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Electronic Supplementary Information

Palladium/Silver Co-catalyzed *syn*-Stereoselective Asymmetric Ring-Opening Reactions of Azabenzonorbornadienes with Amides

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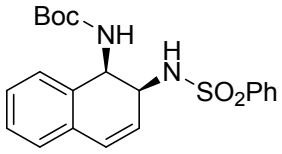
A: General Information

The reactions and manipulations were performed under an atmosphere of argon by using standard Schlenk techniques and Drybox (Mikrouna, Supper 1220/750). Anhydrous DCE (Dichloroethane) was distilled from calcium hydride and stored under argon. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded on Bruker-Avance 400 MHz spectrometer. $CDCl_3$ was used as solvent. Chemical shifts (δ) were reported in ppm with tetramethylsilane as internal standard, and *J* values were given in Hz. The enantioselective excesses were determined by Agilent 1260 Series HPLC using Daicel AD-H、AS-H、OJ-H and OD-H chiral columns eluted with a mixture of isopropyl alcohol and hexane. Melting points were measured on X-4 melting point apparatus and uncorrected. High resolution mass spectra (HRMS) were performed on a VG Autospec-3000 spectrometer. Column chromatography was performed with silica gel (200-300 mesh) with petroleum ether and ethyl acetate as eluents.

B: Procedure for the reactions

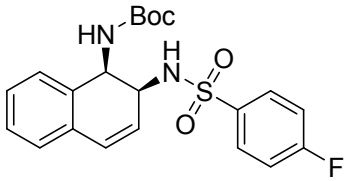
Typical procedure for the asymmetric ring-opening reaction of azabenzonorbornadiene: $Pd(OAc)_2$ (2.3 mg, 0.01 mmol), (*R*)-BINAP (7.5 mg, 0.012 mmol) and 1.0 mL DCE were added to a Schlenk tube under argon atmosphere. The resulting solution was stirred at room temperature for 30 min, then $AgBF_4$ (3.9 mg, 0.02 mmol) was added and stirred for additional 10 min. Azabenzonorbornadiene **1a** (48.6 mg, 0.2 mmol) in DCE (1.0 mL) was added to the above mixture and stirred for additional 10 min. After the addition of benzenesulfonamide **2a** (94.3 mg, 0.6 mmol) the mixture was stirred at 60 °C under argon atmosphere with TLC monitoring until the complete consumption of **1a**. The residue was purified by chromatography on a silica gel column to afford the desired product **3aa** (75.3mg, 94% yield).

C: Characterization Data of Products

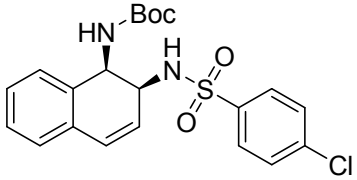


***tert*-butyl ((1*R*,2*S*)-2-(phenylsulfonamido)-1,2-dihydronaphthalen1-yl)carbamate (3aa):** White solid, 75.3 mg, 94% yield, 98% ee. mp 186 - 187 °C. $[\alpha]^{26}_{D}$= +177.7 (c = 0.78, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.6Hz, 2H), 7.63 (t, *J* = 8.0Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 2H), 7.29-7.25 (m, 3H), 7.08 (t, *J* = 4.0 Hz, 1H), 6.45 (d, *J* = 9.6 Hz, 1H), 5.65 (dd, *J* = 9.3, 5.2 Hz, 1H), 5.20 (d, *J* = 9.5 Hz, 1H), 4.97 - 4.90 (m, 2H), 4.25 - 3.78 (m, 1H), 1.51 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1,

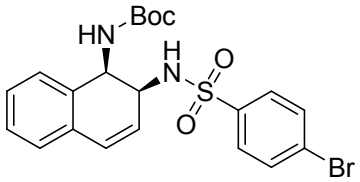
140.4, 133.5, 132.9, 131.9, 129.9, 129.3, 128.8, 128.2, 127.1, 126.4, 125.9, 80.2, 51.3, 51.0, 28.4. HRMS (EI-TOF) calcd for $C_{21}H_{24}N_2O_4S$ $[M]^+$: 400.1462. Found: 400.1457. The ee of **3aa** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 9.3 min, t_{major} = 17.5 min.



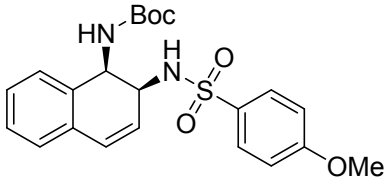
***tert*-butyl((1*R*,2*S*)-2-(4-fluorophenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ab):** White solid, 70.3 mg, 84% yield, 98% ee. mp 195 - 197 °C. $[\alpha]^{26}_D$ = +157.9 (c = 1.10, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 - 7.88 (m, 2H), 7.29 - 7.20 (m, 6H), 7.09 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.48 (d, *J* = 9.6 Hz, 1H), 5.71 (dd, *J* = 9.5, 5.1 Hz, 1H), 5.09 (d, *J* = 9.6 Hz, 1H), 4.89 (t, *J* = 7.4 Hz, 2H), 4.03-3.99 (m, 1H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.4, 163.9, 156.1, 136.6, 133.4, 131.9, 130.1, 129.9, 129.8, 128.8, 128.3, 127.1, 126.3, 126.0, 116.7, 116.4, 80.3, 51.3, 51.2, 28.4. HRMS (EI-TOF) calcd for $C_{21}H_{23}FN_2O_4S$ $[M]^+$: 418.1362. Found: 418.1363. The ee of **3ab** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 7.1 min, t_{major} = 12.3 min.



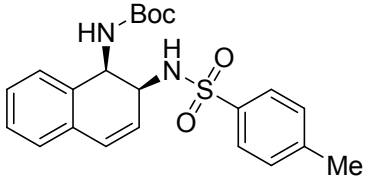
***tert*-butyl((1*R*,2*S*)-2-(4-chlorophenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ac):** White solid, 71.3 mg, 82% yield, 98% ee. mp 189 - 191 °C. $[\alpha]^{26}_D$= +176.4 (c = 0.84, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 - 7.79 (m, 2H), 7.51 -7.49 (m, 2H), 7.29 - 7.23 (m, 4H), 7.09 - 7.07 (m, 1H), 6.47 (d, *J* = 9.6 Hz, 1H), 5.71 (dd, *J* = 9.5, 5.1 Hz, 1H), 5.10 (d, *J* = 9.6 Hz, 1H), 5.01 (d, *J* = 9.0 Hz, 1H), 4.88 (dd, *J* = 9.5, 5.3 Hz, 1H), 4.03 - 3.98 (m, 1H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 139.4, 138.9, 133.3, 131.9, 130.1, 129.6, 128.8, 128.6, 128.3, 127.2, 126.3, 80.3, 51.3, 51.2, 28.4. HRMS (EI-TOF) calcd for $C_{21}H_{23}ClN_2O_4S$ $[M]^+$: 434.1085. Found: 434.1067. The ee of **3ac** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor}= 8.0 min, t_{major} = 15.6 min.



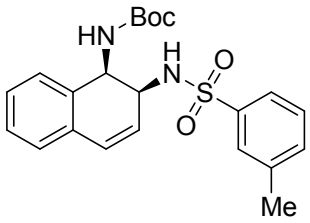
***tert*-butyl((1*R*,2*S*)-2-(4-bromophenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ad):** White solid, 73.8 mg, 77% yield, 98% ee. mp 197 - 199 °C. $[\alpha]^{26}_{D}$= +128.8 (c = 0.08, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 - 7.72 (m, 2H), 7.68 - 7.66 (m, 2H), 7.30 - 7.24 (m, 3H), 7.10 - 7.06 (m, 1H), 6.48 (d, *J* = 9.6 Hz, 1H), 5.72 (dd, *J* = 9.5, 5.1 Hz, 1H), 5.10 (d, *J* = 9.6 Hz, 1H), 4.99 (d, *J* = 9.0 Hz, 1H), 4.89 (dd, *J* = 9.5, 5.3 Hz, 1H), 4.03 - 3.99 (m, 1H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 139.5, 133.3, 132.6, 131.9, 130.1, 128.8, 128.7, 128.4, 127.9, 127.2, 126.3, 126.1, 80.4, 51.3, 28.4. HRMS (EI-TOF) calcd for $C_{21}H_{23}BrN_2O_4S$ $[M]^+$: 478.0558. Found: 478.0562. The ee of **3ad** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 8.7 min, t_{major} = 18.5 min.



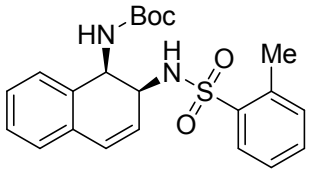
***tert*-butyl((1*R*,2*S*)-2-(4-methoxyphenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ae):** White solid, 80.1 mg, 93% yield, 98% ee. mp 185 - 187 °C. $[\alpha]^{26}_{D}$ = +179.6 (c = 1.00, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 - 7.78 (m, 2H), 7.31 - 7.22 (m, 3H), 7.08 - 7.06 (m, 1H), 7.01 - 6.97 (m, 2H), 6.45 (d, *J* = 9.6 Hz, 1H), 5.66 (dd, *J* = 9.5, 5.3 Hz, 1H), 5.20 (d, *J* = 9.7 Hz, 1H), 4.90 (dd, *J* = 9.6, 5.4 Hz, 1H), 4.75 (d, *J* = 9.2 Hz, 1H), 3.97 - 3.92 (m, 1H), 3.89 (s, 3H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 156.1, 133.6, 131.9, 129.9, 129.3, 128.8, 128.2, 127.0, 126.6, 125.9, 114.4, 80.1, 55.7, 51.3, 50.9, 28.4. HRMS (EI-TOF) calcd for $C_{22}H_{26}N_2O_5S$ $[M]^+$: 430.1568. Found: 430.1562. The ee of **3ae** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 13.1 min, t_{major} = 22.7 min.



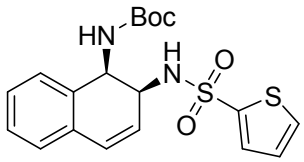
***tert*-butyl((1*R*,2*S*)-2-(4-methylphenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3af):** White solid, 72.1 mg, 87% yield, 98% ee. mp 182 - 184 °C. $[\alpha]^{26}_{D}$= +180.6 (c = 1.02, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 - 7.73 (m, 2H), 7.33 - 7.22 (m, 5H), 7.07 (dd, *J* = 5.7, 2.9 Hz, 1H), 6.44 (d, *J* = 9.6 Hz, 1H), 5.64 (dd, *J* = 9.5, 5.3 Hz, 1H), 5.20 (d, *J* = 9.7 Hz, 1H), 4.90 (dd, *J* = 9.6, 5.4 Hz, 1H), 4.78 (d, *J* = 9.1 Hz, 1H), 3.99 - 3.95 (m, 1H), 2.45 (s, 3H), 1.50 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2, 143.8, 137.4, 133.5, 131.9, 129.9, 128.8, 128.2, 127.1, 127.0, 126.5, 125.9, 80.1, 51.3, 50.9, 28.4, 21.6. HRMS (EI-TOF) calcd for $C_{22}H_{26}N_2O_4S$ $[M]^+$: 414.1620. Found: 414.1613. The ee of **3af** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, 254 nm; t_{minor} = 10.2 min, t_{major} = 18.6 min.



***tert*-butyl((1*R*,2*S*)-2-(3-methylphenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ag):** White solid, 76.3 mg, 92% yield, 98% ee. mp 83 - 85 °C. $[\alpha]^{26}_{D}$ = +154.9 (c = 1.24, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (dd, *J* = 8.8, 4.6 Hz, 2H), 7.41 (d, *J* = 4.6 Hz, 2H), 7.30 – 7.23 (m, 3H), 7.10 - 7.06 (m, 1H), 6.44 (d, *J* = 9.6 Hz, 1H), 5.64 (dd, *J* = 9.4, 5.2 Hz, 1H), 5.20 (d, *J* = 9.4 Hz, 1H), 4.90 - 4.84 (m, 2H), 4.02 - 3.97 (m, 1H), 2.44 (s, 3H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2, 140.2, 139.5, 133.8, 133.5, 131.9, 129.9, 129.2, 128.8, 128.2, 127.4, 127.1, 126.4, 126.0, 124.2, 80.2, 51.3, 51.0, 28.4, 21.8. HRMS (EI-TOF) calcd for $C_{22}H_{26}N_2O_4S$ $[M]^+$: 414.1642. Found: 414.1613. The ee of **3ag** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 8.3 min, t_{major} = 12.4 min.

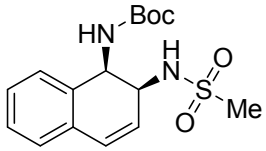


***tert*-butyl((1*R*,2*S*)-2-(2-methylphenylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ah):** White solid, 66.3 mg, 80% yield, 96% ee. mp 93 - 95 °C. $[\alpha]^{26}_{D}$ = +97.5 (c = 1.42, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 - 7.99 (m, 1H), 7.52-7.48 (m, 1H), 7.36 – 7.32 (m, 2H), 7.29 - 7.24 (m, 3H), 7.11 – 7.07 (m, 1H), 6.47 (d, *J* = 9.6 Hz, 1H), 5.67 (dd, *J* = 9.6, 5.1 Hz, 1H), 5.15 (d, *J* = 9.6 Hz, 1H), 4.93 (d, *J* = 9.2 Hz, 1H), 4.87 (dd, *J* = 9.5, 5.3 Hz, 1H), 3.96 – 3.91 (m, 1H), 2.59 (s, 3H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 137.9, 136.9, 133.5, 133.2, 131.9, 129.8, 128.8, 128.3, 127.1, 126.6, 126.4, 126.2, 80.2, 51.4, 51.1, 28.4, 20.3. HRMS (EI-TOF) calcd for $C_{22}H_{26}N_2O_4S$ $[M]^+$: 414.1618. Found: 414.1613. The ee of **3ah** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 8.4 min, t_{major} = 13.8 min.

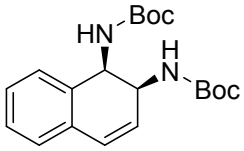


***tert*-butyl((1*R*,2*S*)-2-(thiophene-2-sulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3ai):** White solid, 73.2 mg, 90% yield, 98% ee. mp 177 - 179 °C.

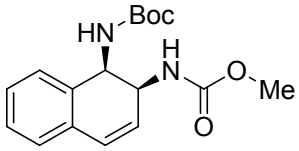
$[\alpha]^{26}_D$ = +237.5 (c = 1.26, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (dd, *J* = 3.4, 2.0 Hz, 2H), 7.32 - 7.25 (m, 3H), 7.12 - 7.07 (m, 2H), 6.49 (d, *J* = 9.6 Hz, 1H), 5.73 (dd, *J* = 9.5, 5.2 Hz, 1H), 5.20 (d, *J* = 9.5 Hz, 1H), 5.01 (d, *J* = 9.0 Hz, 1H), 4.93 (dd, *J* = 9.4, 5.3 Hz, 1H), 4.12 -4.07 (m, 1H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 141.4, 133.5, 132.5, 132.4, 131.9, 130.2, 128.8, 128.3, 127.6, 127.1, 126.1, 126.0, 80.2, 51.5, 51.2, 28.4. HRMS (EI-TOF) calcd for $C_{19}H_{22}N_2O_4S_2[M]^+$: 406.1019. Found: 406.1021. The ee of **3ai** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 10.4 min, t_{major} = 19.9 min.



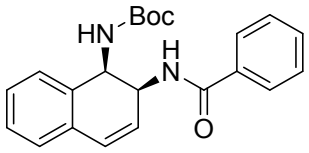
***tert*-butyl((1*R*,2*S*)-2-(methylsulfonamido)-1,2-dihydronaphthalen-1-yl)carbamate (3aj):** White solid, 48.7 mg, 72% yield, 98% ee. mp 114 - 117°C. $[\alpha]^{26}_D$ = +163.4 (c = 0.64, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 8.3, 4.8 Hz, 1H), 7.30 - 7.24 (m, 2H), 7.14 - 7.11 (m, 1H), 6.59 (d, *J* = 9.6 Hz, 1H), 6.10 (dd, *J* = 9.5, 4.9 Hz, 1H), 5.22 (d, *J* = 9.7 Hz, 1H), 5.01 (dd, *J* = 9.5, 5.5 Hz, 1H), 4.82 (d, *J* = 9.4 Hz, 1H), 4.24 - 4.19 (m, 1H), 2.98 (s, 3H), 1.48 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.0, 133.4, 131.9, 130.1, 128.8, 128.4, 127.2, 126.9, 126.2, 80.3, 51.5, 51.2, 41.6, 28.4. HRMS (EI-TOF) calcd for $C_{16}H_{22}N_2O_4S$ $[M]^+$: 338.1297. Found: 338.1300. The ee of **3aj** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, 254 nm; t_{minor} = 10.9 min, t_{major} = 12.4 min.



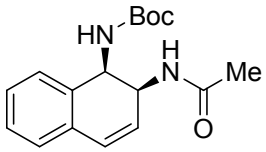
di-*tert*-butyl ((1*R*,2*S*)-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3ak): White solid, 96% yield, 69.2 mg, 95% ee. mp 98 - 100 °C. $[\alpha]^{26}_D$ = +60.7 (c = 0.96, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 3.5 Hz, 1H), 7.28 - 7.22 (m, 2H), 7.10 (dd, *J* = 5.7, 3.0 Hz, 1H), 6.54 (d, *J* = 9.7 Hz, 1H), 5.98 (dd, *J* = 9.5, 4.5 Hz, 1H), 5.01 (s, 2H), 4.72 (d, *J* = 8.2 Hz, 1H), 4.57 (s, 1H), 1.46 (d, *J* = 13.8 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): 155.8, 155.6, 134.1, 132.2, 129.1, 128.3, 128.1, 127.9, 126.9, 126.7, 79.8, 51.3, 48.1, 28.4, 28.3. HRMS (EI-TOF) calcd for $C_{20}H_{28}N_2O_4$ $[M]^+$: 360.2056. Found: 360.2049. The ee of **3ak** was determined by HPLC analysis using Daicel Chiralcel OD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 98/2, 0.5 mL/min, 254 nm; t_{minor} = 24.6 min, t_{major} = 31.7 min.



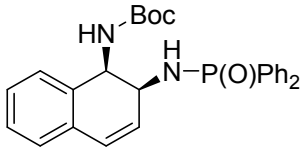
***tert*-butyl methyl ((1*R*,2*S*)-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3al):** White solid, 59.9 mg, 94% yield, 90% ee. mp 169 - 171 °C. $[\alpha]^{26}_{D}$ = +61.7 (c = 0.58, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (s, 1H), 7.30 - 7.24 (m, 2H), 7.12 (d, *J* = 7.9 Hz, 1H), 6.56 (d, *J* = 9.6 Hz, 1H), 5.99 (dd, *J* = 9.0, 3.6 Hz, 1H), 5.08 - 4.78 (m, 3H), 4.57 (d, *J* = 42.5 Hz, 1H), 3.71 (d, *J* = 23.8 Hz, 3H), 1.48 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 155.9, 134.0, 132.1, 129.2, 128.4, 128.3, 127.8, 126.9, 126.8, 80.0, 52.3, 51.3, 49.2, 28.4. HRMS (EI-TOF) calcd for $C_{17}H_{22}N_2O_4$ $[M]^+$: 318.1592. Found: 318.1580. The ee of **3al** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{major} = 12.3min, t_{minor} = 14.5 min.



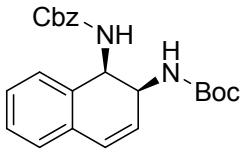
***tert*-butyl ((1*R*,2*S*)-2-benzamido-1,2-dihydronaphthalen-1-yl)carbamate (3am):** White solid, 66.3 mg, 91% yield, 90% ee. mp 188 - 189 °C. $[\alpha]^{26}_{D}$ = +15.5 (c = 0.10, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.37 - 7.28 (m, 3H), 7.17 (d, *J* = 7.1 Hz, 2H), 6.60 (d, *J* = 9.4 Hz, 1H), 6.09 (d, *J* = 8.5 Hz, 1H), 5.08 - 5.01 (m, 3H), 1.48 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 134.2, 131.6, 128.8, 128.4, 128.3, 127.7, 127.1, 127.0, 80.4, 51.6, 50.2, 28.3. HRMS (EI-TOF) calcd for $C_{22}H_{24}N_2O_3$ $[M]^+$: 364.1794. Found: 364.1787. The ee of **3am** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 254 nm; t_{minor} = 11.3 min, t_{major} = 17.7 min.



