391
2	19.053	9491.3	142.3	0.9381	49.817	0.501



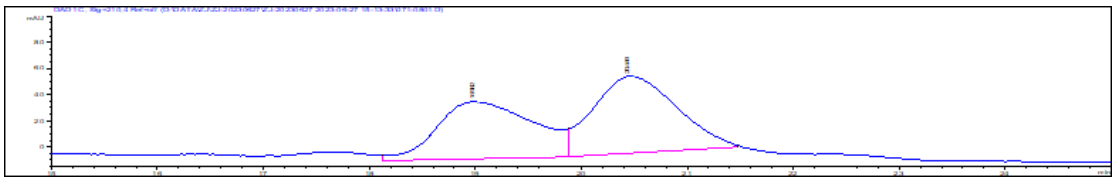
#	Time	Area	Height	Width	Area%	Symmetry
1	15.785	52893.3	741.4	0.9264	100.000	0.249

Supplementary Figure 159. HPLC spectra for compound 2h

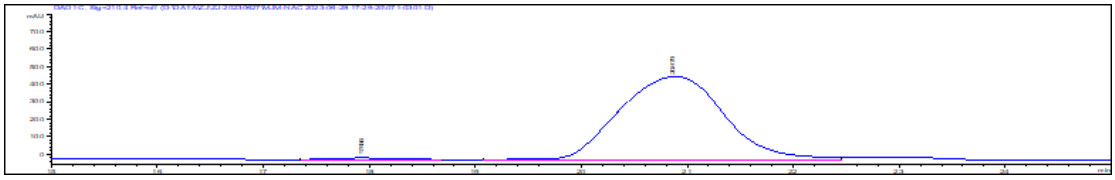


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

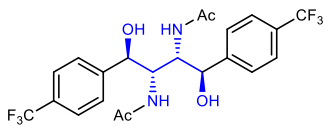


#	Time	Area	Height	Width	Area%	Symmetry
1	18.982	2844.2	44.2	1.0722	47.024	0.587
2	20.449	3204.1	58.8	0.9077	52.976	0.754



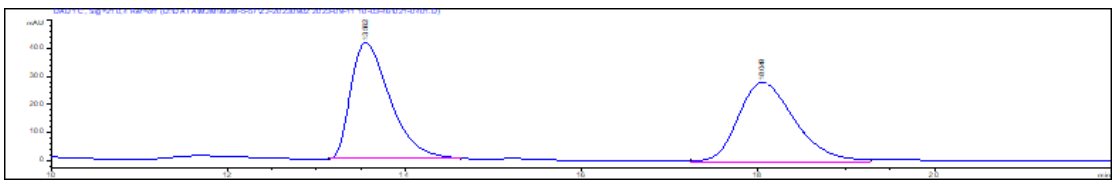
#	Time	Area	Height	Width	Area%	Symmetry
1	17.936	377.8	9.9	0.4491	1.138	0.751
2	20.879	32809.6	476.2	0.8438	98.862	1.065

Supplementary Figure 160. HPLC spectra for compound 2i

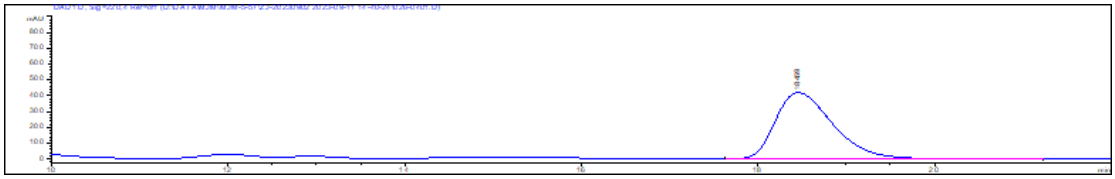


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

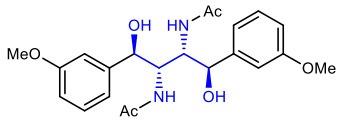


#	Time	Area	Height	Width	Area%	Symmetry
1	13.562	12805.1	414.8	0.5145	50.115	0.605
2	18.049	12746.2	285	0.7454	49.885	0.72



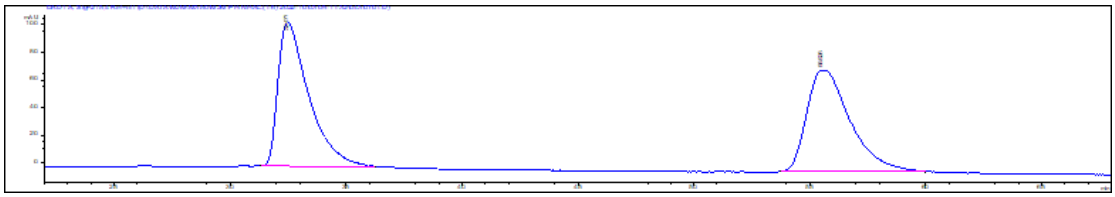
#	Time	Area	Height	Width	Area%	Symmetry
1	18.459	19181.4	419.1	0.6879	100.000	0.616

Supplementary Figure 161. HPLC spectra for compound 2j

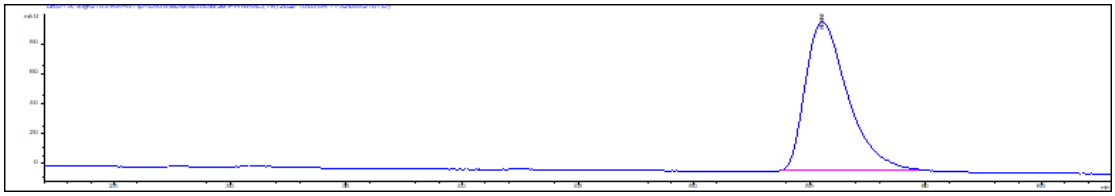


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

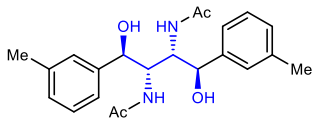


#	Time	Area	Height	Width	Area%	Symmetry
1	32.477	9645.1	104.2	1.1961	50.236	0.415
2	55.526	9554.3	73.8	1.5291	49.764	0.531



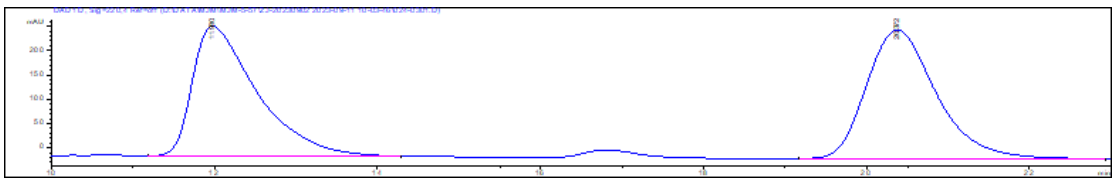
#	Time	Area	Height	Width	Area%	Symmetry
1	55.554	12602.5	99.8	1.4868	100.000	0.662