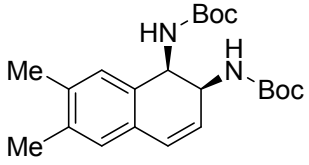
***tert*-butyl ((1*R*,2*S*)-2-acetamido-1,2-dihydronaphthalen-1-yl)carbamate (3an):** White solid, 53.8 mg, 89% yield, 91% ee. mp 202 - 204 °C. $[\alpha]^{26}_{D}$ = +34.9 (c = 0.72, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.25 (m, 3H), 7.13 (d, *J* = 6.9 Hz, 1H), 6.56 (d, *J* = 9.6 Hz, 1H), 6.08 (d, *J* = 7.0 Hz, 1H), 5.95 (d, *J* = 6.4 Hz, 1H), 5.08 (d, *J* = 8.3 Hz, 1H), 4.93 - 4.88 (m, 2H), 1.99 (s, 3H), 1.47 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 156.2, 133.9, 132.2, 129.0, 128.4, 128.4, 128.1, 127.1, 126.9, 80.1, 51.5, 48.2, 28.3, 23.5. HRMS (EI-TOF) calcd for $C_{17}H_{22}N_2O_3$ $[M]^+$: 302.1634. Found: 302.1630. The ee of **3an** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 0.5 mL/min, 254 nm; t_{minor} = 9.6 min, t_{major} =13.3 min.



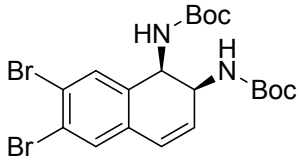
***tert*-butyl((1*R*,2*S*)-2-((diphenylphosphoryl)amino)-1,2-dihydronaphthalen-1-yl)carbamate (3ao):** White solid, 60.8 mg, 66% yield, 99% ee. mp 116 - 118 °C. $[\alpha]^{26}_D$ = +67.5 (c = 0.32, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 - 7.88 (m, 4H), 7.55 - 7.36 (m, 7H), 7.31 - 7.21 (m, 2H), 7.09 - 7.07 (m, 1H), 6.67 (d, *J* = 9.5 Hz, 1H), 6.54 (d, *J* = 9.6 Hz, 1H), 6.17 (dd, *J* = 9.5, 5.3 Hz, 1H), 4.97 (dd, *J* = 9.5, 5.0 Hz, 1H), 3.77 - 3.69 (m, 1H), 3.03 (dd, *J* = 11.3, 6.8 Hz, 1H), 1.49 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 134.4, 132.5, 132.4, 132.3, 132.2, 129.1, 128.9, 128.8, 128.7, 128.7, 128.6, 128.5, 127.8, 127.7, 126.7, 126.5, 79.5, 52.7, 49.9, 28.6. ^{31}P NMR ($CDCl_3$): δ 23.46. HRMS (EI-TOF) calcd for $C_{27}H_{29}N_2O_3P$ $[M]^+$: 460.1923. Found: 460.1916. The ee of **3ao** was determined by HPLC analysis using Daicel Chiralcel OD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, 254 nm; t_{minor} = 6.1 min, t_{major} = 9.9 min.



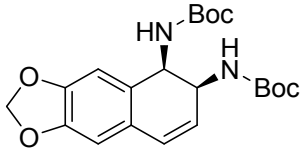
benzyl *tert*-butyl ((1*R*,2*S*)-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3bk): White solid, 95% yield, 96% ee. mp 174 - 176 °C. $[\alpha]^{24}_D$ = +72.3 (c = 0.52, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 - 7.30 (m, 6H), 7.26 - 7.23 (m, 2H), 7.10 (dd, *J* = 7.0, 1.7 Hz, 1H), 6.54 (d, *J* = 9.6 Hz, 1H), 5.98 (dd, *J* = 9.4, 4.3 Hz, 1H), 5.34 (d, *J* = 9.3 Hz, 1H), 5.15 (s, 2H), 5.05 (dd, *J* = 9.5, 5.4 Hz, 1H), 4.72 (s, 1H), 4.59 (s, 1H), 1.42 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.5, 155.7, 136.3, 133.8, 132.1, 129.3, 128.6, 128.4, 128.3, 128.2, 128.2, 127.7, 126.9, 126.6, 80.0, 67.1, 52.2, 48.2, 28.3. HRMS (EI-TOF) calcd for $C_{23}H_{26}N_2O_4$ $[M]^+$: 394.1896. Found: 394.1893. The ee of **3bk** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 240 nm; t_{minor} = 8.7 min, t_{major} = 14.6 min.



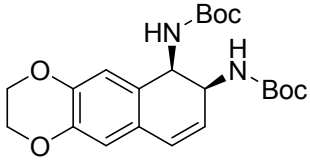
di-*tert*-butyl ((1*R*,2*S*)-6,7-dimethyl-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3ck): White solid, 73.8 mg, 95% yield, 96% ee. mp 179 - 180 °C. $[\alpha]^{24}_D$ = +64.6 (c = 1.48, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.09 (s, 1H), 6.87 (s, 1H), 6.46 (d, *J* = 9.6 Hz, 1H), 5.88 (dd, *J* = 9.2, 4.0 Hz, 1H), 4.93 (s, 2H), 4.73 (d, *J* = 8.3 Hz, 1H), 4.53 (s, 1H), 2.23 (d, *J* = 8.5 Hz, 6H), 1.45 (d, *J* = 16.7 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.9, 155.6, 136.8, 136.3, 131.4, 129.9, 128.9, 128.2, 128.1, 126.9, 79.7, 51.1, 48.4, 28.4, 28.3, 19.8, 19.4. HRMS (EI-TOF) calcd for $C_{22}H_{32}N_2O_4$ $[M]^+$: 388.2368. Found: 388.2362. The ee of **3ck** was determined by HPLC analysis using Daicel ChiralcelAD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, 240 nm; t_{minor} = 5.7 min, t_{major} = 7.4 min.



di-*tert*-butyl ((1*R*,2*S*)-6,7-dibromo-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3dk): White solid, 78% yield, 91% ee. mp 89 - 91 °C. $[\alpha]^{24}_{D}$ = +49.0 (c = 0.62, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (s, 1H), 7.34 (s, 1H), 6.44 (d, *J* = 9.6 Hz, 1H), 6.09 (dd, *J* = 9.4, 4.4 Hz, 1H), 5.03 (d, *J* = 8.6 Hz, 1H), 4.94 - 4.91(m, 1H), 4.55 (d, *J* = 9.8 Hz, 2H), 1.45 (d, *J* = 21.2 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 155.5, 134.9, 132.9, 131.7, 131.4, 129.5, 127.4, 124.1, 124.0, 80.3, 80.2, 50.9, 47.2, 28.4, 28.3. HRMS (EI-TOF) calcd for $C_{20}H_{26}N_2O_4Br_2$ $[M]^+$: 516.0251. Found: 516.0259. The ee of **3dk** was determined by HPLC analysis using Daicel Chiralcel AD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 0.5 mL/min, 240 nm; t_{minor} = 9.4 min, t_{major} = 10.5 min.

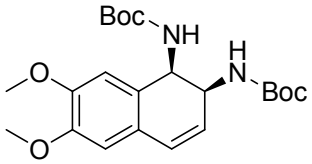


di-*tert*-butyl((5*R*,6*S*)-5,6-dihydronaphtho[2,3-d][1,3]dioxole-5,6-diyl)dicarbamate (3ek): White solid, 78% yield, 96% ee. mp 120 - 122 °C. $[\alpha]^{24}_{D}$ = +38.3 (c = 0.80, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.84 (s, 1H), 6.57 (s, 1H), 6.38 (d, *J* = 9.7 Hz, 1H), 5.92 (d, *J* = 6.8 Hz, 2H), 5.83 (dd, *J* = 9.4, 4.1 Hz, 1H), 4.98 (d, *J* = 8.8 Hz, 1H), 4.91 - 4.80 (m, 1H), 4.75 (d, *J* = 7.8 Hz, 1H), 4.50 (s, 1H), 1.44 (d, *J* = 8.3 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.8, 155.7, 147.3, 147.2, 128.8, 128.6, 126.4, 126.0, 107.9, 107.4, 79.9, 51.4, 48.4, 28.4, 28.3. HRMS (EI-TOF) calcd for $C_{21}H_{28}N_2O_6$ $[M]^+$: 404.1944. Found: 404.1947. The ee of **3ek** was determined by HPLC analysis using Daicel ChiralcelAD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 240 nm; t_{minor} = 6.4 min, t_{major} = 7.6 min.



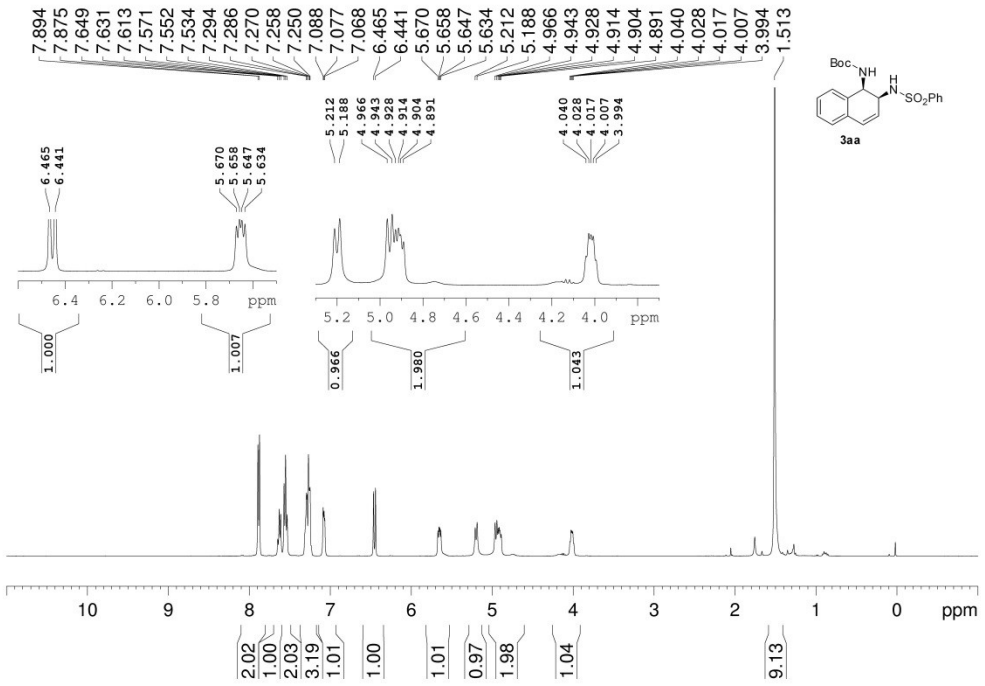
di-*tert*-butyl((6*R*,7*S*)-2,3,6,7-tetrahydronaphtho[2,3-b][1,4]dioxine-6,7-diyl)dicarbamate (3fk): White solid, 92% yield, 96% ee. mp 91 - 93 °C. $[\alpha]^{24}_{D}$ = +57.8 (c = 0.46, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.84 (s, 1H), 6.61 (s, 1H), 6.39 (d, *J* = 9.6 Hz, 1H), 5.85 (dd, *J* = 9.5, 4.6 Hz, 1H), 4.95 - 4.86 (m, 2H), 4.68 (d, *J* = 8.2 Hz, 1H), 4.48 (s, 1H), 4.23 (s, 4H), 1.44 (d, *J* = 12.9 Hz, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.8, 143.3, 142.9, 128.5, 127.7, 126.1, 126.0, 116.1, 115.8, 79.8, 64.5, 64.4, 51.0, 48.1, 28.4, 28.3. HRMS (EI-TOF) calcd for $C_{22}H_{30}N_2O_6$ $[M]^+$: 418.2102. Found: 418.2104. The ee of **3fk** was determined by HPLC analysis using

Daicel ChiralcelAD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 240 nm; t_{minor} = 7.2 min, t_{major} = 9.9 min.



di-*tert*-butyl((1*R*,2*S*)-6,7-dimethoxy-1,2-dihydronaphthalene-1,2-diyl)dicarbamate (3gk): White solid, 69.0 mg, 82% yield, 97% ee. mp 91 - 93 °C. $[\alpha]^{24}_{D}$ = +4.4 (c = 1.34, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.81 (s, 1H), 6.56 (s, 1H), 6.36 (d, *J* = 9.5 Hz, 1H), 5.76-5.74 (m, 1H), 4.88 - 4.72 (m, 3H), 4.48 (s, 1H), 3.80 (d, *J* = 6.5 Hz, 6H), 1.39 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.9, 155.7, 148.8, 148.6, 128.6, 127.0, 126.1, 124.9, 110.6, 110.2, 79.8, 56.1, 56.1, 51.1, 48.8, 28.4. HRMS (EI-TOF) calcd for $C_{22}H_{32}N_2O_6[M]^+$: 420.2248. Found: 420.2260. The ee of **3gk** was determined by HPLC analysis using Daicel Chiralcel OD-H columns (25 cm × 0.46 cm ID), conditions: *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, 240 nm; t_{minor} = 5.0 min, t_{major} = 6.7 min.

D: NMR Spectra of Products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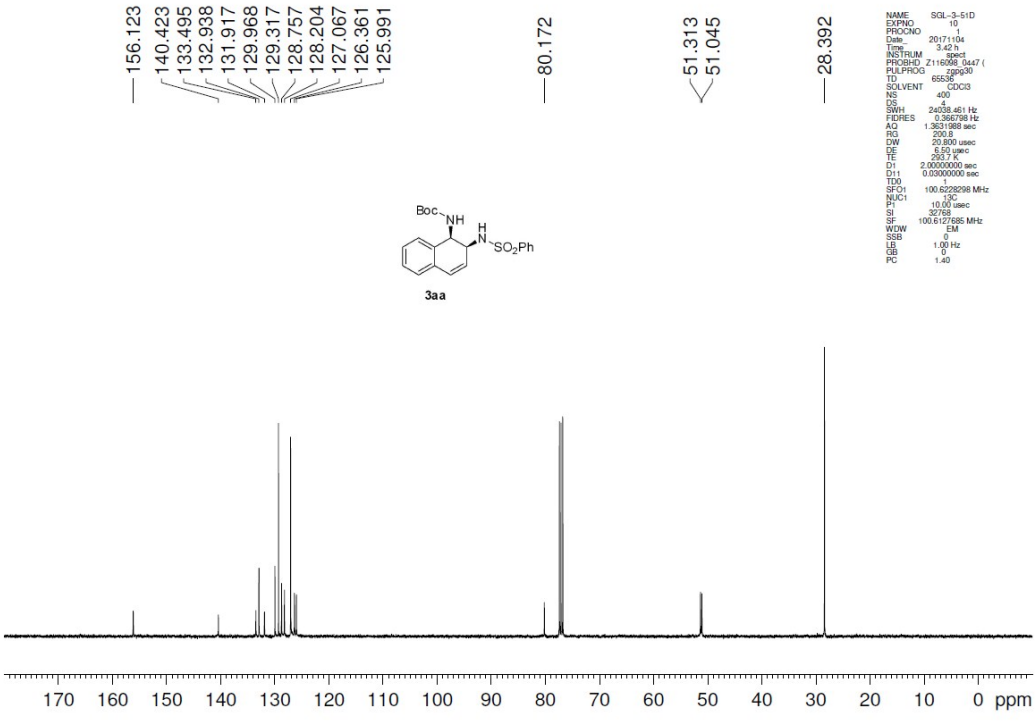
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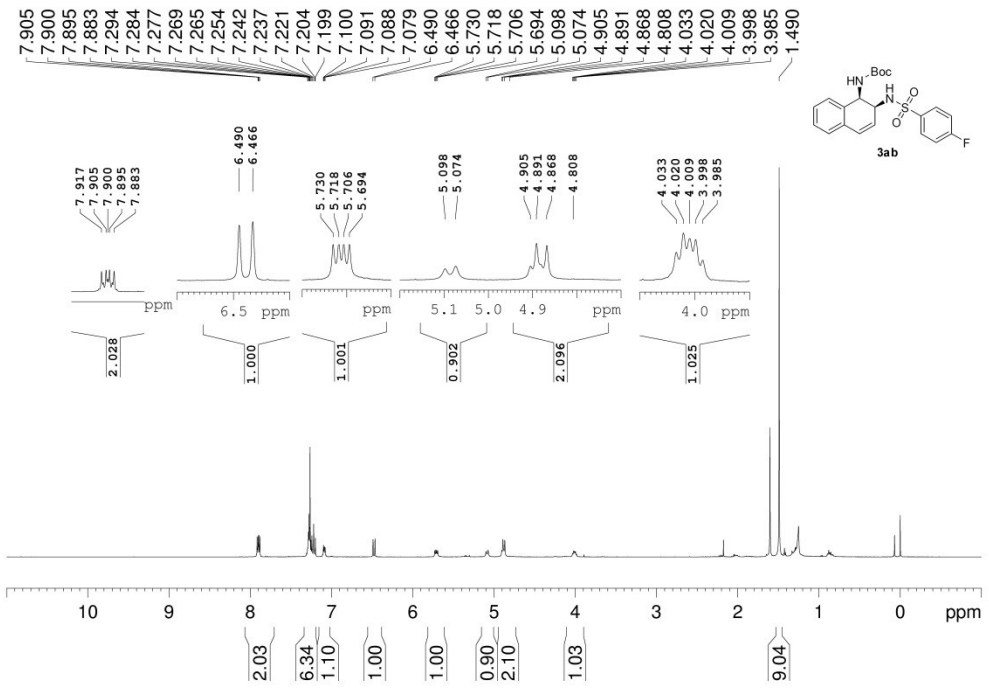
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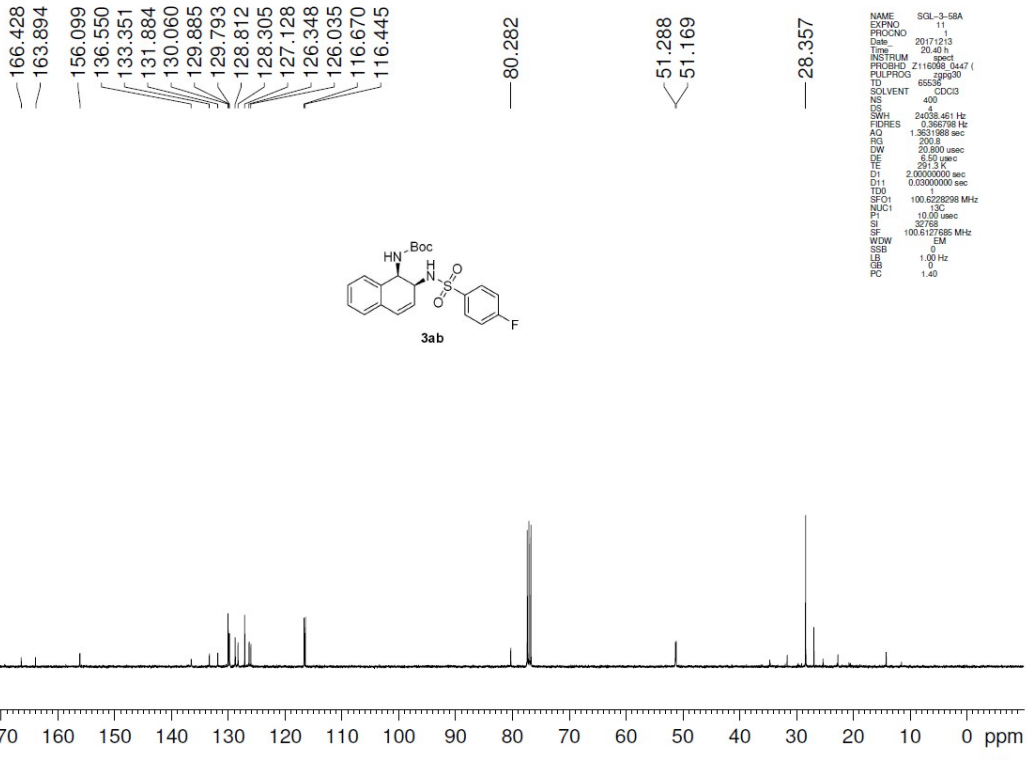
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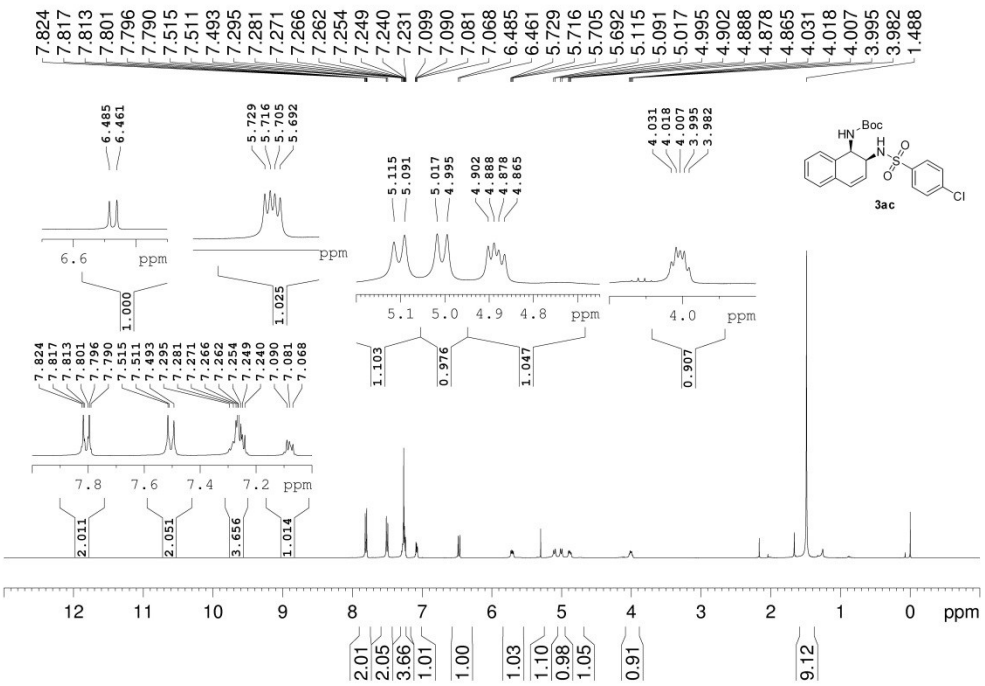
DW 20.800 usec
DE 6.50 usec
TE 291.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
H
N
S
O
O
F
3ab
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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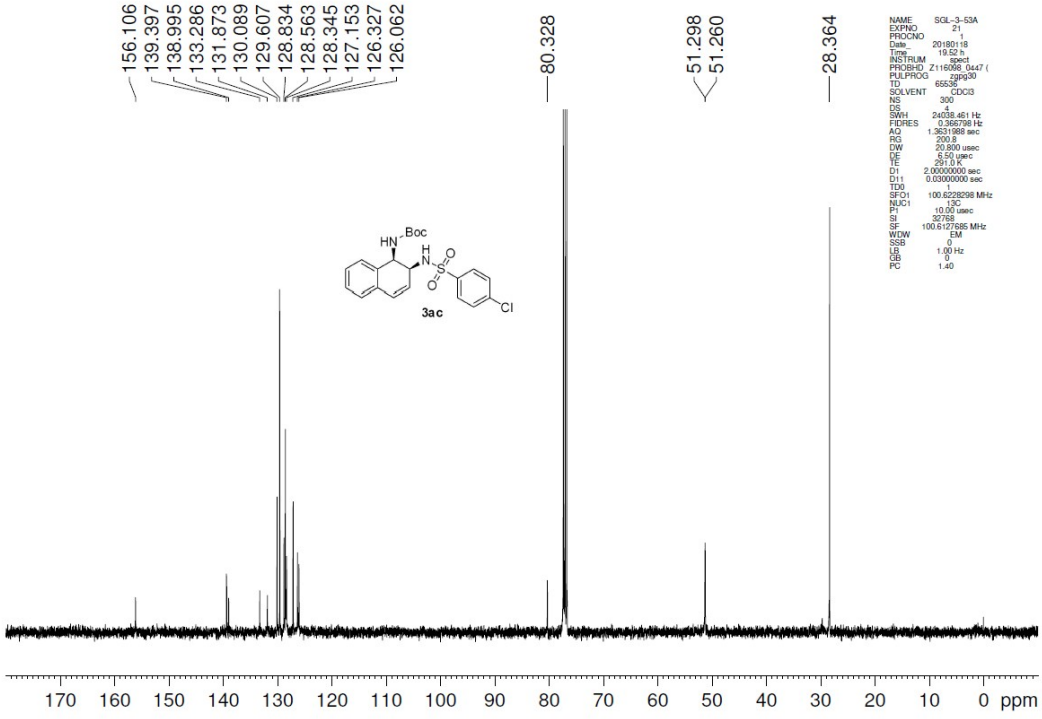
7.813
7.801
7.796
7.790
7.515
7.511
7.493
7.295
7.281
7.271
7.266
7.262
7.254
7.249
7.240
7.231
7.099
7.090
7.081
7.068
6.485
6.461
5.729
5.716
5.705
5.692
5.115
5.091
5.017
4.995
4.902
4.888
4.878
4.865
4.031
4.018
4.007
3.995
3.982
1.488
Boc
HN
H
N
S
O
O
Cl
3ac
6.6 ppm
1.000
1.025
5.1 5.0 4.9 4.8 ppm
4.0 ppm
1.103
0.976
1.047
0.907
7.8 7.6 7.4 7.2 ppm
2.011
2.051
3.656
1.014
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.01
2.05
3.66
1.01
1.00
1.03
1.10
0.98
1.05
0.91
9.12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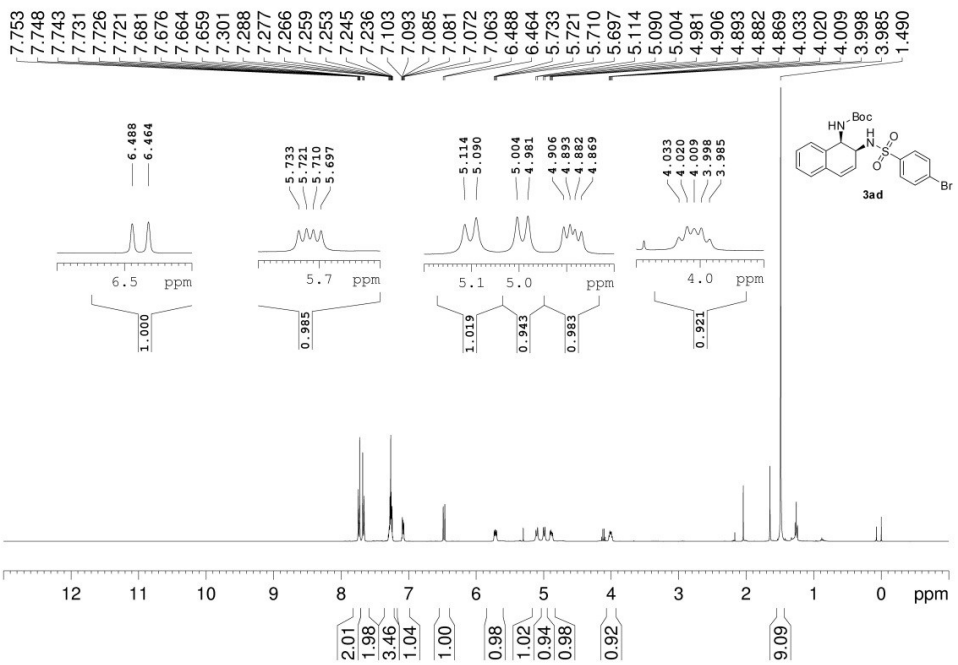
139.397
138.995
133.286
131.873
130.089
129.607
128.834
128.563
128.345
127.153
126.327
126.062
80.328
51.298
51.260
28.364
NAME SGL-3-53A
EXPNO 21
PROCNO 1
Date_ 20180118
Time 19.52 h
INSTRUM spect
PROBHD Z116098_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 291.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
H
N
S
O
O
Cl
3ac
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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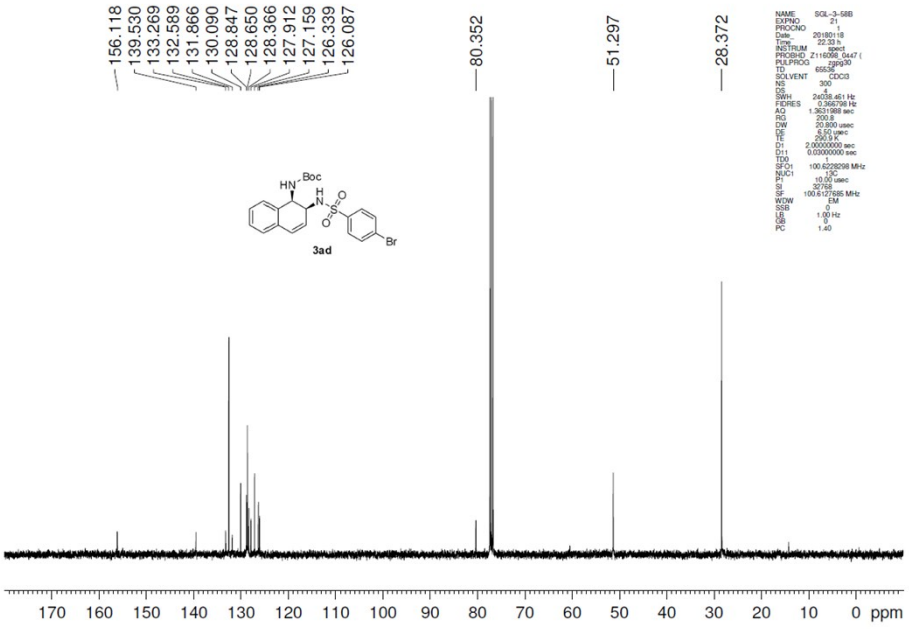
7.748
7.743
7.731
7.726
7.721
7.681
7.676
7.664
7.659
7.301
7.288
7.277
7.266
7.259
7.253
7.245
7.236
7.103
7.093
7.085
7.081
7.072
7.063
6.488
6.464
5.733
5.721
5.710
5.697
5.114
5.090
5.004
4.981
4.906
4.893
4.882
4.869
4.033
4.020
4.009
3.998
3.985
1.490
HN
Boc
H
N
S
O
O
Br
3ad
6.488
6.464
5.733
5.721
5.710
5.697
5.114
5.090
5.004
4.981
4.906
4.893
4.882
4.869
4.033
4.020
4.009
3.998
3.985
6.5 ppm
5.7 ppm
5.1 5.0 ppm
4.0 ppm
1.000
0.985
1.019
0.943
0.983
0.921
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.01
1.98
3.46
1.04
1.00
0.98
1.02
0.94
0.98
0.92
9.09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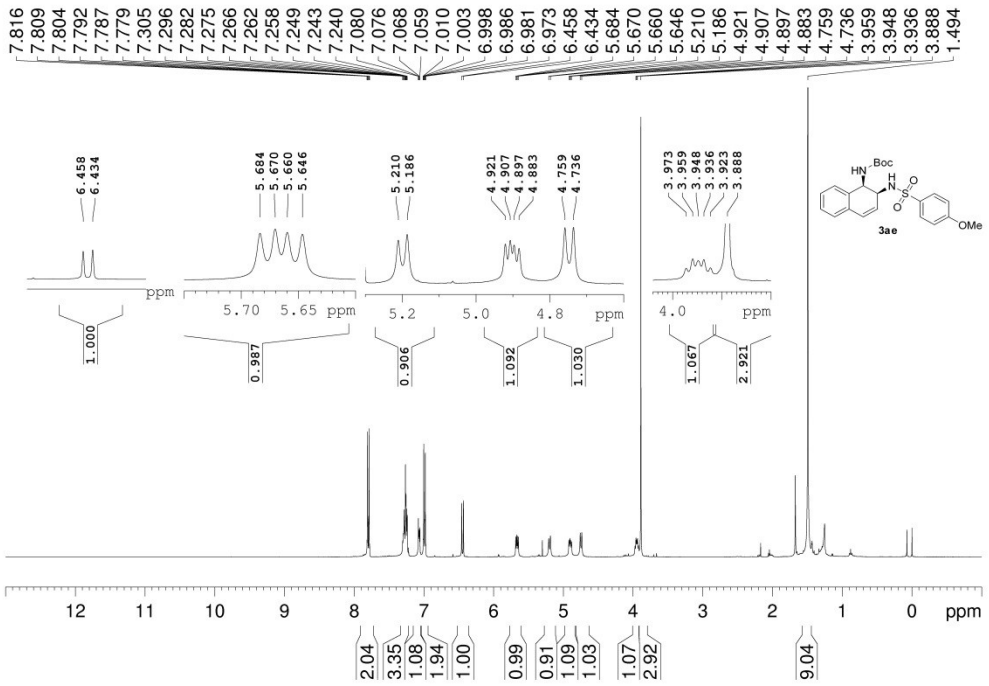
139.530
133.269
132.589
131.866
130.090
128.847
128.650
128.366
127.912
127.159
126.339
126.087
80.352
51.297
28.372
HN
Boc
H
N
S
O
O
Br
3ad
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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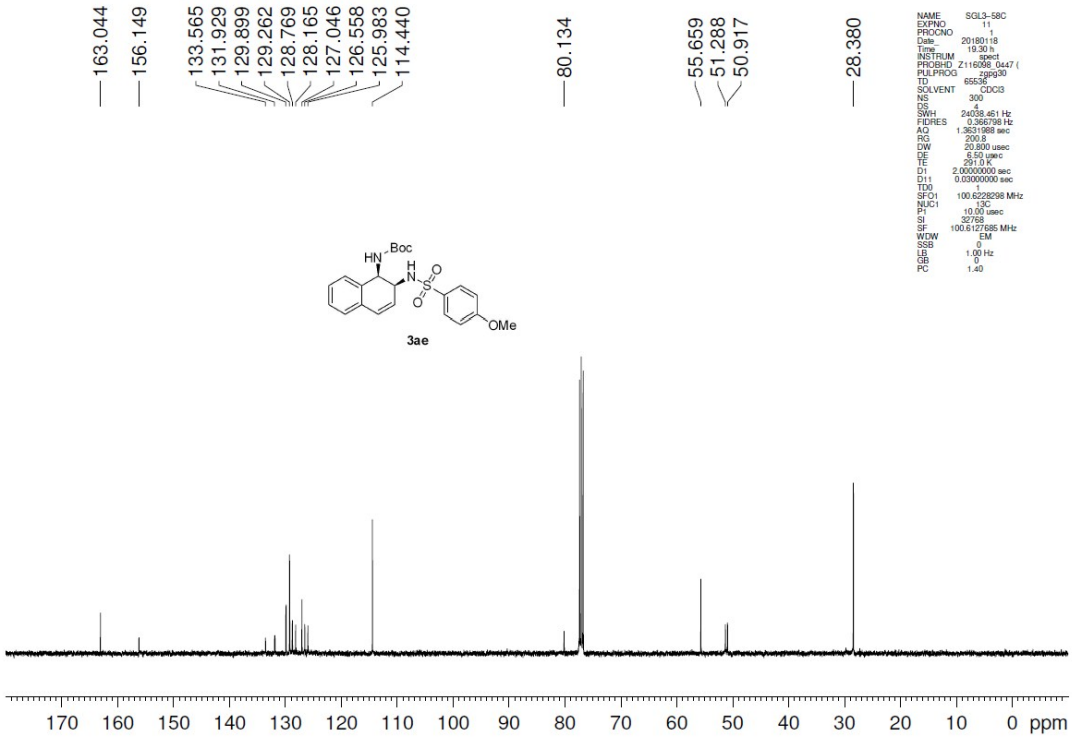
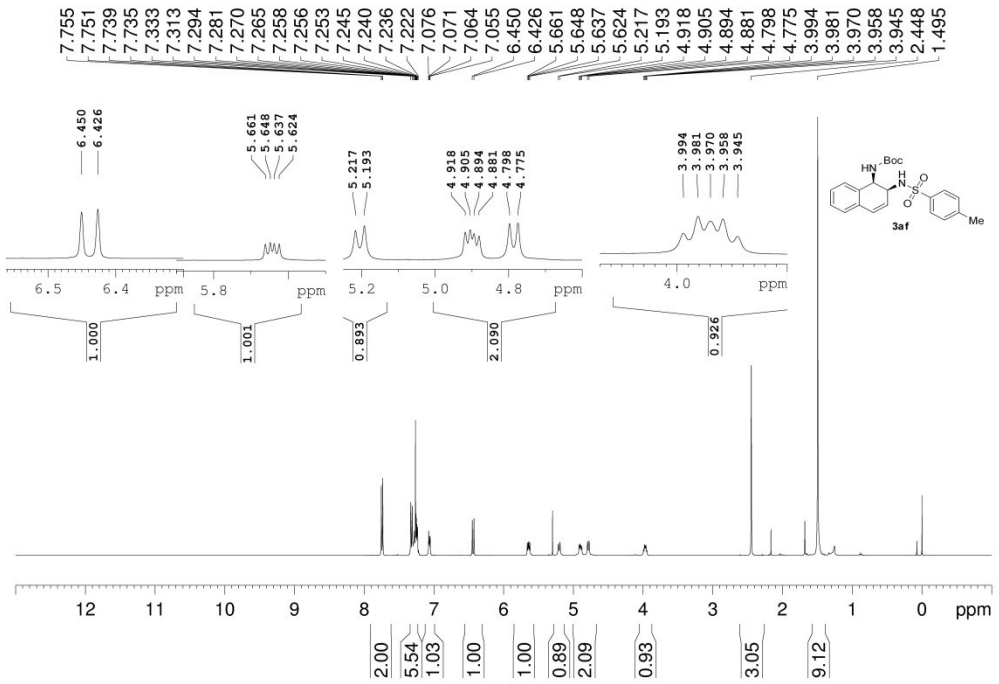
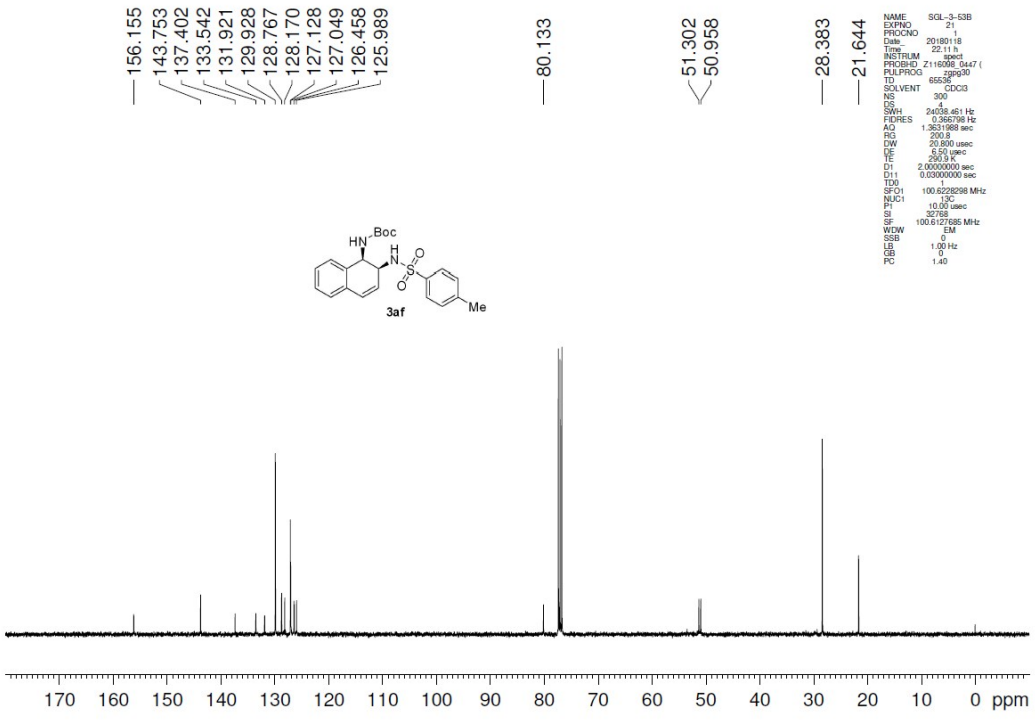
7.809
7.804
7.792
7.787
7.779
7.305
7.296
7.282
7.275
7.266
7.262
7.258
7.249
7.243
7.240
7.080
7.076
7.068
7.059
7.010
7.003
6.998
6.986
6.981
6.973
6.458
6.434
5.684
5.670
5.660
5.646
5.210
5.186
4.921
4.907
4.897
4.883
4.759
4.736
3.959
3.948
3.936
3.888
1.494
3ae
ppm
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm


163.044
156.149
133.565
131.929
129.899
129.262
128.769
128.165
127.046
126.558
125.983
114.440
80.134
55.659
51.288
50.917
28.380
3ae
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.755
7.751
7.739
7.735
7.333
7.313
7.294
7.281
7.270
7.265
7.258
7.256
7.253
7.245
7.240
7.236
7.222
7.076
7.071
7.064
7.055
6.450
6.426
5.661
5.648
5.637
5.624
5.217
5.193
4.918
4.905
4.894
4.881
4.798
4.775
3.994
3.981
3.970
3.958
3.945
3.958
3.945
2.448
1.495
Boc
HN
N
S
O
O
Me
3af
ppm
1.000
1.001
0.893
2.090
0.926
2.00
5.54
1.03
1.00
1.00
0.89
2.09
0.93
3.05
9.12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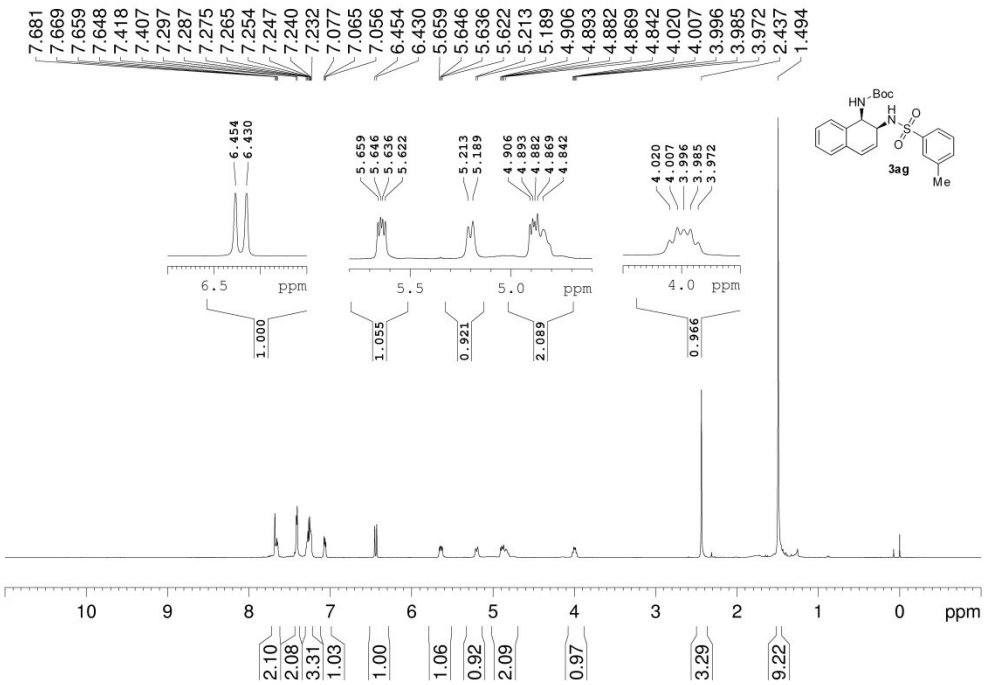
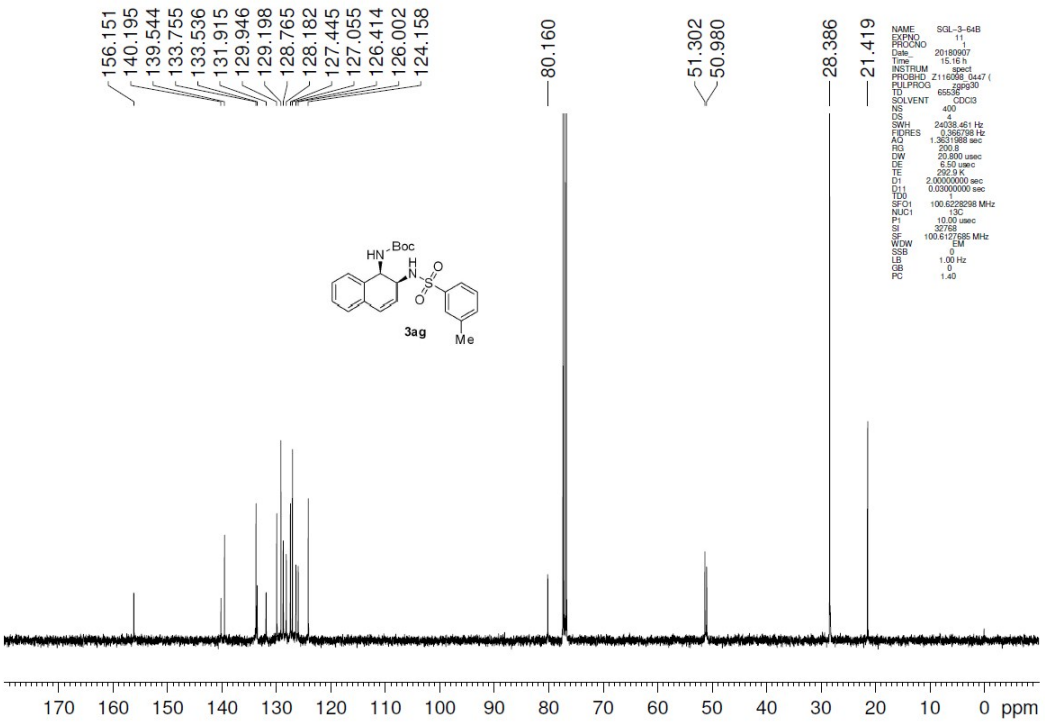
143.753
137.402
133.542
131.921
129.928
128.767
128.170
127.128
127.049
126.458
125.989
80.133
51.302
50.958
28.383
21.644
Boc
HN
N
S
O
O
Me
3af
ppm


7.681
7.669
7.659
7.648
7.418
7.407
7.297
7.287
7.275
7.265
7.254
7.247
7.240
7.232
7.077
7.065
7.056
6.454
6.430
5.659
5.646
5.636
5.622
5.213
5.189
4.906
4.893
4.882
4.869
4.842
4.020
4.007
3.996
3.985
3.972
2.437
1.494
Boc
HN
H
N
S
O
O
3ag
Me
6.5 ppm
5.5
5.0 ppm
4.0 ppm
1.000
1.055
0.921
2.089
0.966
10 9 8 7 6 5 4 3 2 1 0 ppm
2.10
2.08
3.31
1.03
1.00
1.06
0.92
2.09
0.97
3.29
9.22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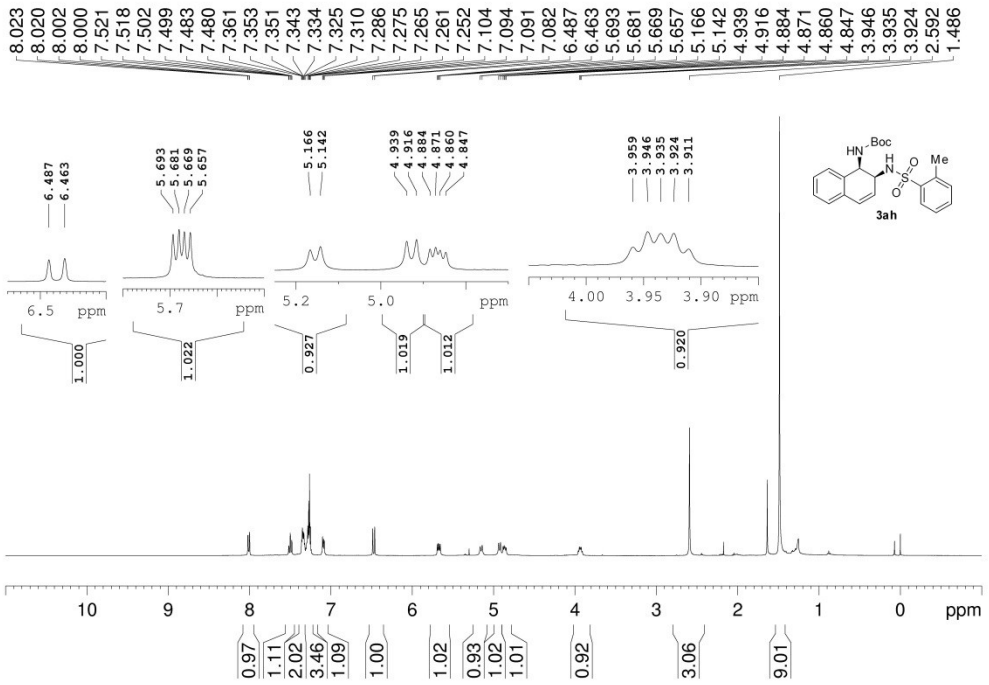
140.195
139.544
133.755
133.536
131.915
129.946
129.198
128.765
128.182
127.445
127.055
126.414
126.002
124.158
80.160
51.302
50.980
28.386
21.419
NAME SGL-3-64B
EXPNO 11
PROCNO 1
Date_ 20180907
Time 15.16 h
INSTRUM spect
PROBHD Z116068_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
H
N
S
O
O
3ag
Me
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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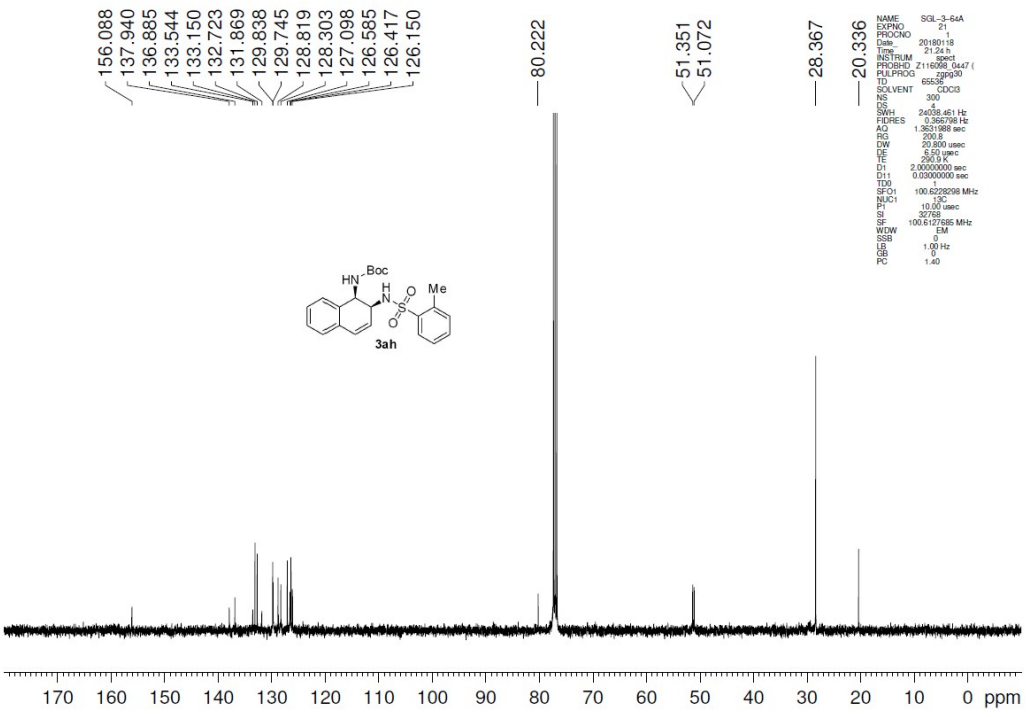
8.020
8.002
8.000
7.521
7.518
7.502
7.499
7.483
7.480
7.361
7.353
7.351
7.343
7.334
7.325
7.310
7.286
7.275
7.265
7.261
7.252
7.104
7.094
7.091
7.082
6.487
6.463
5.693
5.681
5.669
5.657
5.166
5.142
4.939
4.916
4.884
4.871
4.860
4.847
3.959
3.946
3.935
3.924
3.911
3.946
3.935
3.924
3.911
4.939
4.916
4.884
4.871
4.860
4.847
5.166
5.142
5.693
5.681
5.669
5.657
6.487
6.463
Boc
HN
H
N
S
O
O
Me
3ah
6.5 ppm
5.7 ppm
5.2
5.0 ppm
4.00 3.95 3.90 ppm
1.000
1.022
0.927
1.019
1.012
0.920
10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
1.11
2.02
3.46
1.09
1.00
1.02
0.93
1.02
1.01
0.92
3.06
9.01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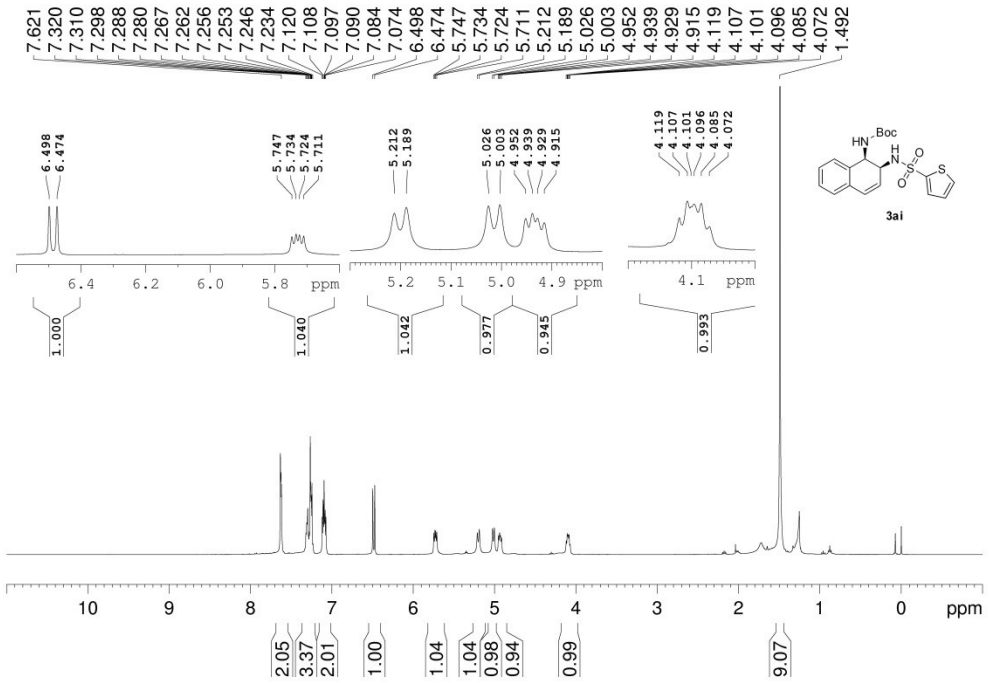
137.940
136.885
133.544
133.150
132.723
131.869
129.838
129.745
128.819
128.303
127.098
126.585
126.417
126.150
80.222
51.351
51.072
28.367
20.336
NAME SGL-3-64A
EXPNO 21
PROCNO 1
Date_ 20180118
Time_ 21.24 h
INSTRUM spect
PROBHD Z116098 0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
H
N
S
O
O
Me
3ah
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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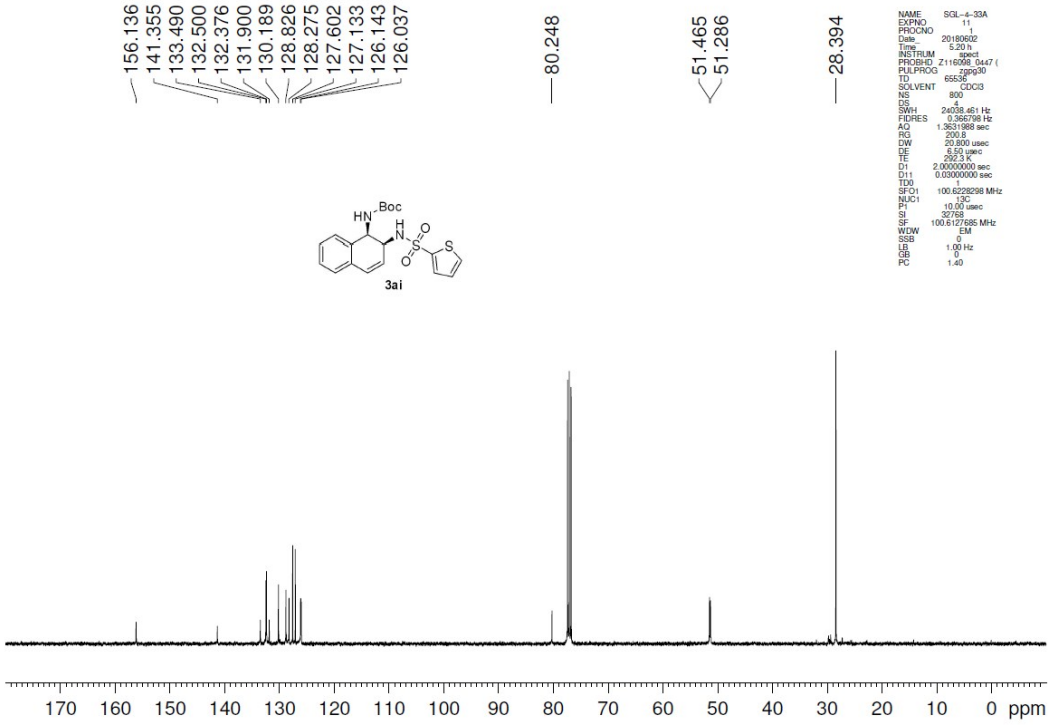
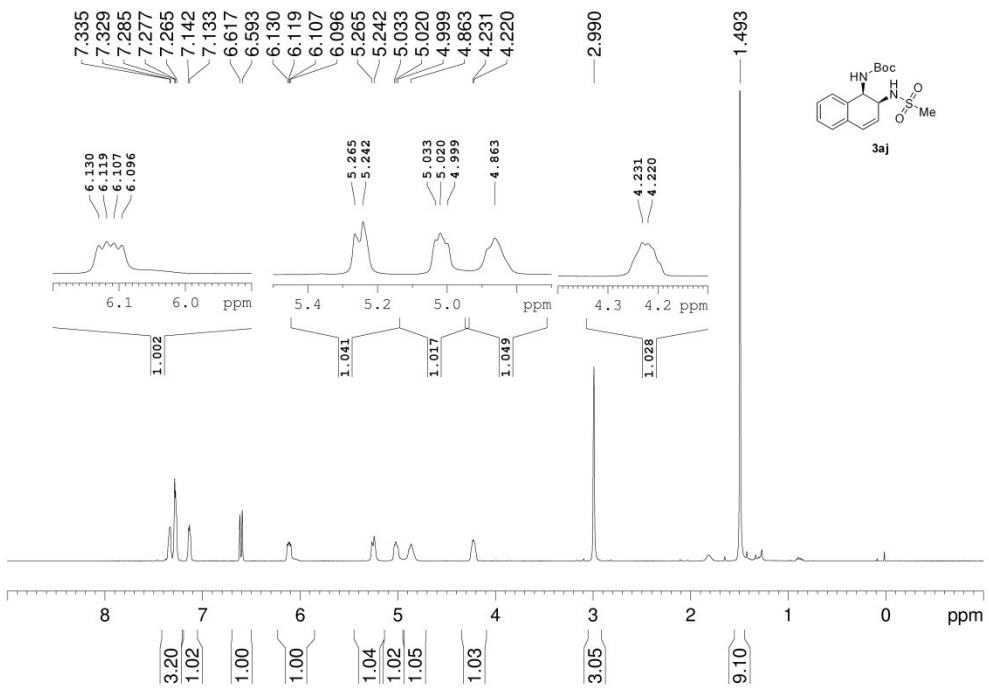
7.310
7.298
7.288
7.280
7.267
7.262
7.256
7.253
7.246
7.234
7.120
7.108
7.097
7.090
7.084
7.074
6.498
6.474
5.747
5.734
5.724
5.711
5.212
5.189
5.026
5.003
4.952
4.939
4.929
4.915
4.119
4.107
4.101
4.096
4.085
4.072
1.492
HN
Boc
H
N
O
O
S
S
3ai
6.4
6.2
6.0
5.8
ppm
5.2
5.1
5.0
4.9 ppm
4.1
ppm
1.000
1.040
1.042
0.977
0.945
0.993
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
3.37
2.01
1.00
1.04
1.04
0.98
0.94
0.99
9.07