Supplementary Figure 162. HPLC spectra for compound 2k



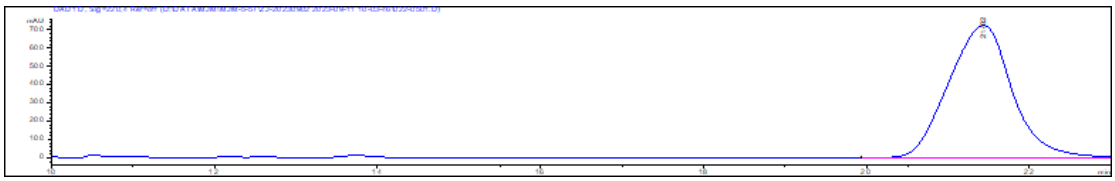
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H



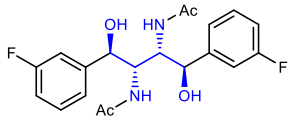
#	Time	Area	Height	Width	Area%	Symmetry

1	11.98	14706.8	267.7	0.797	48.246	0.435
2	20.372	15776	265.3	0.8837	51.754	0.738



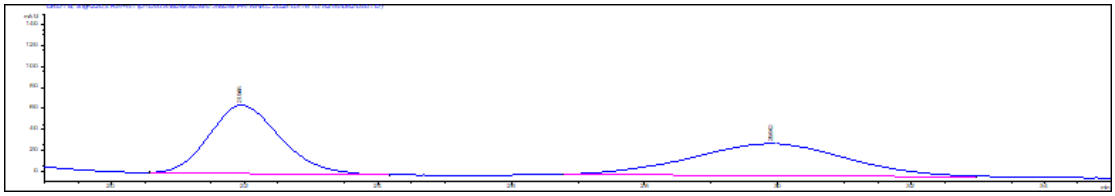
#	Time	Area	Height	Width	Area%	Symmetry
1	21.432	39029.6	725	0.7701	100.000	1.149

Supplementary Figure 163. HPLC spectra for compound 2l

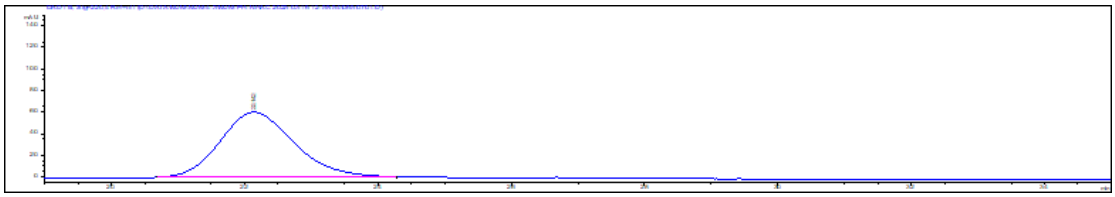


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

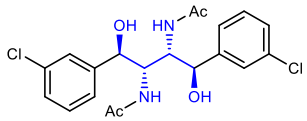


#	Time	Area	Height	Width	Area%	Symmetry
1	21.946	4803.4	64.8	0.893	49.964	0.809
2	29.887	4810.3	29.9	2.6781	50.036	1.093



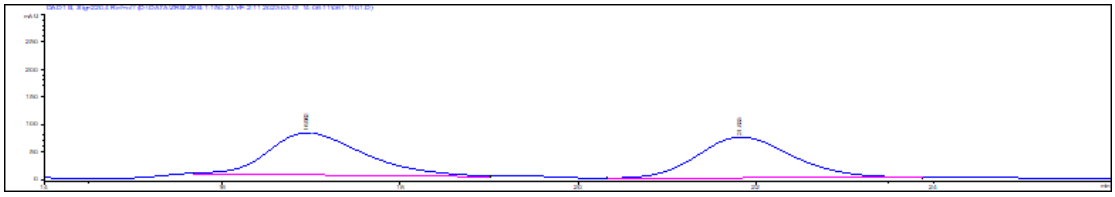
#	Time	Area	Height	Width	Area%	Symmetry
1	22.142	4632.7	60.2	0.9254	100.000	0.838

Supplementary Figure 164. HPLC spectra for compound 2m



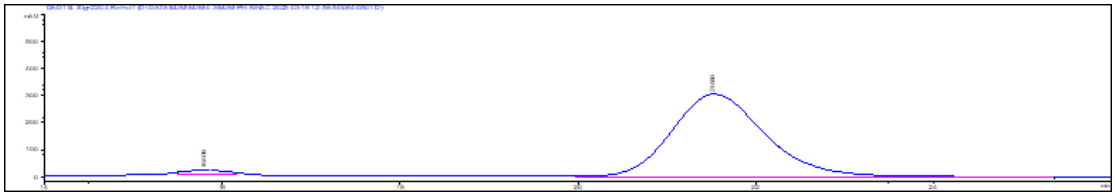
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H



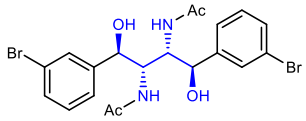
#	Time	Area	Height	Width	Area%	Symmetry
1	16.952	5745.1	76.4	1.2527	51.153	0.659

2	21.833	5486.1	73.5	0.8842	48.847	0.82



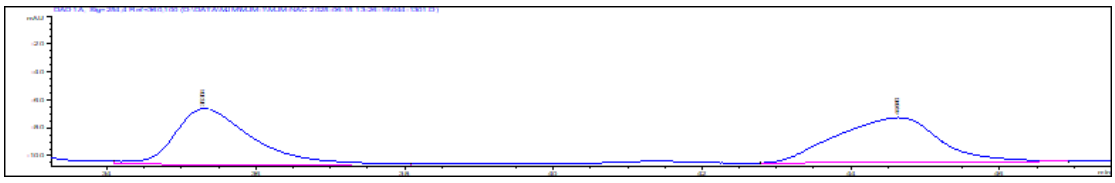
#	Time	Area	Height	Width	Area%	Symmetry
1	15.809	453.7	15.1	0.5011	2.007	0.936
2	21.53	22151.1	304.2	1.0356	97.993	0.777