156.136
141.355
133.490
132.500
132.376
131.900
130.189
128.826
128.275
127.602
127.133
126.143
126.037
80.248
51.465
51.286
28.394
NAME SGL-4-33A
EXPNO 11
PROCNO 1
Date_ 20180602
Time 5.20 h
INSTRUM spect
PROBHD Z116008_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
Boc
H
N
O
O
S
S
3ai
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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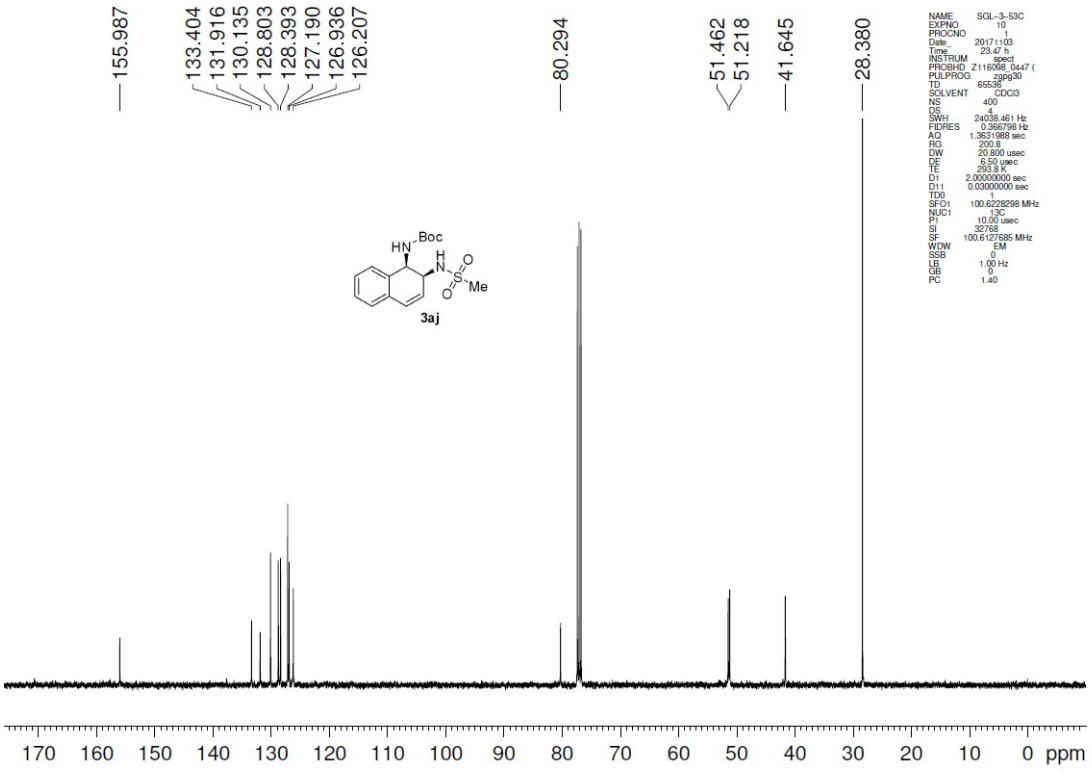
7.277
7.265
7.142
7.133
6.617
6.593
6.130
6.119
6.107
6.096
5.265
5.242
5.033
5.020
4.999
4.863
4.231
4.220
2.990
1.493
3aj
6.1
6.0 ppm
5.4
5.2
5.0 ppm
4.3
4.2 ppm
1.002
1.041
1.017
1.049
1.028
8
7
6
5
4
3
2
1
0 ppm
3.20
1.02
1.00
1.00
1.04
1.02
1.05
1.03
3.05
9.10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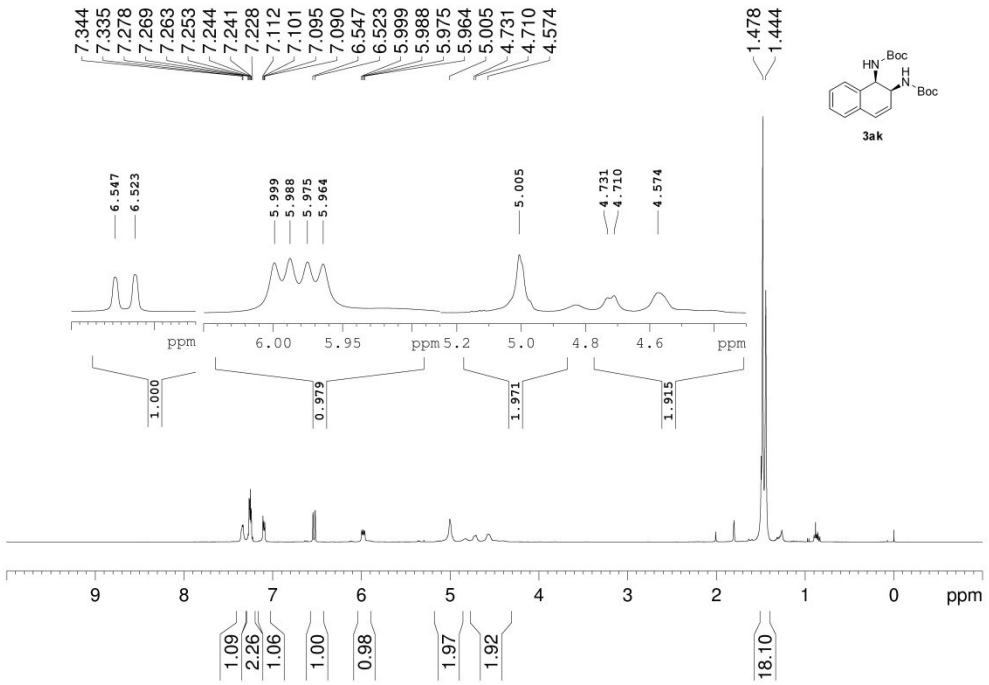
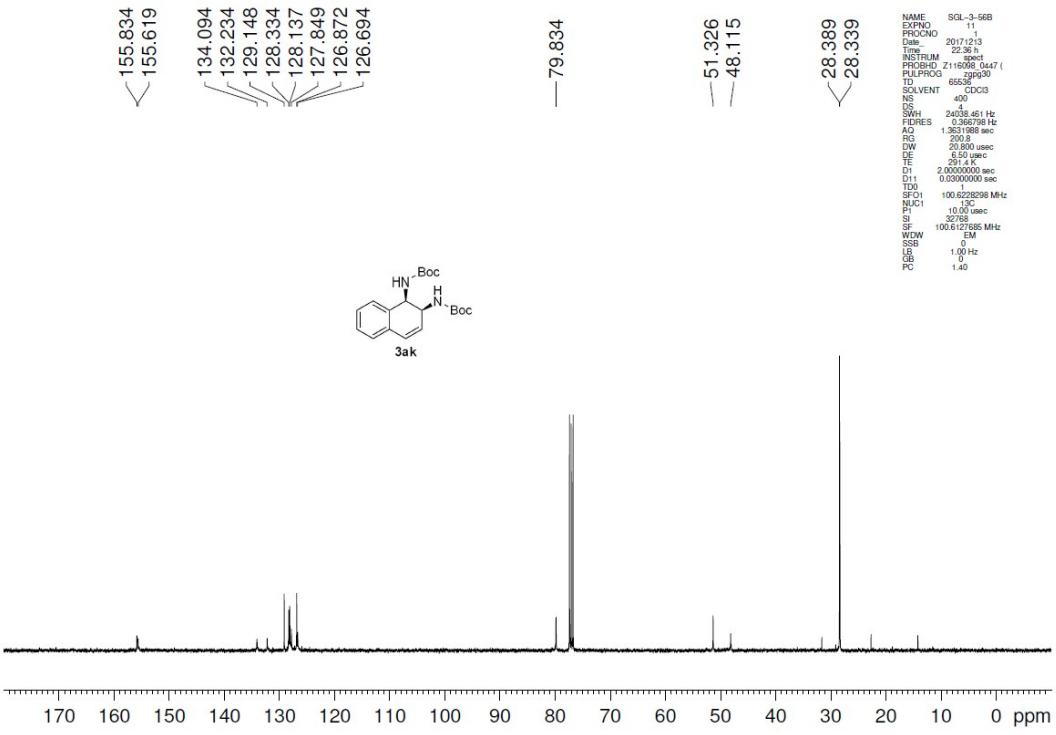
133.404
131.916
130.135
128.803
128.393
127.190
126.936
126.207
80.294
51.462
51.218
41.645
28.380
3aj
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


7.344
7.335
7.278
7.269
7.263
7.253
7.244
7.241
7.228
7.112
7.101
7.095
7.090
6.547
6.523
5.999
5.988
5.975
5.964
5.005
4.731
4.710
4.574
1.478
1.444
Boc
HN
N
Boc
3ak
ppm
6.00
5.95
5.2
5.0
4.8
4.6
1.000
0.979
1.971
1.915
9
8
7
6
5
4
3
2
1
0
ppm
1.09
2.26
1.06
1.00
0.98
1.97
1.92
18.10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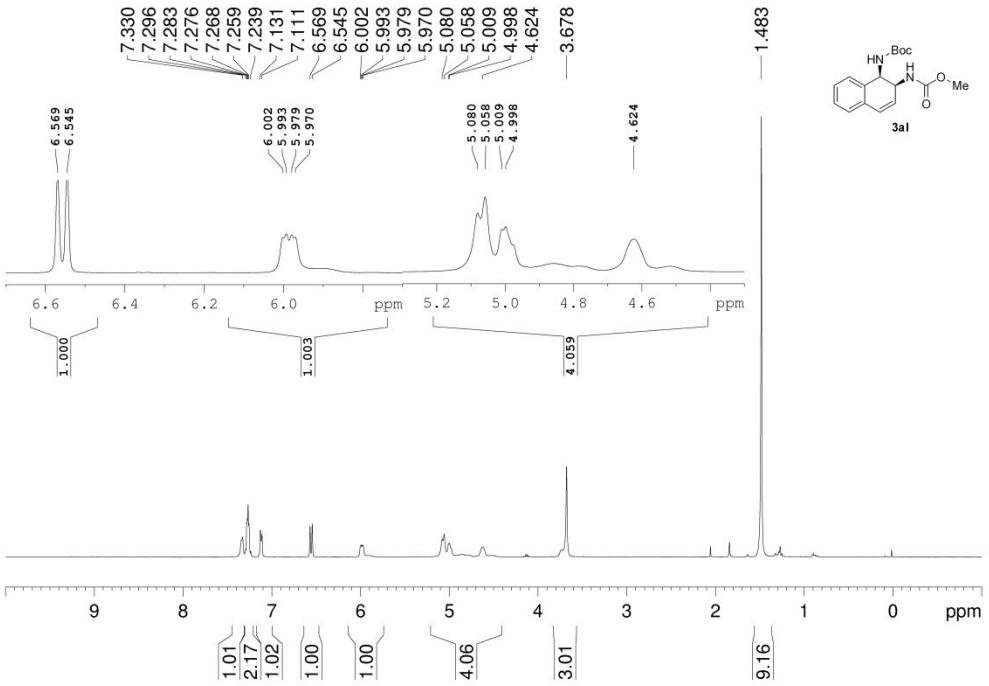
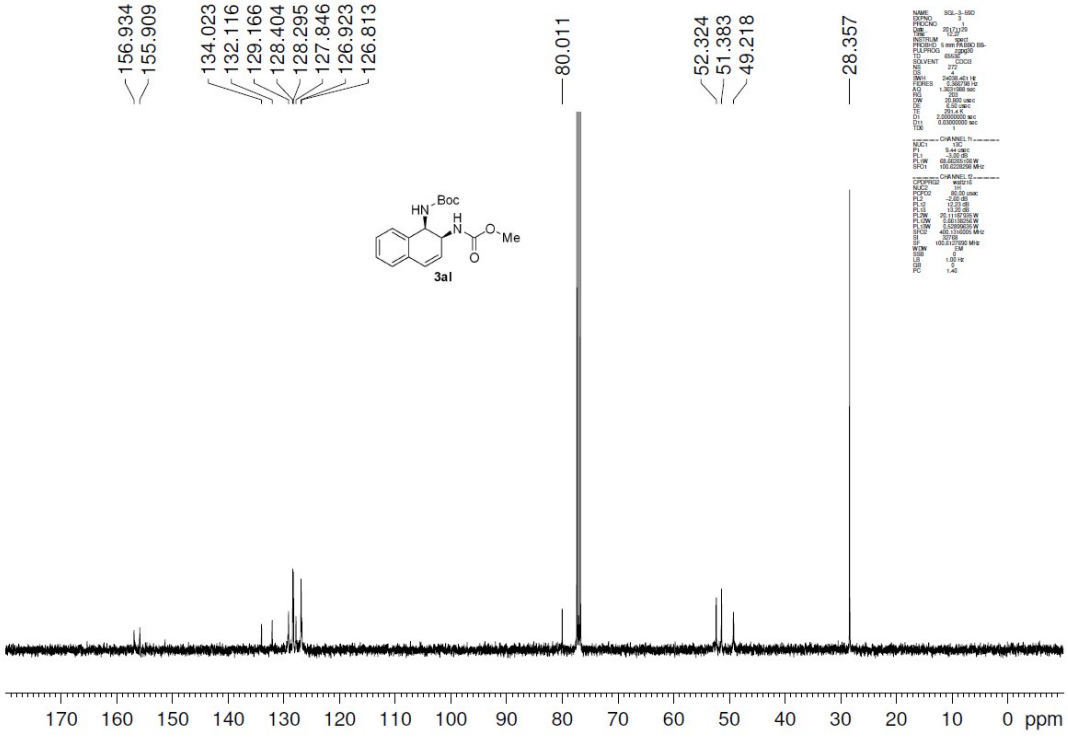
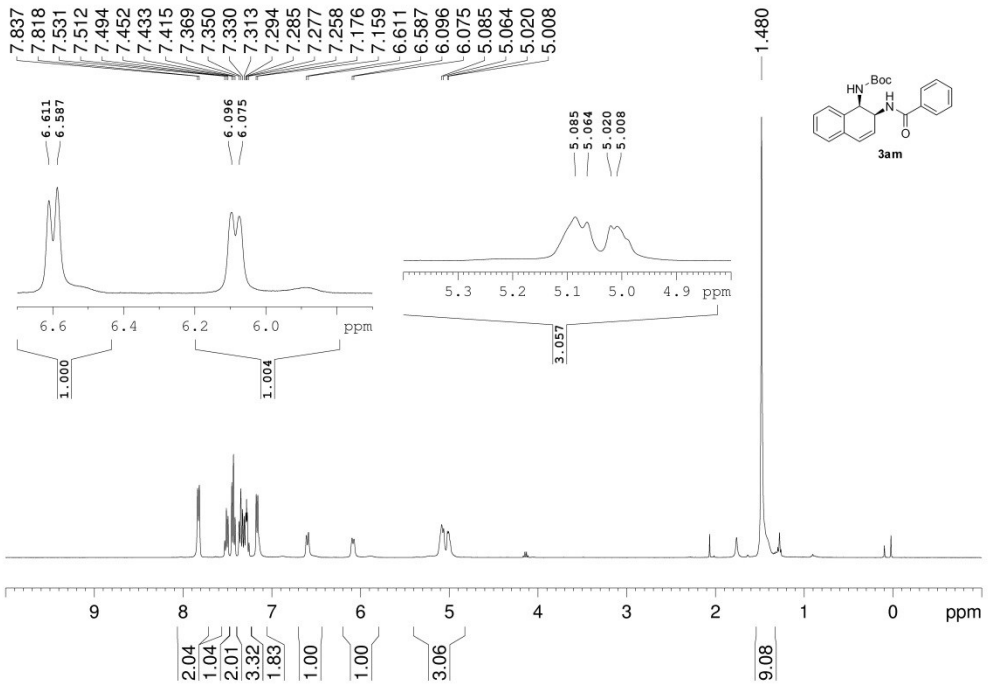
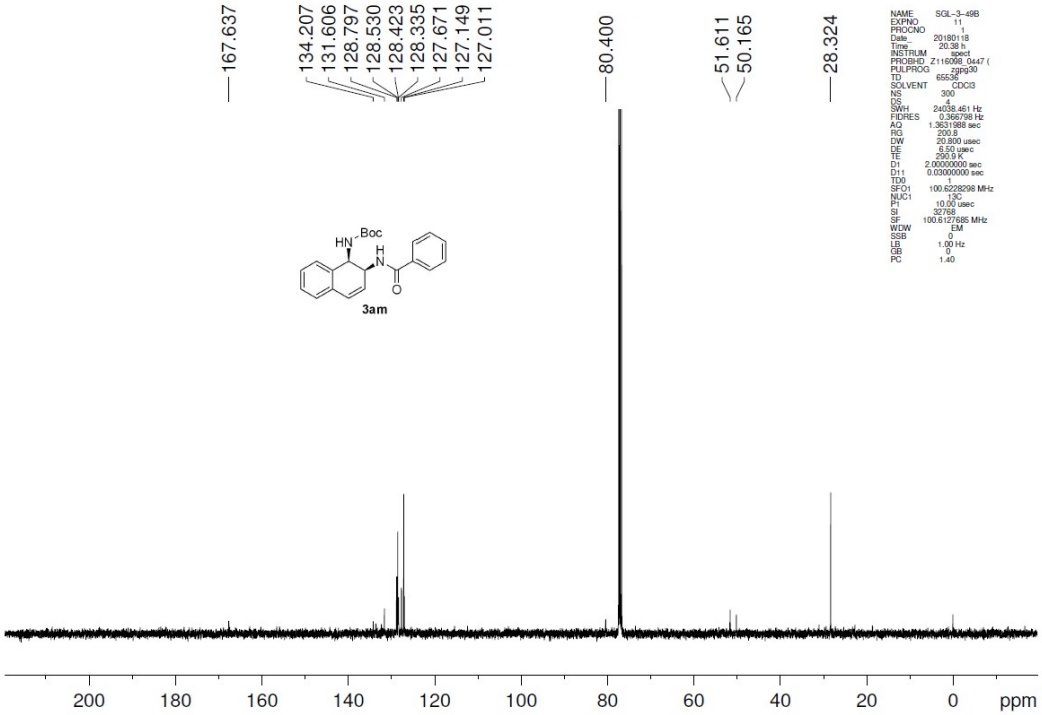
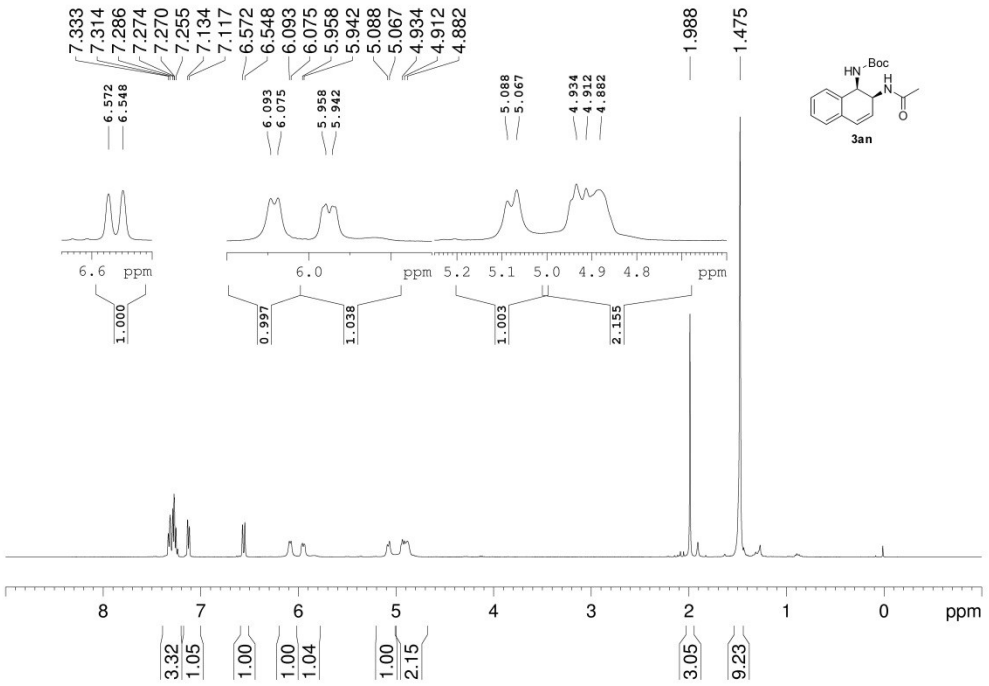
134.094
132.234
129.148
128.334
128.137
127.849
126.872
126.694
79.834
51.326
48.115
28.389
28.339
NAME SGL-3-56B
EXPNO 11
PROCNO 1
Date_ 20171213
Time 22.36 h
INSTRUM spect
PROBHD Z116098_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
N
Boc
3ak
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

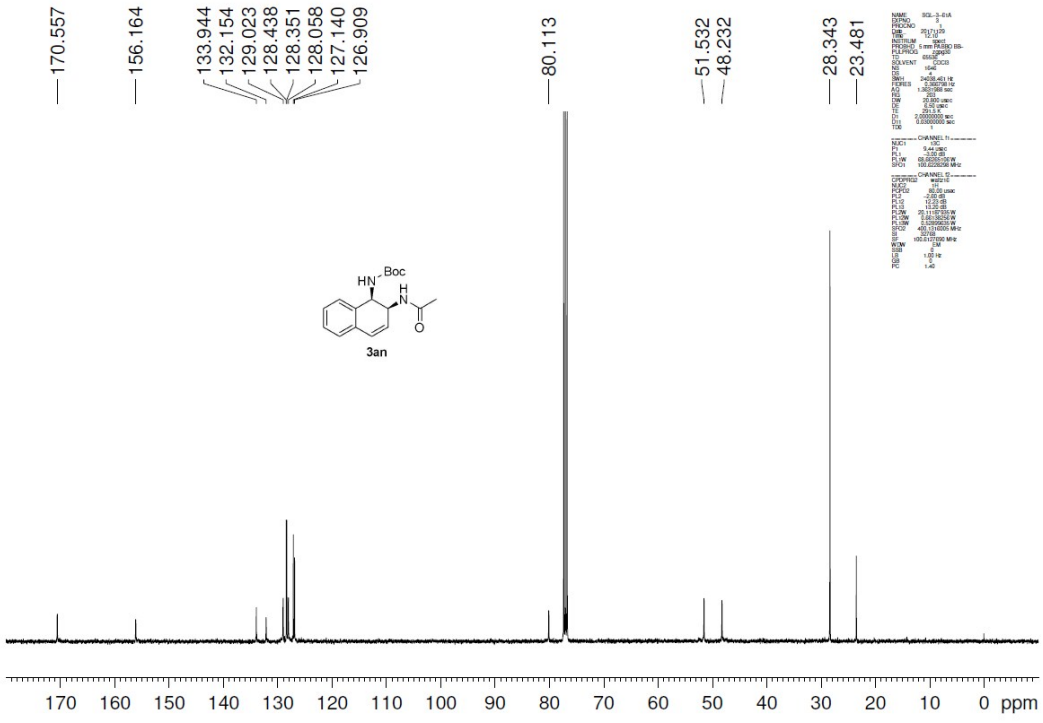

7.330
7.296
7.283
7.276
7.268
7.259
7.239
7.131
7.111
6.569
6.545
6.002
5.993
5.979
5.970
5.080
5.058
5.009
4.998
4.624
3.678
1.483
Boc
HN
H
N
O
Me
3al
6.6
6.4
6.2
6.0
ppm
5.2
5.0
4.8
4.6
ppm
1.000
1.003
4.059
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.17
1.02
1.00
1.00
4.06
3.01
9.16