Supplementary Figure 165. HPLC spectra for compound 2n

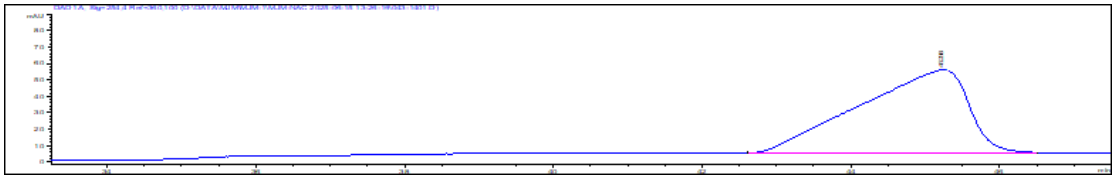


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 254 nm, Chiral AD-H

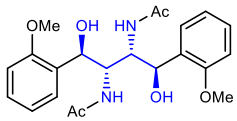


#	Time	Area	Height	Width	Area%	Symmetry
1	35.304	2994.3	40	1.2463	49.481	0.573
2	44.64	3057.1	32	1.1298	50.519	1.454



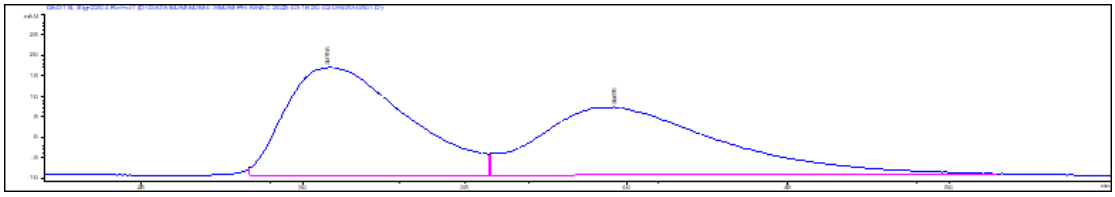
#	Time	Area	Height	Width	Area%	Symmetry
1	45.236	5208.3	50.8	1.7082	100.000	2.977

Supplementary Figure 166. HPLC spectra for compound 2o



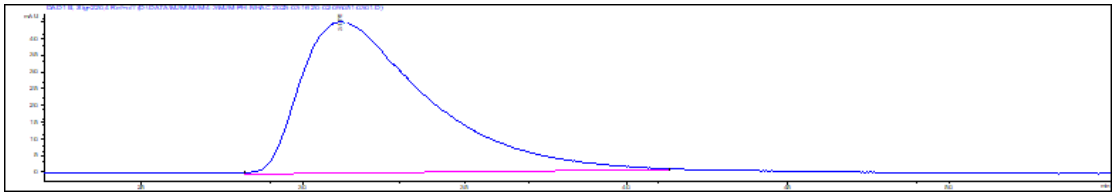
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AS-H



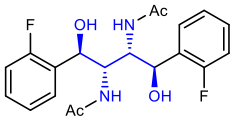
#	Time	Area	Height	Width	Area%	Symmetry
1	30.786	6825.6	26.3	4.3216	51.436	0.535

2	39.676	6444.5	16.5	6.4942	48.564	0.692



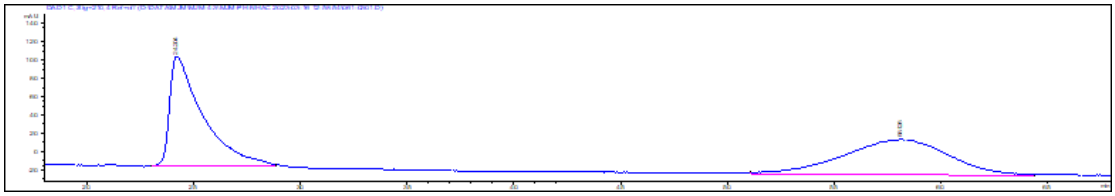
#	Time	Area	Height	Width	Area%	Symmetry
1	31.175	12355.9	45.2	4.56	100.000	0.455

Supplementary Figure 167. HPLC spectra for compound 2p

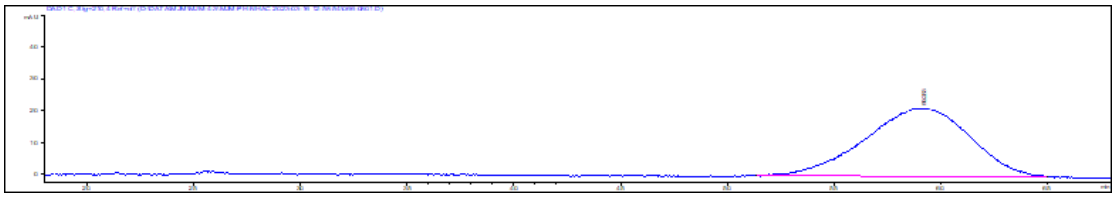


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 210 nm, Chiral AD-H

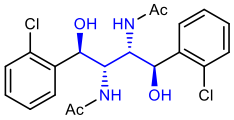


#	Time	Area	Height	Width	Area%	Symmetry
1	24.204	12830.6	119.8	1.2691	49.385	0.279
2	58.126	13150	38.5	5.697	50.615	1.148



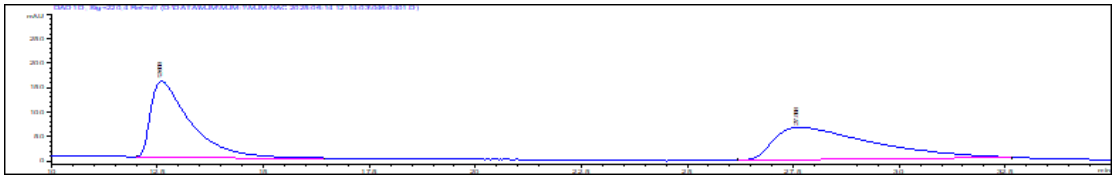
#	Time	Area	Height	Width	Area%	Symmetry
1	59.283	7281	21.4	5.6785	100.000	1.292

Supplementary Figure 168. HPLC spectra for compound 2q

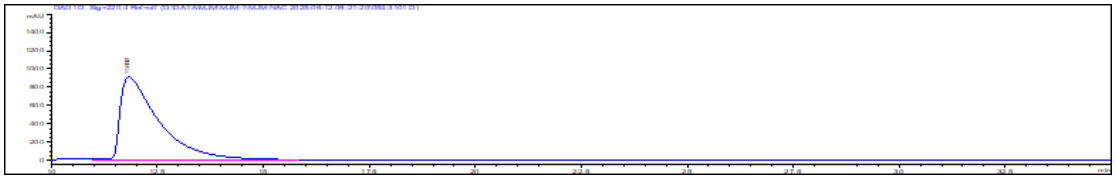


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral OD-H

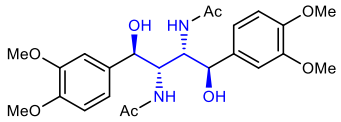


#	Time	Area	Height	Width	Area%	Symmetry
1	12.609	10252.1	155.8	1.0964	49.139	0.376
2	27.598	10611.2	66.3	1.8702	50.861	0.311