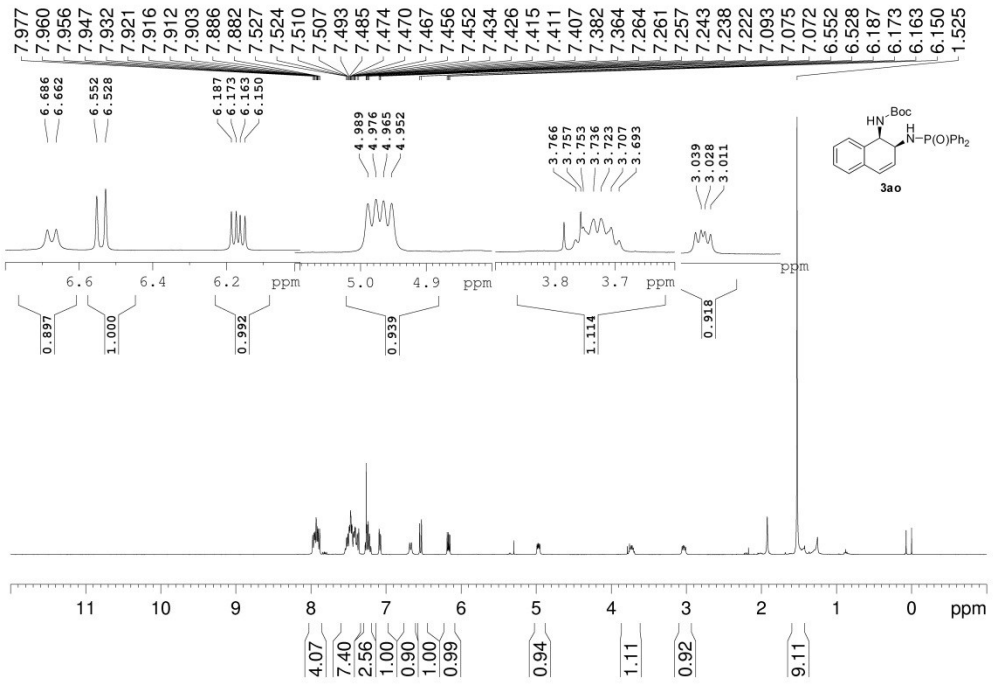

156.934
155.909
134.023
132.116
129.166
128.404
128.295
127.846
126.923
126.813
80.011
52.324
51.383
49.218
28.357
Boc
HN
H
N
O
Me
3al
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

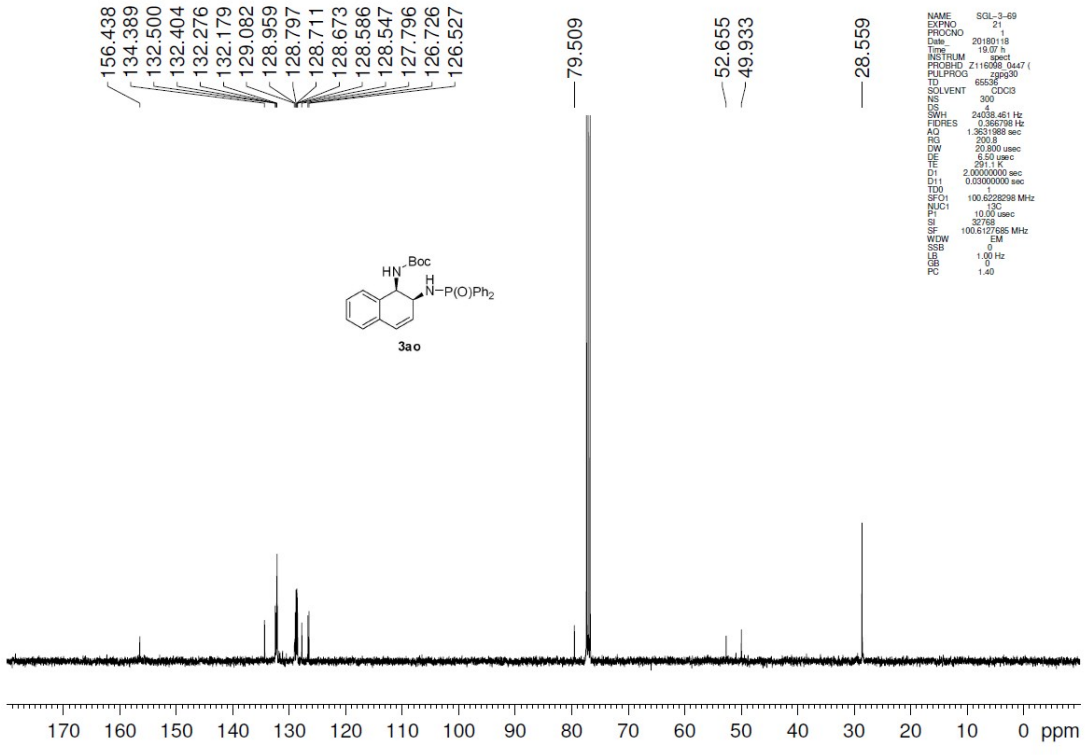

7.837
7.818
7.531
7.512
7.494
7.452
7.433
7.415
7.369
7.350
7.330
7.313
7.294
7.285
7.277
7.258
7.176
7.159
6.611
6.587
6.096
6.075
6.075
5.085
5.064
5.020
5.008
1.480
3am
6.6
6.4
6.2
6.0
ppm
5.3
5.2
5.1
5.0
4.9
ppm
1.000
1.004
3.057
9
8
7
6
5
4
3
2
1
0
ppm
2.04
1.04
2.01
3.32
1.83
1.00
1.00
3.06
9.08

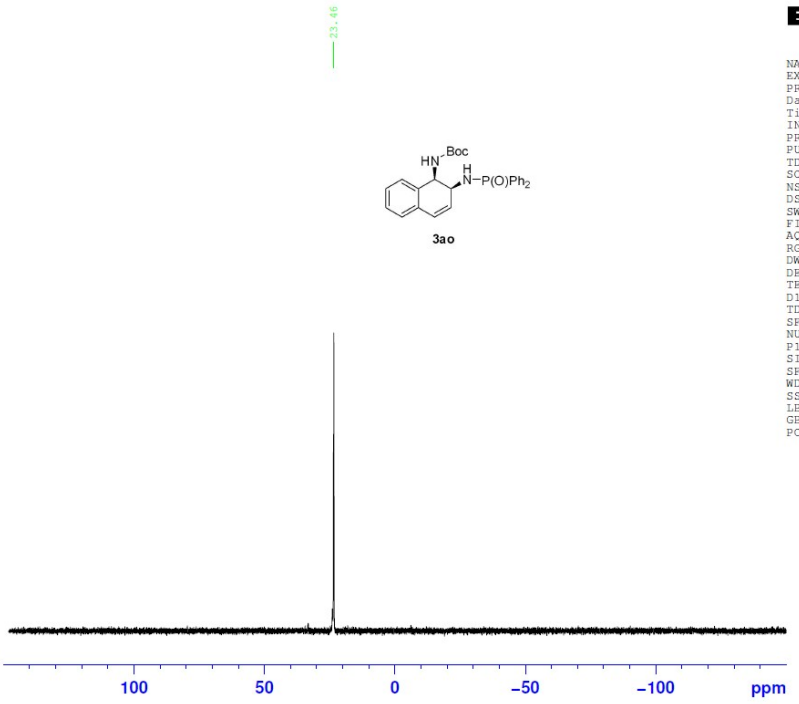

167.637
134.207
131.606
128.797
128.530
128.423
128.335
127.671
127.149
127.011
80.400
51.611
50.165
28.324
3am
200
180
160
140
120
100
80
60
40
20
0
ppm


7.333
7.314
7.286
7.274
7.270
7.255
7.134
7.117
6.572
6.548
6.093
6.075
5.958
5.942
5.088
5.067
4.934
4.912
4.882
1.988
1.475
HN
Boc
H
N
O
3an
6.6 ppm
6.0 ppm
5.2 5.1 5.0 4.9 4.8 ppm
1.000
0.997
1.038
1.003
2.155
8 7 6 5 4 3 2 1 0 ppm
3.32
1.05
1.00
1.00
1.04
1.00
2.15
3.05
9.23


170.557
156.164
133.944
132.154
129.023
128.438
128.351
128.058
127.140
126.909
80.113
51.532
48.232
28.343
23.481
HN
Boc
H
N
O
3an
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

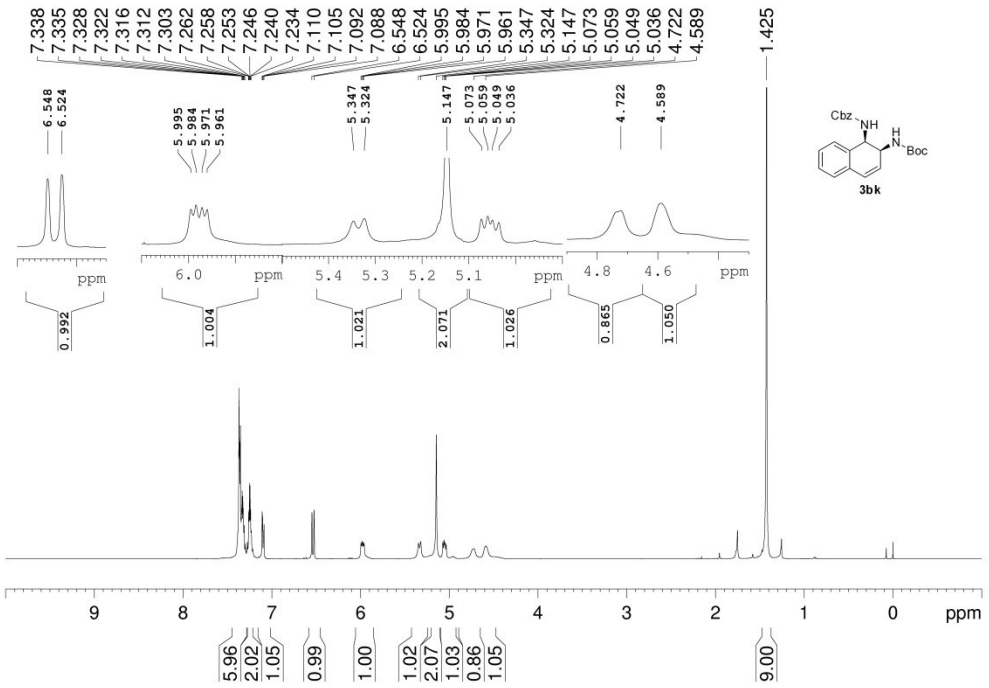

7.977
7.960
7.956
7.947
7.932
7.921
7.916
7.912
7.903
7.886
7.882
7.527
7.524
7.510
7.507
7.493
7.485
7.474
7.470
7.467
7.456
7.452
7.434
7.426
7.415
7.411
7.407
7.382
7.364
7.264
7.261
7.257
7.243
7.238
7.222
7.093
7.075
7.072
6.552
6.528
6.187
6.173
6.163
6.150
1.525
6.686
6.662
6.552
6.528
6.187
6.173
6.163
6.150
4.989
4.976
4.965
4.952
3.766
3.757
3.753
3.736
3.723
3.707
3.693
3.039
3.028
3.011
HN
Boc
N–P(O)Ph2
3ao
ppm
6.6
6.4
6.2
5.0
4.9
3.8
3.7
0.897
1.000
0.992
0.939
1.114
0.918
11
10
9
8
7
6
5
4
3
2
1
0
4.07
7.40
2.56
1.00
0.90
1.00
0.99
0.94
1.11
0.92
9.11

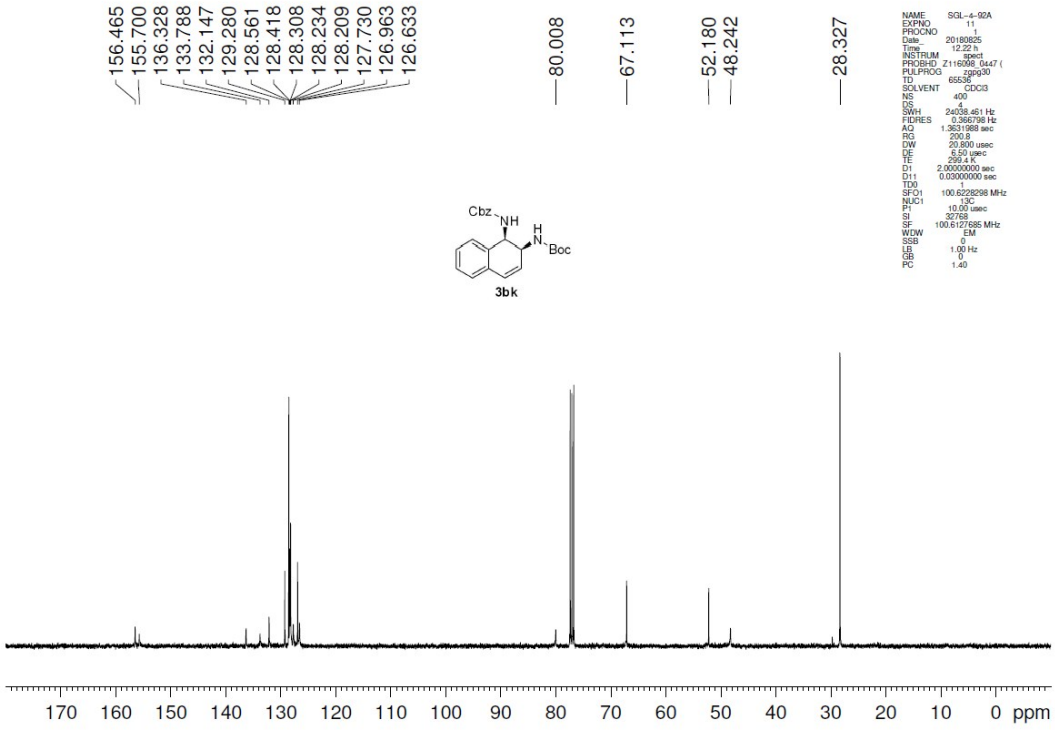

156.438
134.389
132.500
132.404
132.276
132.179
129.082
128.959
128.797
128.711
128.673
128.586
128.547
127.796
126.726
126.527
79.509
52.655
49.933
28.559
NAME SGL-3-69
EXPNO 21
PROCNO 1
Date_ 20180118
Time 19.07 h
INSTRUM spect
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 291.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
Boc
N–P(O)Ph2
3ao
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

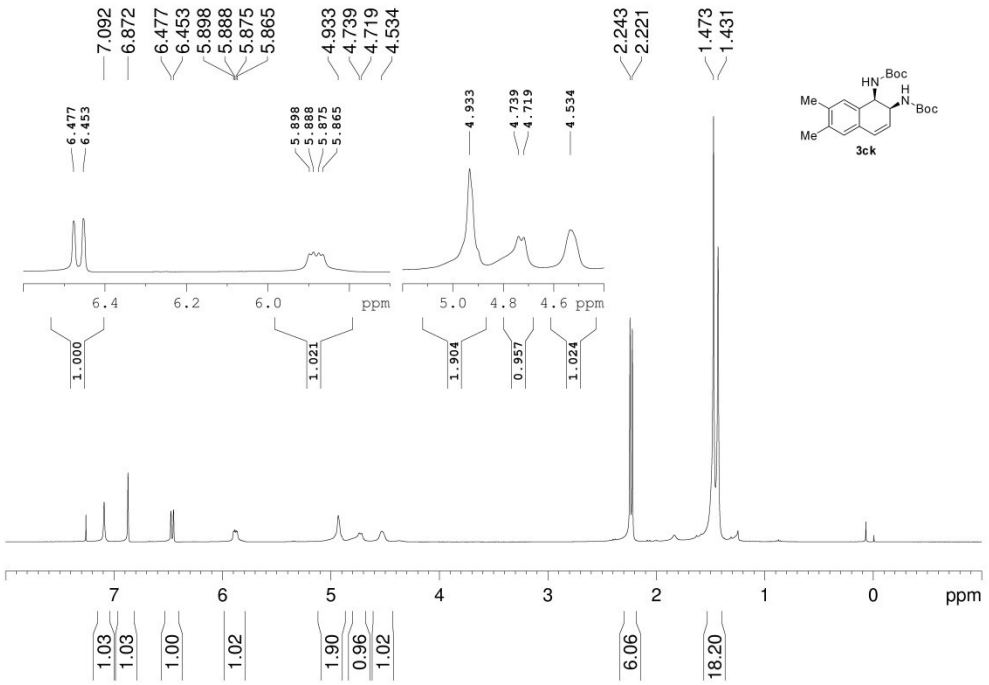


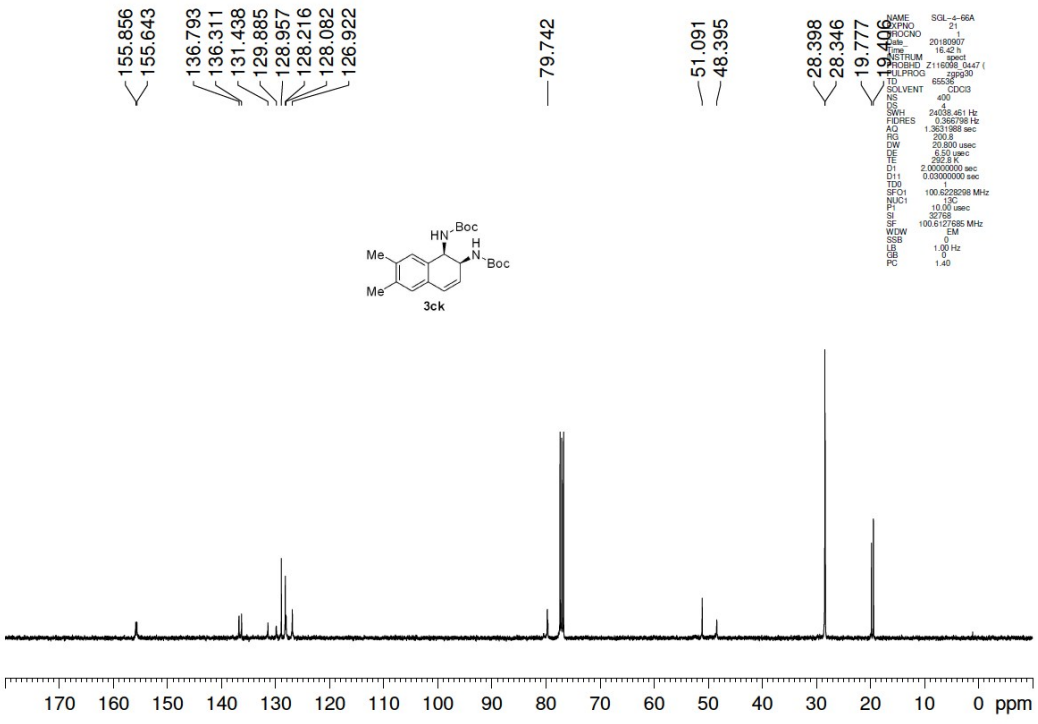
BRUKER

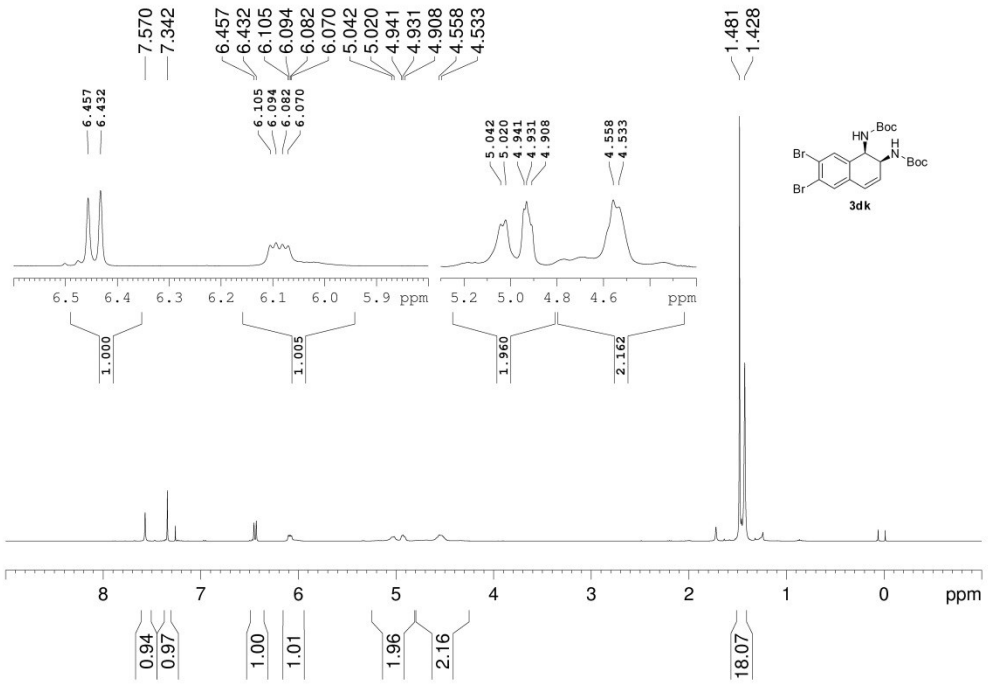
NAME	SGL-3-69
EXPNO	12
PROCNO	1
Date_	20171214
Time	6.45 h
INSTRUM	spect
PROBHD	Z116098_0447 (
PULPROG	zg30
TD	65536
SOLVENT	CDC13
NS	32
DS	4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5112308 sec
RG	200.8
DW	7.800 usec
DE	6.50 usec
TE	290.4 K
D1	2.00000000 sec
TD0	1
SFO1	161.9674942 MHz
NUC1	31P
P1	8.00 usec
SI	32768
SF	161.9755930 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