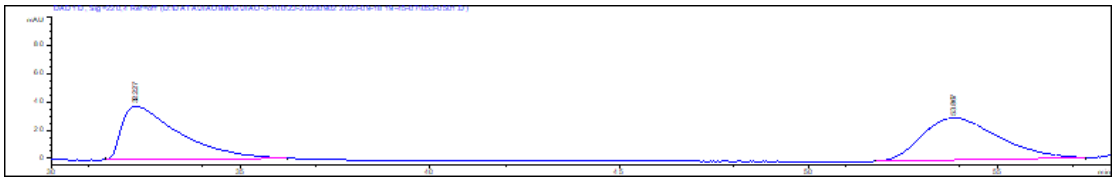
#	Time	Area	Height	Width	Area%	Symmetry
1	11.832	58041.1	906.4	0.753	100.000	0.286

Supplementary Figure 169. HPLC spectra for compound 2r

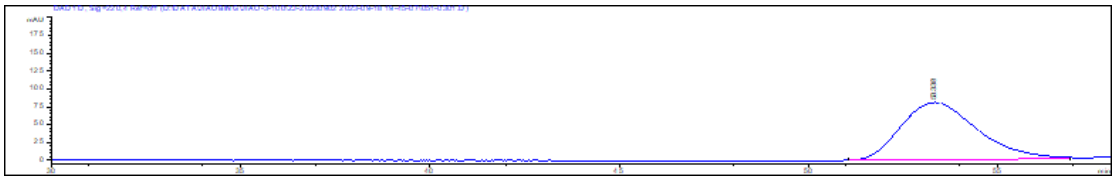


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

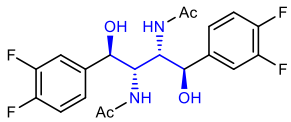


#	Time	Area	Height	Width	Area%	Symmetry
1	32.227	3948.4	37.9	1.2307	48.671	0.306
2	53.867	4163.9	29.9	1.6307	51.329	0.73



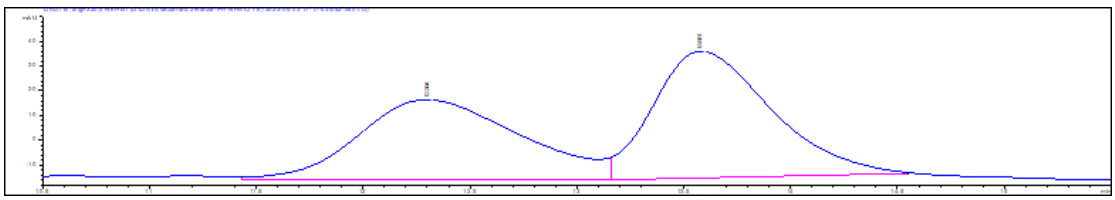
#	Time	Area	Height	Width	Area%	Symmetry
1	53.338	11196.9	79.9	2.3358	100.000	0.772

Supplementary Figure 170. HPLC spectra for compound 2s

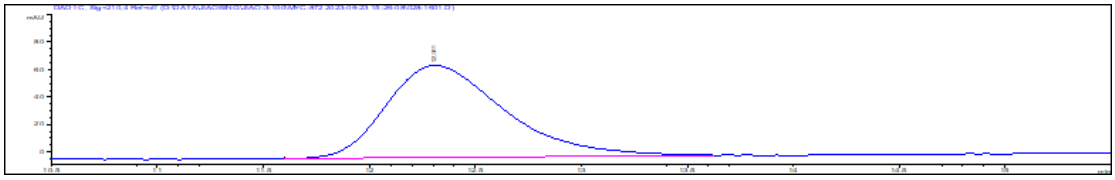


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 210 nm, Chiral AD-H

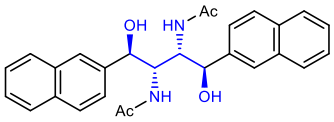


#	Time	Area	Height	Width	Area%	Symmetry
1	12.304	1709.8	32.3	0.8826	46.589	0.745
2	13.581	1960.2	51.1	0.6387	53.411	0.668



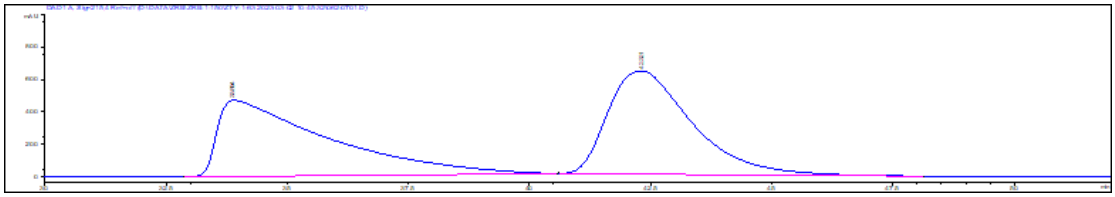
#	Time	Area	Height	Width	Area%	Symmetry
1	12.311	2601.8	67	0.4691	100.000	0.706

Supplementary Figure 171. HPLC spectra for compound 2t

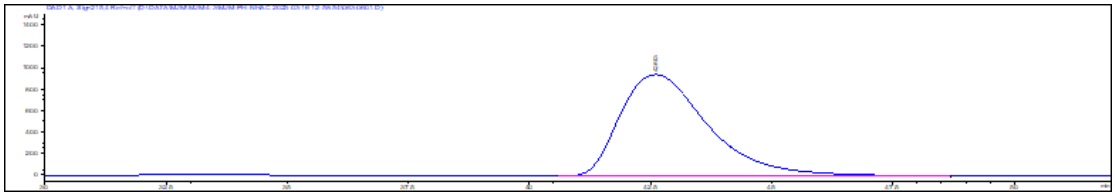


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

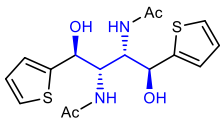


#	Time	Area	Height	Width	Area%	Symmetry
1	33.894	74356.9	467.3	1.9724	49.253	0.159
2	42.321	76610.9	635.5	1.4204	50.747	0.652



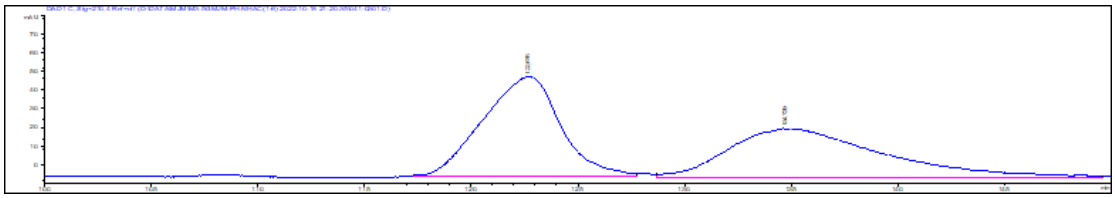
#	Time	Area	Height	Width	Area%	Symmetry
1	42.613	119850.2	947.2	1.4902	100.000	0.667

Supplementary Figure 172. HPLC spectra for compound 2u

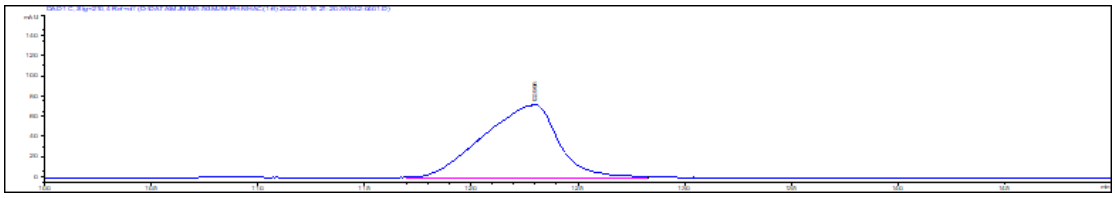


Condition hexane: *i*-PrOH = 92:8,

Flow rate = 0.5 ml/min, λ = 220 nm, Chiral AD-H

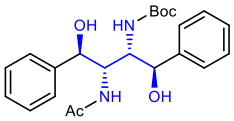


#	Time	Area	Height	Width	Area%	Symmetry
1	122.676	13386.3	53.3	4.1826	49.957	1.31
2	134.729	13409.4	26.4	8.4522	50.043	0.573



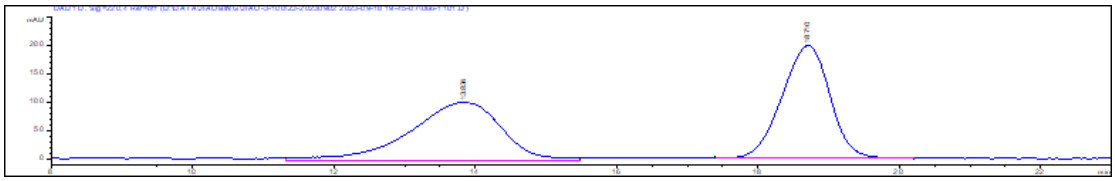
#	Time	Area	Height	Width	Area%	Symmetry
1	122.996	17572	72.1	4.0638	100.000	2.061

Supplementary Figure 173. HPLC spectra for compound 2v

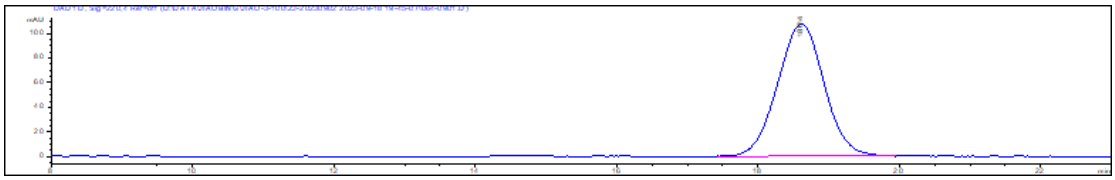


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

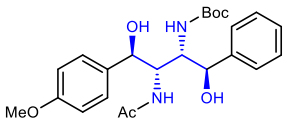


#	Time	Area	Height	Width	Area%	Symmetry
1	13.834	9020.2	101.4	1.4827	49.027	1.379
2	18.71	9378.3	198.5	0.7392	50.973	1.157



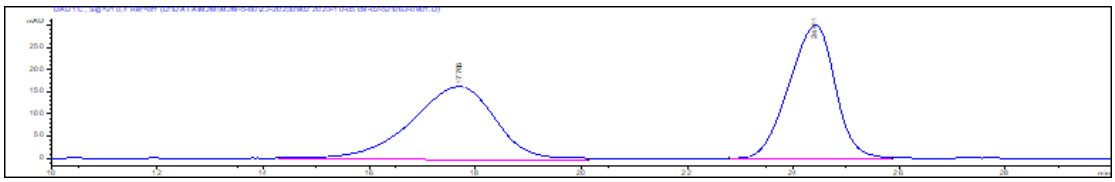
#	Time	Area	Height	Width	Area%	Symmetry
1	18.614	4907.7	107.1	0.7107	100.000	1.015

Supplementary Figure 174. HPLC spectra for compound 2w

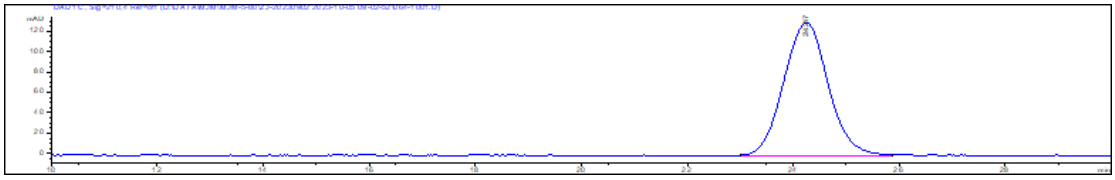


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 210 nm, Chiral AD-H

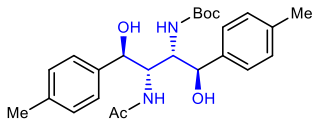


#	Time	Area	Height	Width	Area%	Symmetry
1	17.705	18447.2	165.7	1.8553	50.358	1.314
2	24.411	18184.7	300.6	0.7998	49.642	1.286



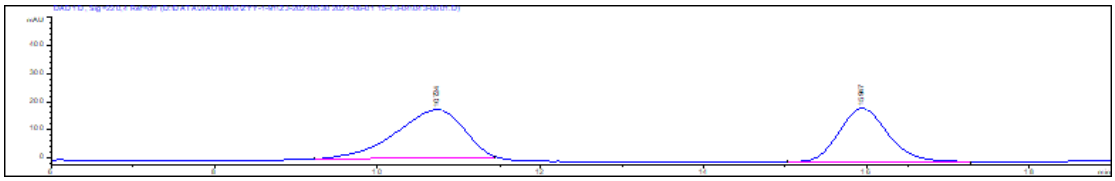
#	Time	Area	Height	Width	Area%	Symmetry
1	24.247	7565.4	130	0.7051	100.000	1.033

Supplementary Figure 175. HPLC spectra for compound 2x

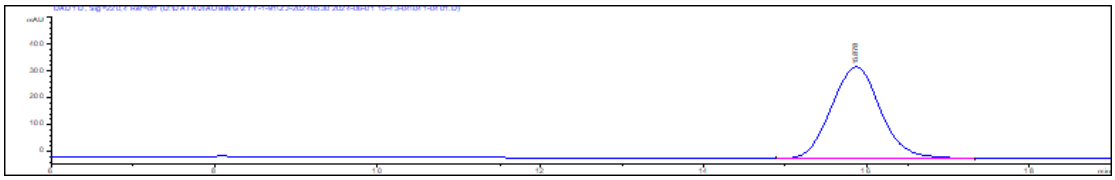


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H

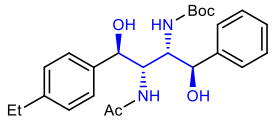


#	Time	Area	Height	Width	Area%	Symmetry
1	10.724	9900.1	171.2	0.9639	54.382	1.514
2	15.947	8304.7	193.5	0.7154	45.618	0.888