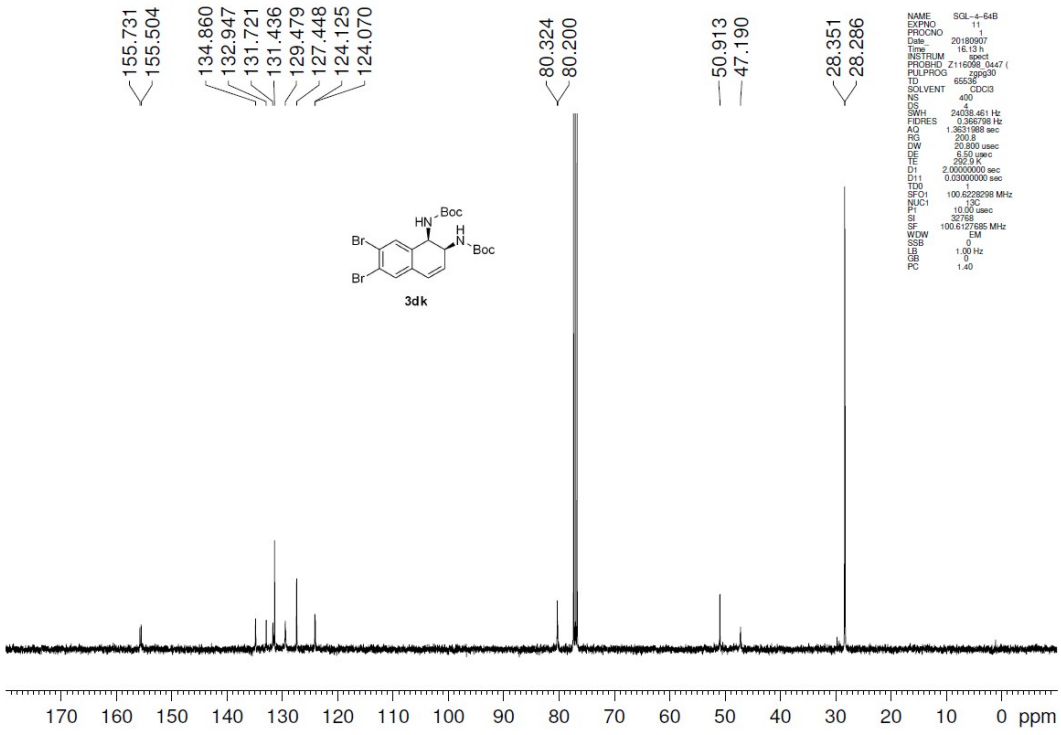

7.338
7.335
7.328
7.322
7.316
7.312
7.303
7.262
7.258
7.253
7.246
7.240
7.234
7.110
7.105
7.092
7.088
6.548
6.524
5.995
5.984
5.971
5.961
5.347
5.324
5.147
5.073
5.059
5.049
5.036
4.722
4.589
1.425
Cbz
NH
H
N
Boc
3bk
ppm
0.992
1.004
1.021
2.071
1.026
0.865
1.050
9
8
7
6
5
4
3
2
1
0
ppm
5.96
2.02
1.05
0.99
1.00
1.02
2.07
1.03
0.86
1.05
9.00

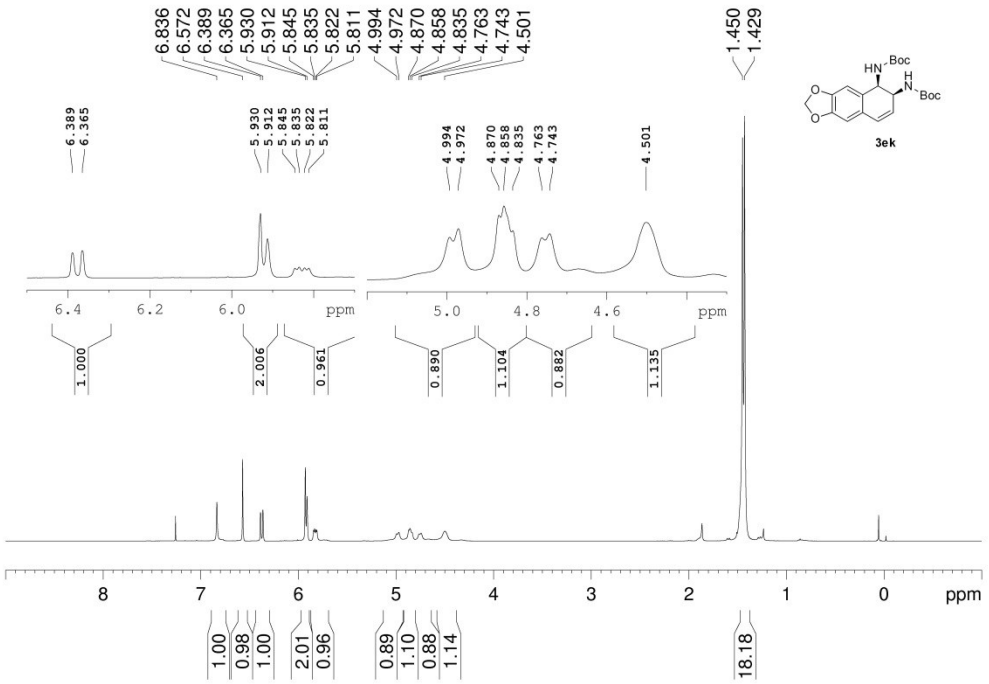

156.465
155.700
136.328
133.788
132.147
129.280
128.561
128.418
128.308
128.234
128.209
127.730
126.963
126.633
80.008
67.113
52.180
48.242
28.327
NAME SGL-4-92A
EXPNO 11
PROCNO 1
Date 20180825
Time 12.22 h
INSTRUM spect
PROBHD Z116098_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
NH
H
N
Boc
3bk
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

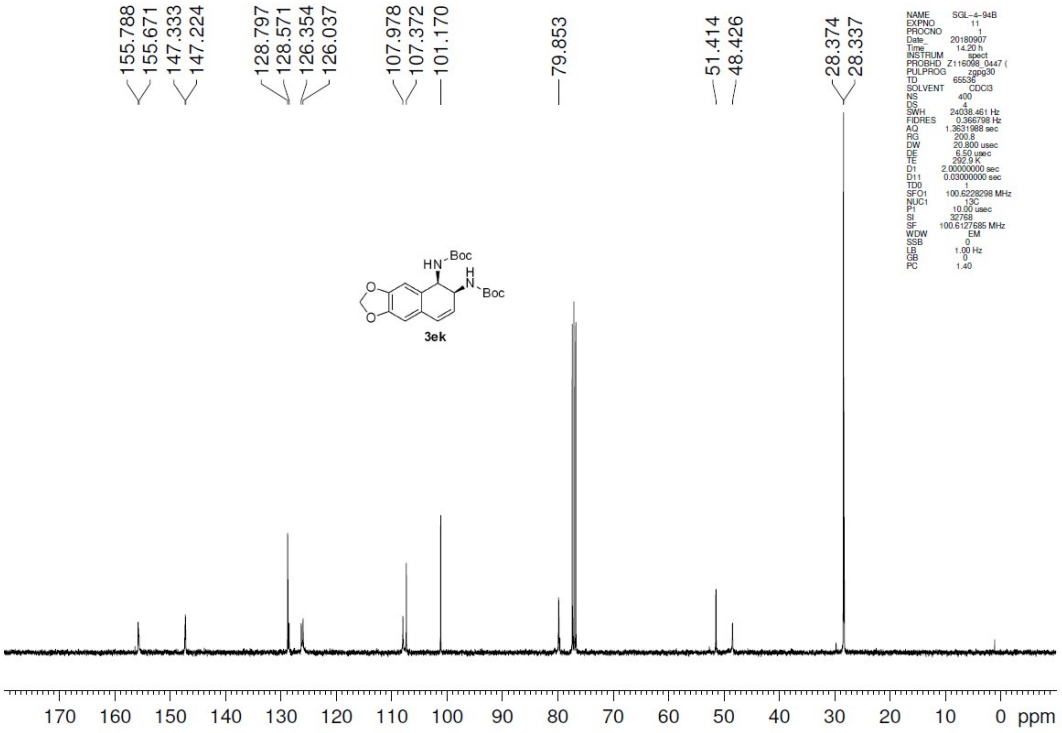

7.092
6.872
6.477
6.453
5.898
5.888
5.875
5.865
4.933
4.739
4.719
4.534
2.243
2.221
1.473
1.431
HN
Boc
H
N
Boc
Me
Me
3ck
ppm
1.03
1.03
1.00
1.02
1.90
0.96
1.02
6.06
18.20

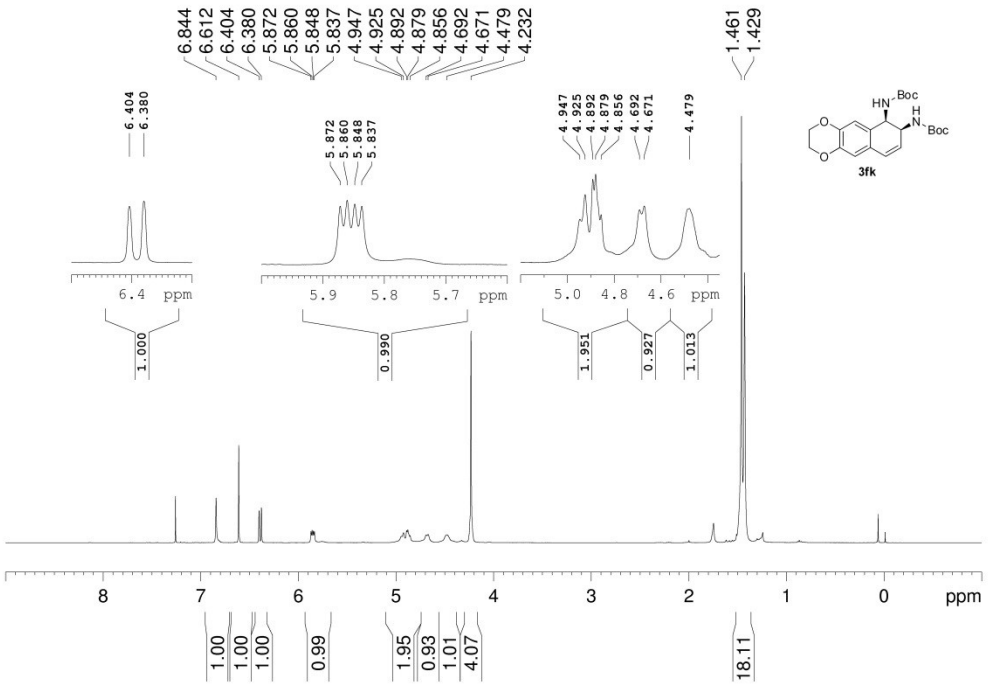

155.856
155.643
136.793
136.311
131.438
129.885
128.957
128.216
128.082
126.922
79.742
51.091
48.395
28.398
28.346
19.777
19.406
HN
Boc
H
N
Boc
Me
Me
3ck
ppm

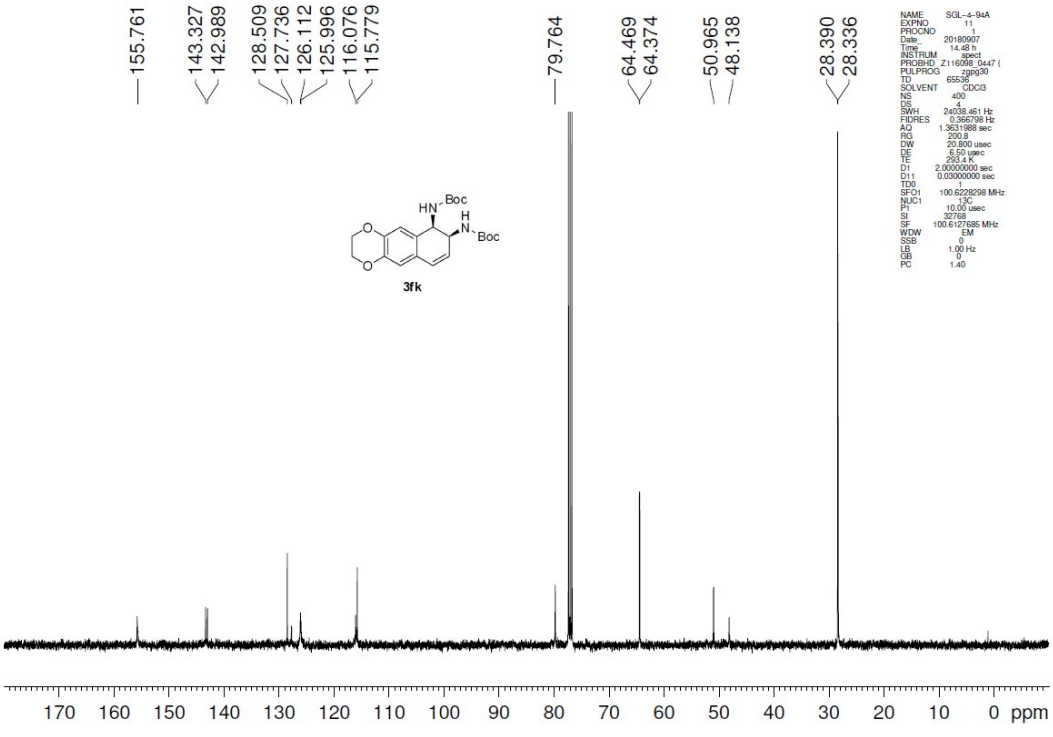

7.570
7.342
6.457
6.432
6.105
6.094
6.082
6.070
5.042
5.020
4.941
4.931
4.908
4.558
4.533
1.481
1.428
Boc
HN
H
N
Boc
Br
Br
3dk
6.5 6.4 6.3 6.2 6.1 6.0 5.9 ppm
5.2 5.0 4.8 4.6 ppm
1.000
1.005
1.960
2.162
8 7 6 5 4 3 2 1 0 ppm
0.94
0.97
1.00
1.01
1.96
2.16
18.07

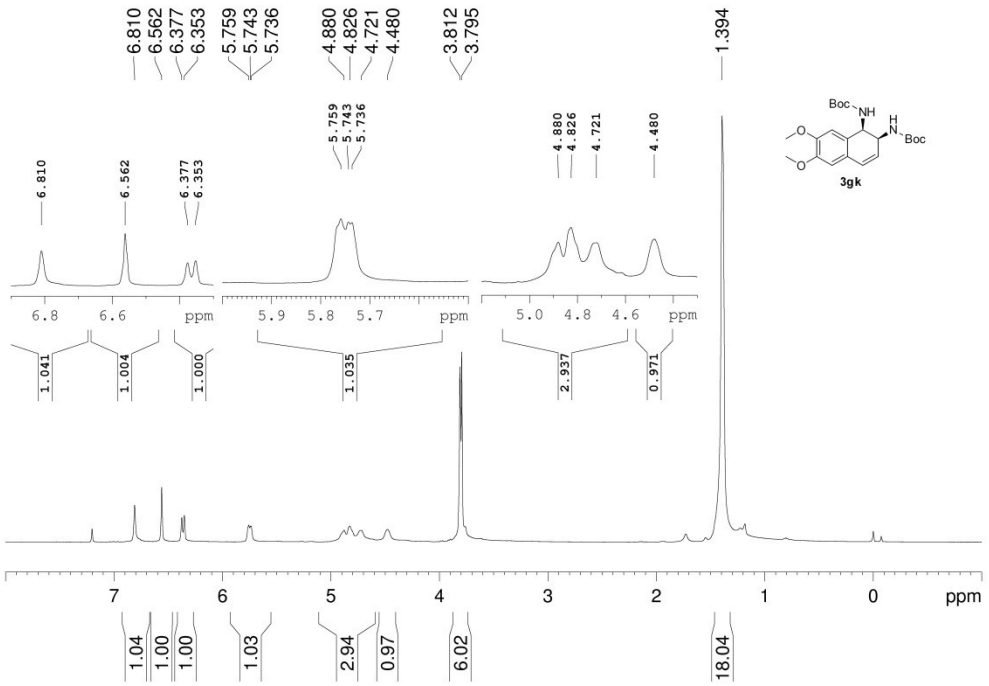

155.731
155.504
134.860
132.947
131.721
131.436
129.479
127.448
124.125
124.070
80.324
80.200
50.913
47.190
28.351
28.286
NAME SGL-s-64B
EXPNO 11
PROCNO 1
Date 20180907
Time 16.13 h
INSTRUM spect
PROBHD Z116098_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.8
DW 20.800 usec
DE 6.50 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
HN
H
N
Boc
Br
Br
3dk
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

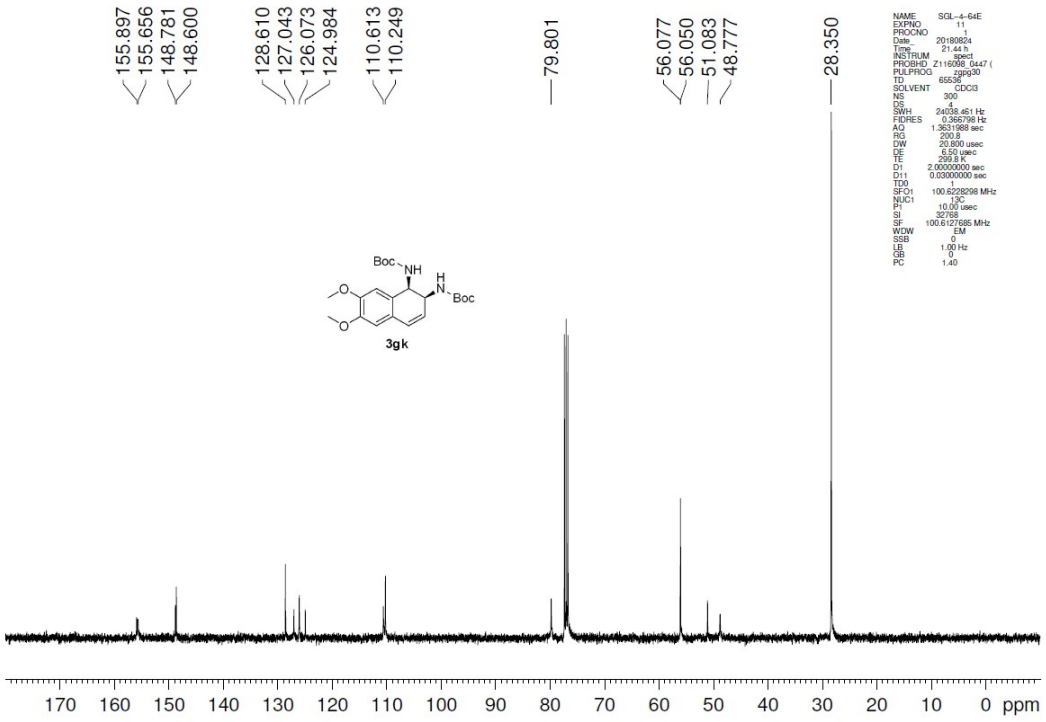

6.836
6.572
6.389
6.365
5.930
5.912
5.845
5.835
5.822
5.811
4.994
4.972
4.870
4.858
4.835
4.763
4.743
4.501
1.450
1.429
HN
Boc
H
N
Boc
3ek
6.4
6.2
6.0
ppm
5.0
4.8
4.6
ppm
1.000
2.006
0.961
0.890
1.104
0.882
1.135
8
7
6
5
4
3
2
1
0
ppm
1.00
0.98
1.00
2.01
0.96
0.89
1.10
0.88
1.14
18.18


155.788
155.671
147.333
147.224
128.797
128.571
126.354
126.037
107.978
107.372
101.170
79.853
51.414
48.426
28.374
28.337
NAME SGL--4-94B
EXPNO 11
PROCNO 1
Date_ 20180907
Time 14.20 h
INSTRUM spect
PROBHD Z116098_0447 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203.8
DW 20.800 usec
DE 6.50 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
Boc
H
N
Boc
3ek
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