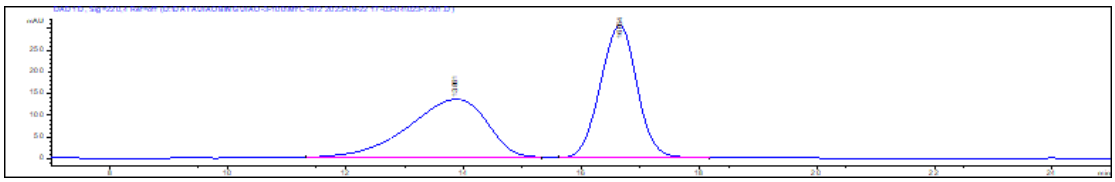
#	Time	Area	Height	Width	Area%	Symmetry
1	15.878	14277.4	341	0.6205	100.000	0.999

Supplementary Figure 176. HPLC spectra for compound 2y

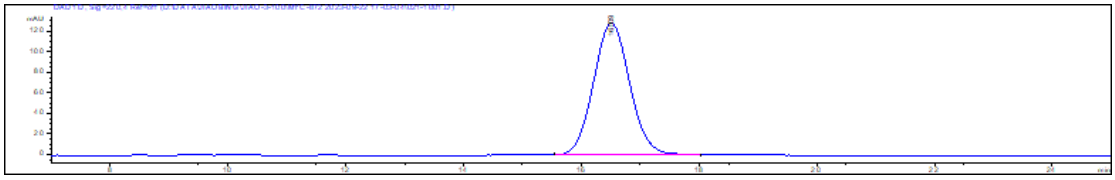


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

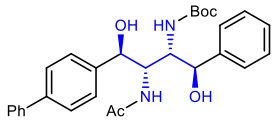


#	Time	Area	Height	Width	Area%	Symmetry
1	13.861	12706.1	135	1.569	49.049	1.474
2	16.654	13198.6	305.4	0.6642	50.951	1.083



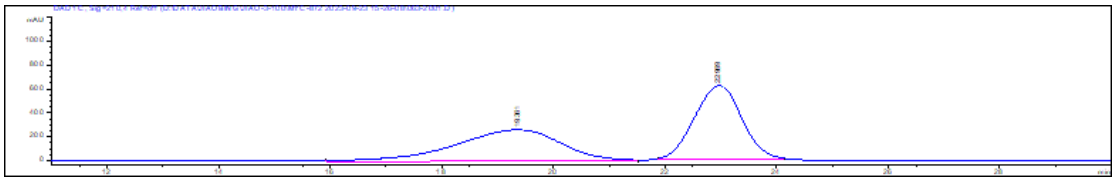
#	Time	Area	Height	Width	Area%	Symmetry
1	16.509	5466.2	128.1	0.658	100.000	0.935

Supplementary Figure 177. HPLC spectra for compound 2z

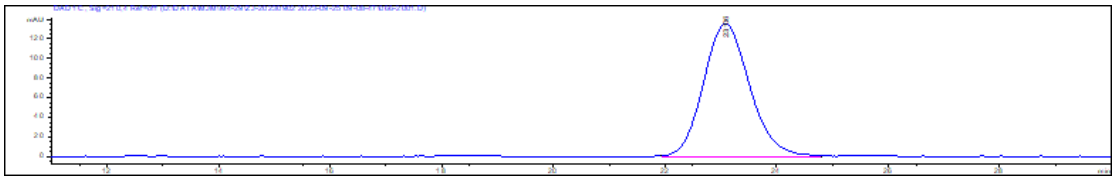


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 210 nm, Chiral AD-H

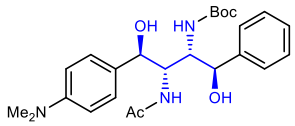


#	Time	Area	Height	Width	Area%	Symmetry
1	19.361	34633.7	261.3	2.2095	49.147	1.437
2	22.969	35835.6	613.5	0.9735	50.853	1.055



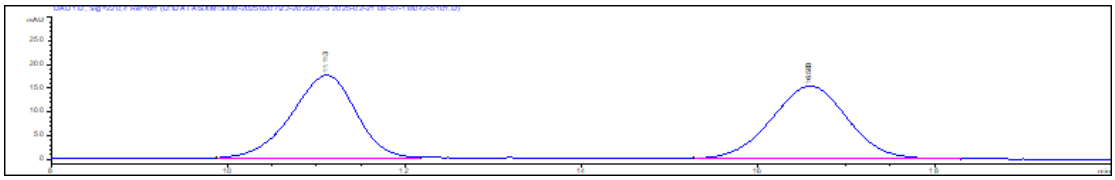
#	Time	Area	Height	Width	Area%	Symmetry
1	23.106	7731.4	134.7	0.7079	100.000	0.978

Supplementary Figure 178. HPLC spectra for compound 2aa



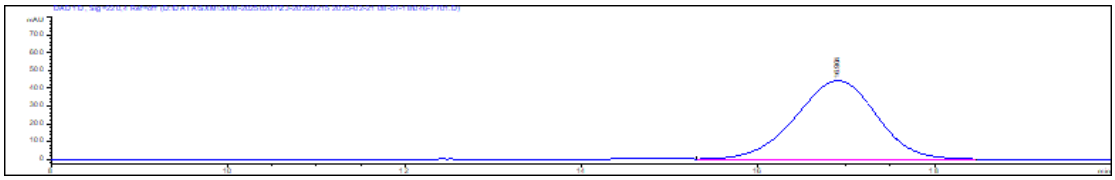
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 215 nm, Chiral AD-H



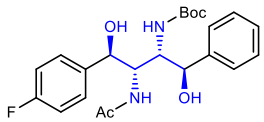
#	Time	Area	Height	Width	Area%	Symmetry
1	11.113	8963.7	175.6	0.6237	49.445	1.13

2	16.583	9164.9	153.4	0.7029	50.555	0.976

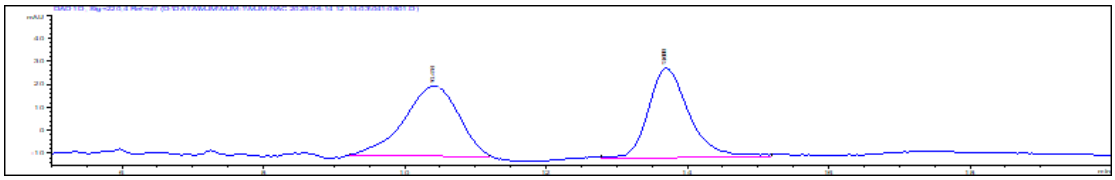


#	Time	Area	Height	Width	Area%	Symmetry
1	16.904	28206.5	443.4	0.7701	100.000	1.079

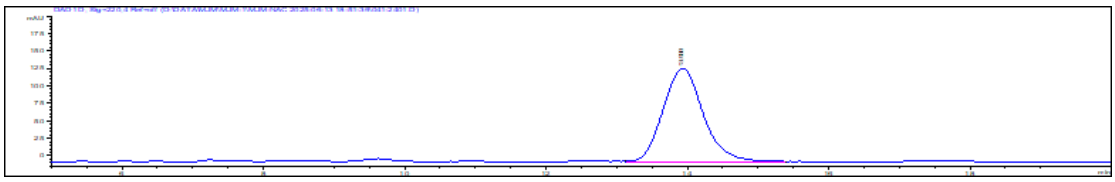
Supplementary Figure 179. HPLC spectra for compound 2ab



Condition hexane: *i*-PrOH = 90:10,
Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

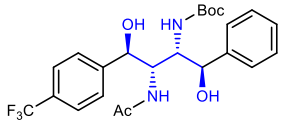


#	Time	Area	Height	Width	Area%	Symmetry
1	10.418	1631.8	30.5	0.8909	51.100	1.205
2	13.699	1561.5	38.9	0.6691	48.900	0.734

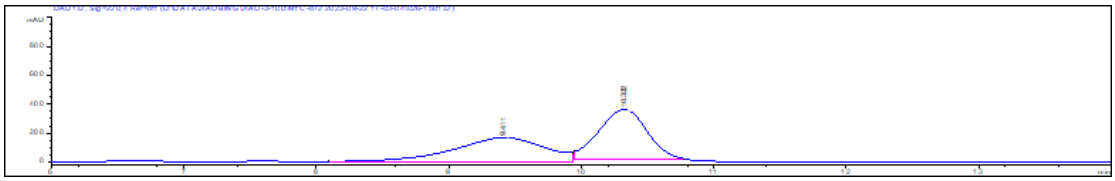


#	Time	Area	Height	Width	Area%	Symmetry
1	13.939	5265.1	133.3	0.5067	100.000	0.94

Supplementary Figure 180. HPLC spectra for compound 2ac

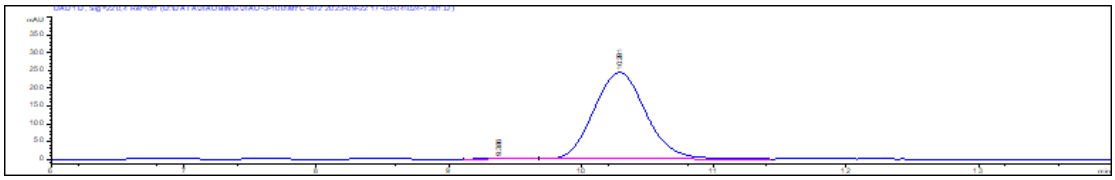


Condition hexane: *i*-PrOH = 90:10,
Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



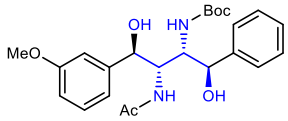
#	Time	Area	Height	Width	Area%	Symmetry
1	9.411	8087.7	168.4	0.8002	47.961	1.228

2	10.322	8775.3	346.4	0.4222	52.039	1.084



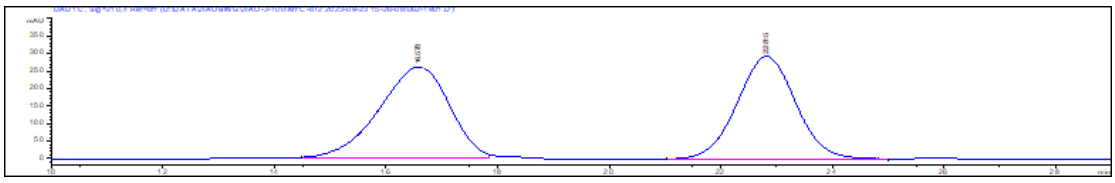
#	Time	Area	Height	Width	Area%	Symmetry
1	9.386	24.2	1.3	0.2229	0.362	1.044
2	10.291	6655.2	244.3	0.4238	99.638	0.927

Supplementary Figure 181. HPLC spectra for compound 2ad

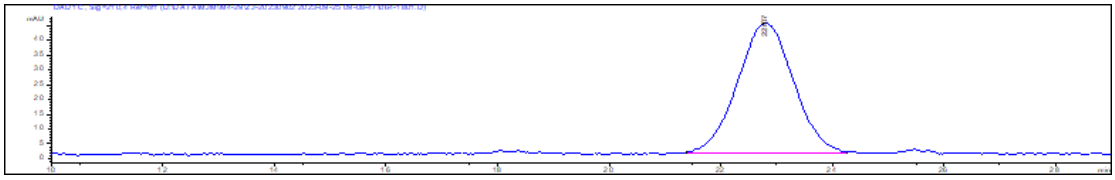


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 210 nm, Chiral AD-H

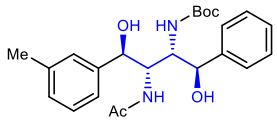


#	Time	Area	Height	Width	Area%	Symmetry
1	16.578	22776.8	260.1	1.4592	51.500	1.242
2	22.815	21449.6	293.4	0.9708	48.500	0.97



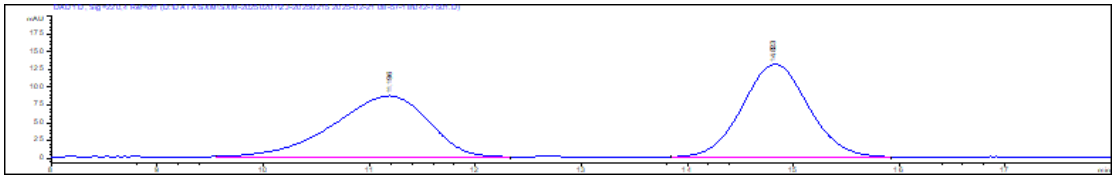
#	Time	Area	Height	Width	Area%	Symmetry
1	22.807	3057.7	43.9	0.8205	100.000	1.004

Supplementary Figure 182. HPLC spectra for compound 2ae



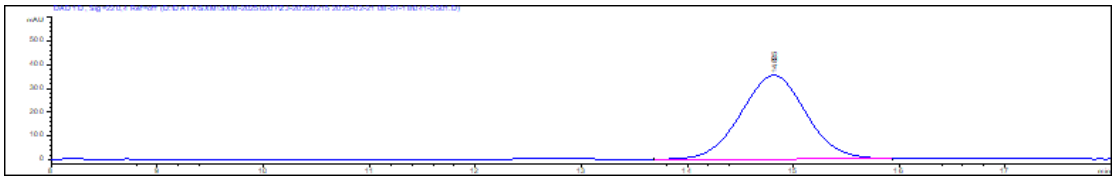
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



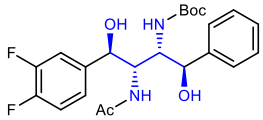
#	Time	Area	Height	Width	Area%	Symmetry
1	11.196	5426.4	86.5	0.7442	48.447	1.464

2	14.823	5774.4	131.4	0.5648	51.553	0.934



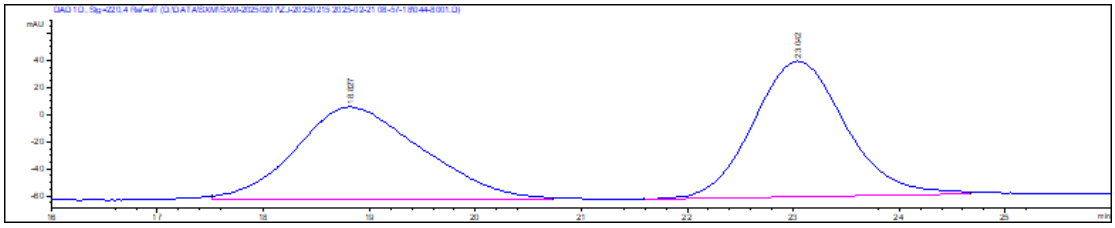
#	Time	Area	Height	Width	Area%	Symmetry
1	14.825	15155.3	353.8	0.6097	100.000	1.037

Supplementary Figure 183. HPLC spectra for compound 2af

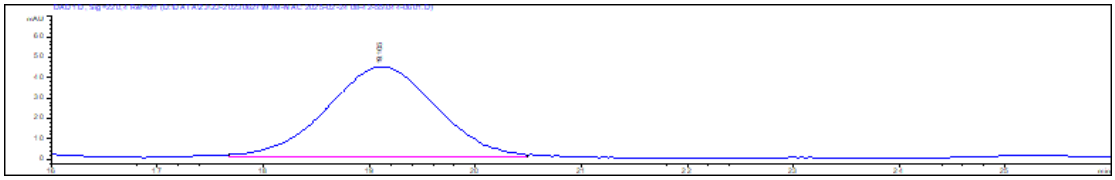


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H

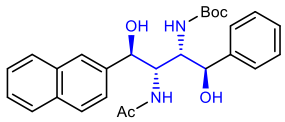


#	Time	Area	Height	Width	Area%	Symmetry
1	18.827	5646.1	68.1	0.9737	48.373	0.768
2	23.042	6025.9	99.8	0.7185	51.627	0.897



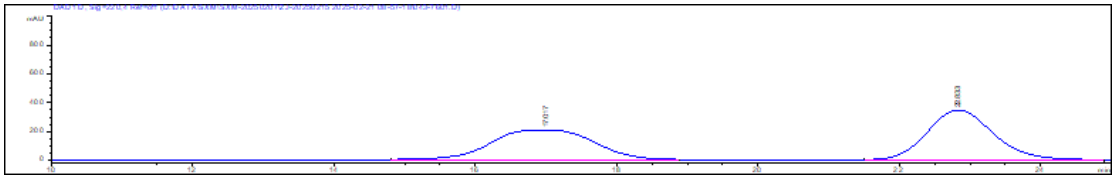
#	Time	Area	Height	Width	Area%	Symmetry
1	19.105	3138.3	44.1	0.8368	100.000	0.954

Supplementary Figure 184. HPLC spectra for compound 2ag



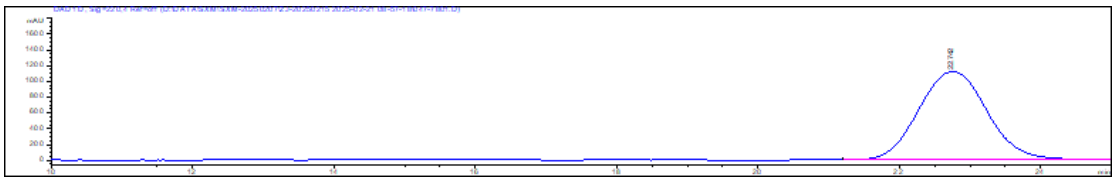
Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



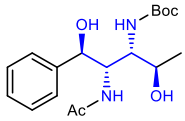
#	Time	Area	Height	Width	Area%	Symmetry

1	17.017	21082.4	208.9	1.1894	48.466	1.149
2	22.833	22417.2	345.6	0.7947	51.534	0.889



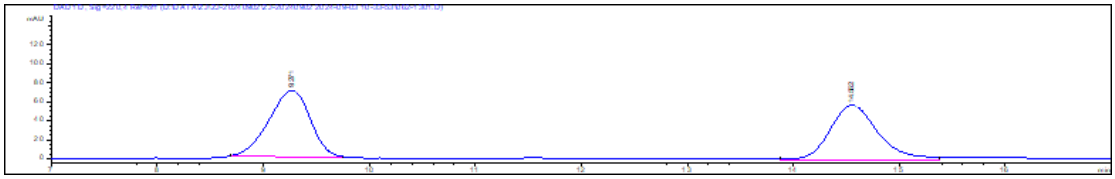
#	Time	Area	Height	Width	Area%	Symmetry
1	22.742	75356.2	1120.3	0.7907	100.000	0.906

Supplementary Figure 185. HPLC spectra for compound 2ah

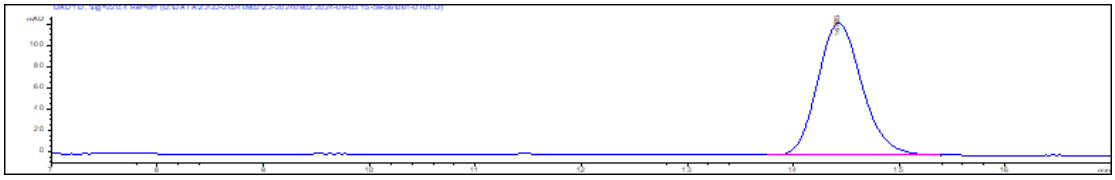


Condition hexane: *i*-PrOH = 90:10,

Flow rate = 1.0 ml/min, λ = 220 nm, Chiral AD-H



#	Time	Area	Height	Width	Area%	Symmetry
1	9.271	1884	69.7	0.4505	50.760	1.246
2	14.552	1827.6	57.5	0.5294	49.240	0.794



#	Time	Area	Height	Width	Area%	Symmetry
1	14.425	3611	124.3	0.4149	100.000	0.852

Supplementary Figure 186. HPLC spectra for compound 2ai