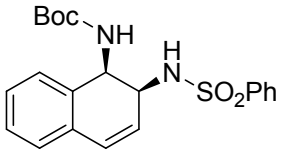

6.844
6.612
6.404
6.380
5.872
5.860
5.848
5.837
4.947
4.925
4.892
4.879
4.856
4.692
4.671
4.479
4.232
1.461
1.429
3fk
6.4 ppm
5.9 5.8 5.7 ppm
5.0 4.8 4.6 ppm
1.000
0.990
1.951
0.927
1.013
8 7 6 5 4 3 2 1 0 ppm
1.00
1.00
1.00
0.99
1.95
0.93
1.01
4.07
18.11


155.761
143.327
142.989
128.509
127.736
126.112
125.996
116.076
115.779
79.764
64.469
64.374
50.965
48.138
28.390
28.336
3fk
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


6.810
6.562
6.377
6.353
5.759
5.743
5.736
4.880
4.826
4.721
4.480
3.812
3.795
1.394
Boc
NH
H
N
Boc
3gk
1.041
1.004
1.000
1.035
2.937
0.971
ppm
1.04
1.00
1.00
1.03
2.94
0.97
6.02
18.04

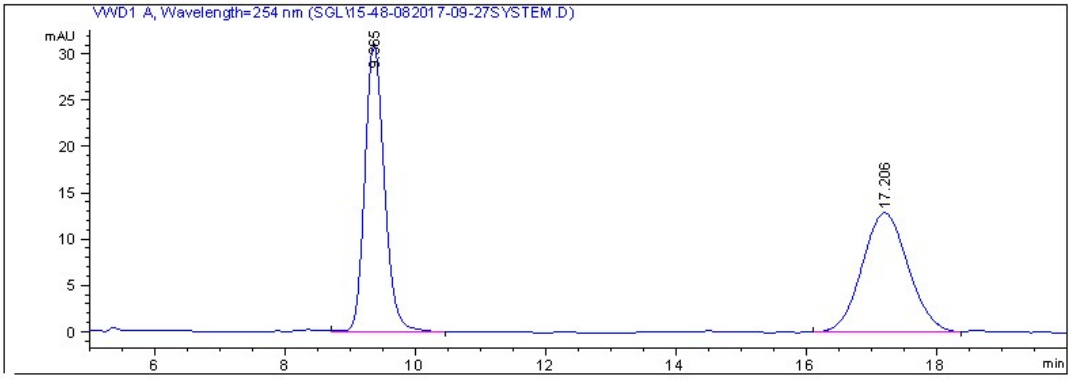

155.897
155.656
148.781
148.600
128.610
127.043
126.073
124.984
110.613
110.249
79.801
56.077
56.050
51.083
48.777
28.350
Boc
NH
H
N
Boc
3gk
ppm

E: HPLC Spectra of Products



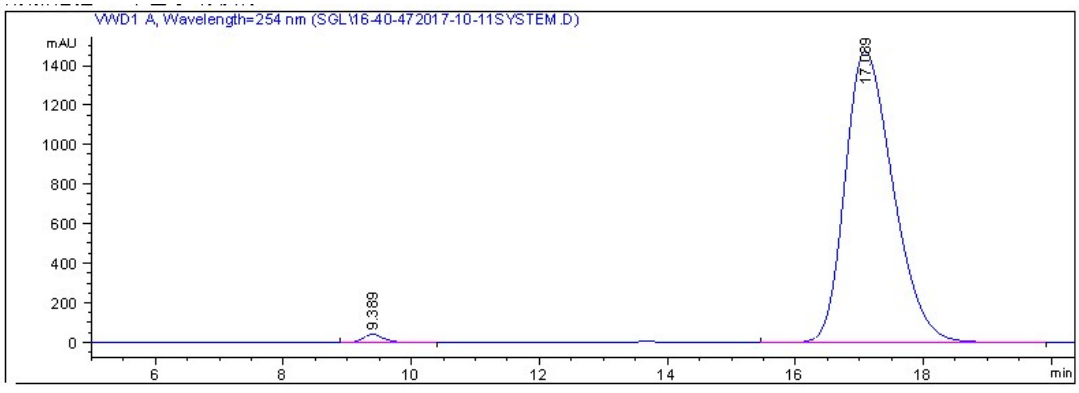
3aa

Racemic:

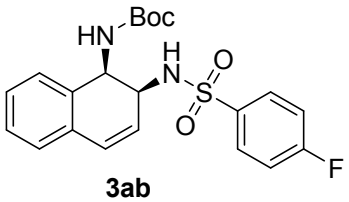


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.365	BB	0.3197	643.53711	31.06831	50.3758
2	17.206	BB	0.7617	633.93622	12.89164	49.6242

Enantioenriched:

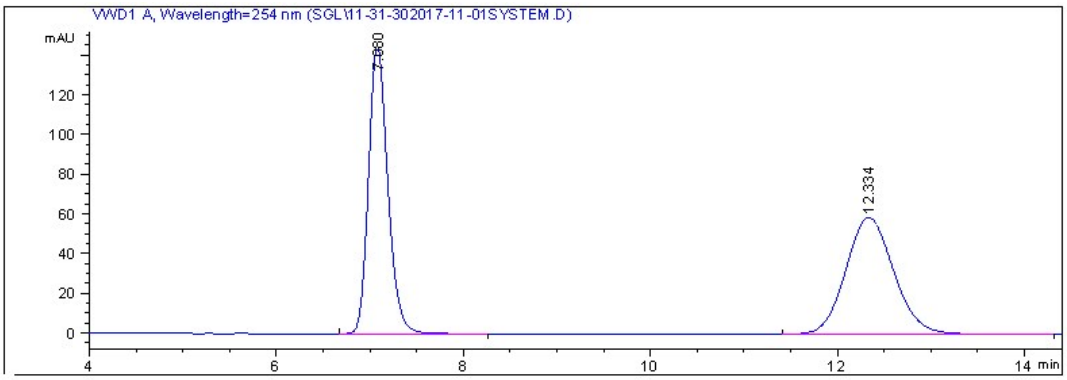


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.389	BB	0.3221	830.87878	39.70722	1.0747
2	17.089	BB	0.8034	7.64798e4	1473.77979	98.9253



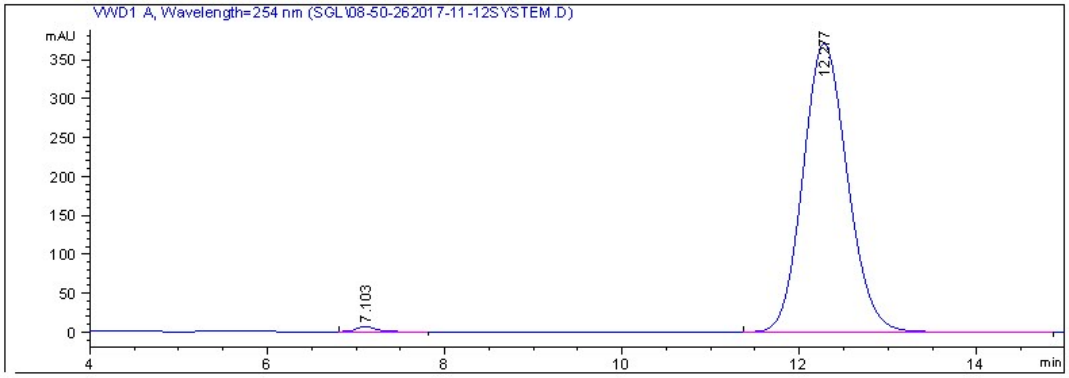
3ab

Racemic:

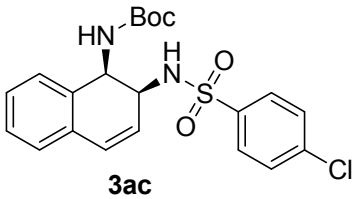


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.080	BB	0.2194	2067.21021	144.91815	49.9044
2	12.334	BB	0.5499	2075.13379	58.63215	50.0956

Enantioenriched:

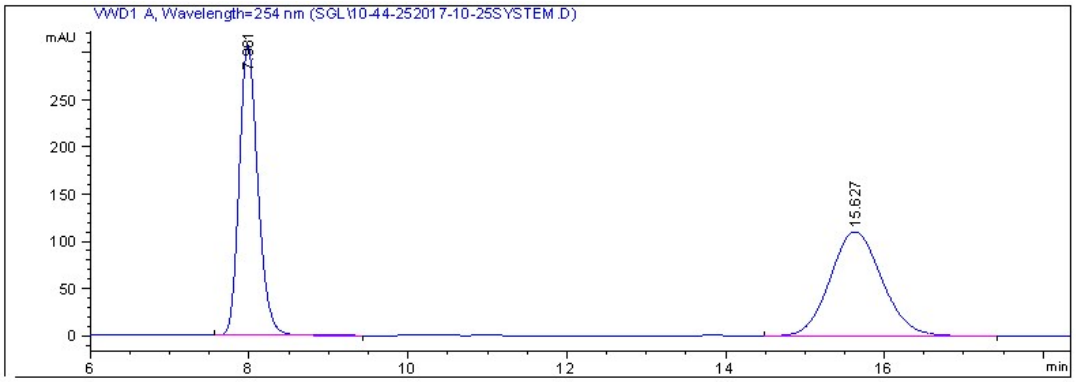


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.103	BB	0.2150	89.83296	6.42899	0.6948
2	12.277	BB	0.5393	1.28391e4	369.60922	99.3052



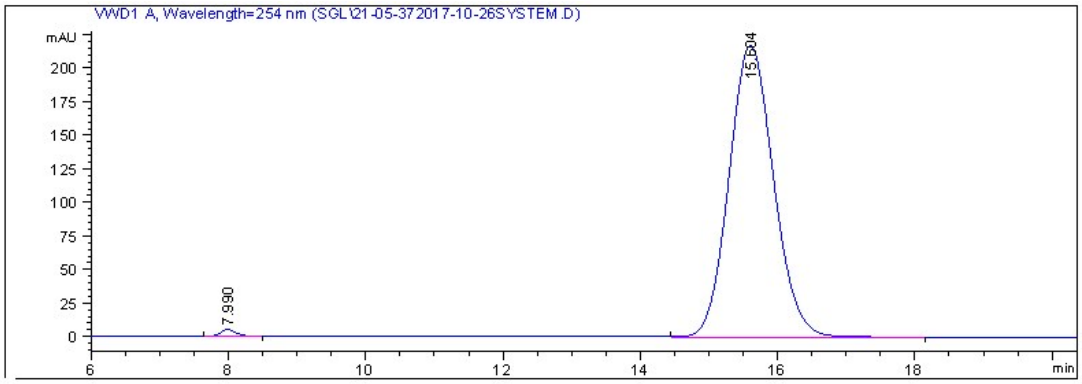
3ac

Racemic:

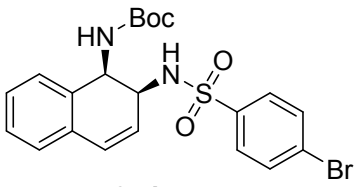


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.981	BB	0.2515	5020.15137	307.41354	50.1666
2	15.627	BB	0.7014	4986.80176	110.31419	49.8334

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.990	BB	0.2483	82.25282	5.09647	0.8352
2	15.604	BB	0.6984	9765.84766	217.25140	99.1648



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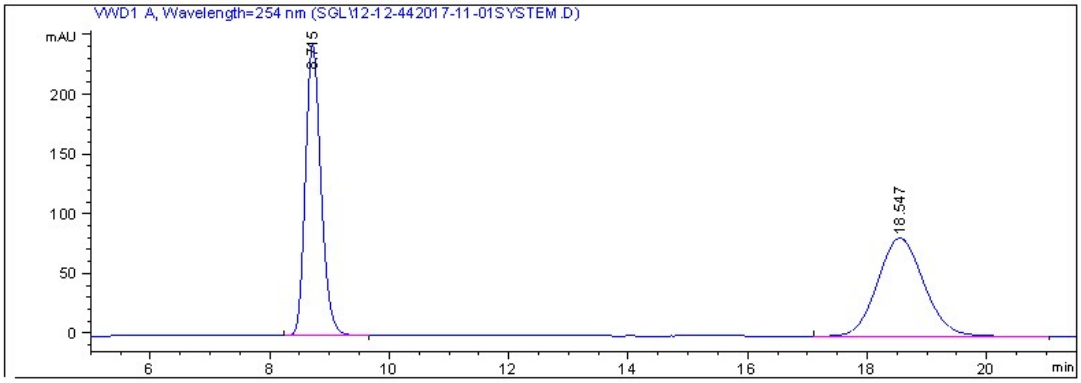
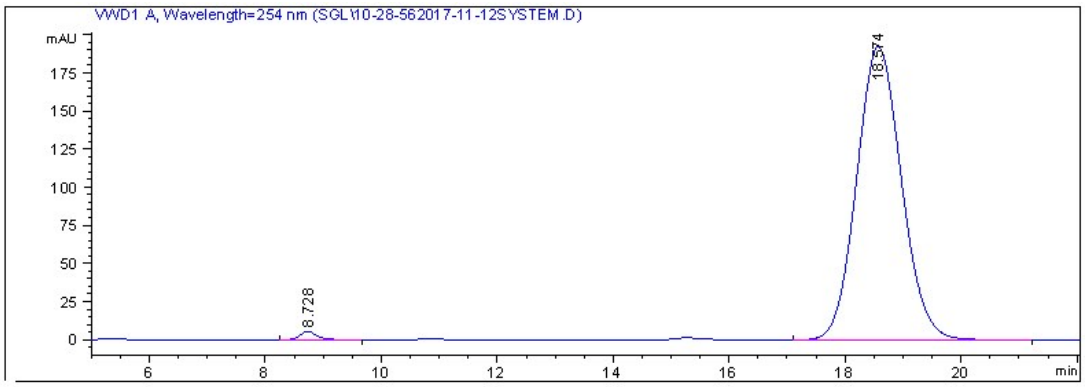
Racemic:

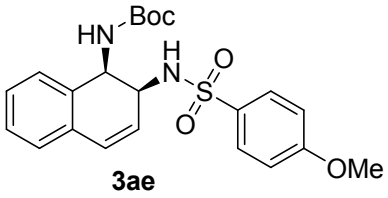


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.715	BB	0.2781	4407.02686	243.60928	50.0661
2	18.547	BB	0.8324	4395.39502	81.99196	49.9339

Enantioenriched:

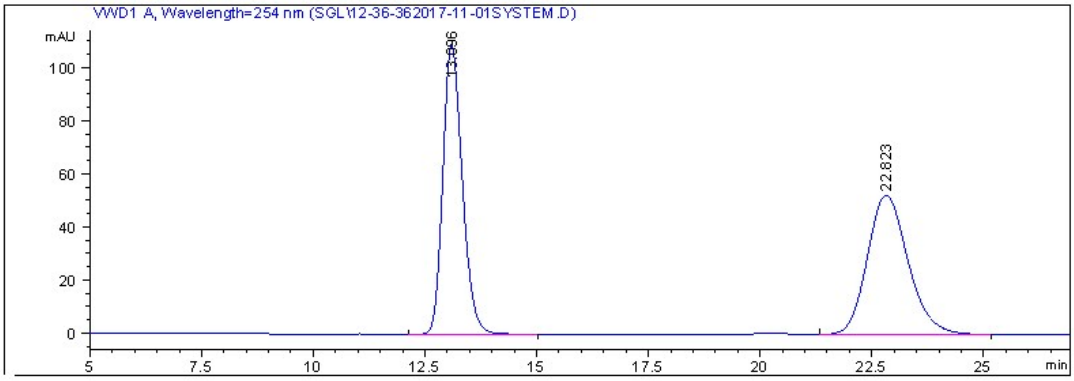


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.728	BB	0.2858	99.55552	5.30921	0.9555
2	18.574	BB	0.8366	1.03194e4	192.44432	99.0445



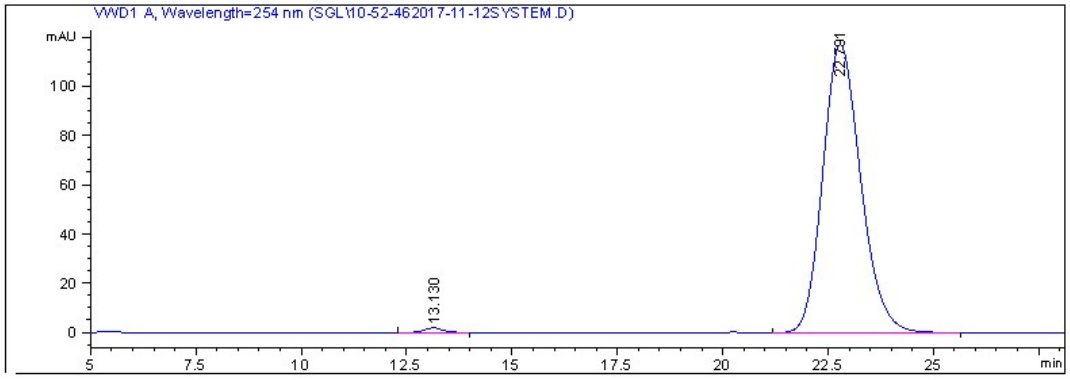
3ae

Racemic:

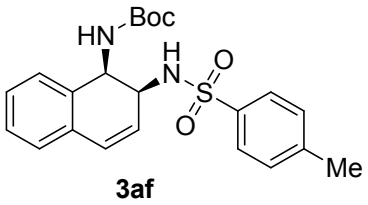


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.096	BB	0.4665	3292.96167	108.99734	49.9347
2	22.823	BB	0.9721	3301.57007	52.32901	50.0653

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.130	BB	0.4539	54.54976	1.80358	0.7384
2	22.791	BB	0.9713	7332.60156	117.13631	99.2616



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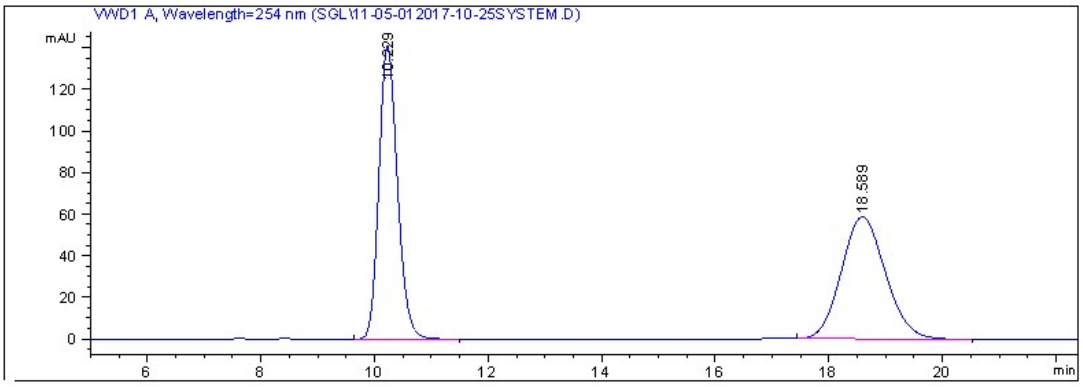
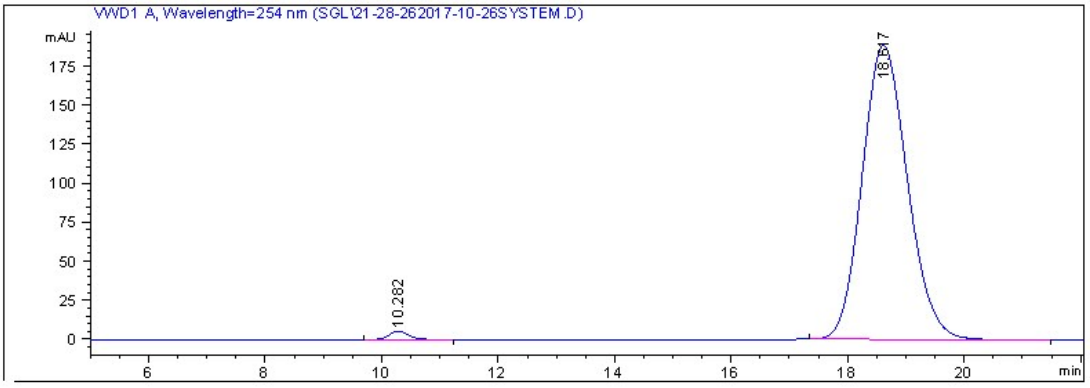
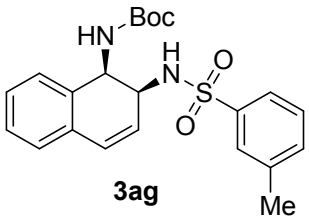


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.229	BB	0.3496	3183.48022	140.88254	50.2645
2	18.589	BB	0.8403	3149.97144	58.58482	49.7355

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.282	BB	0.3519	120.35932	5.26160	1.1704
2	18.617	BB	0.8373	1.01633e4	188.42487	98.8296



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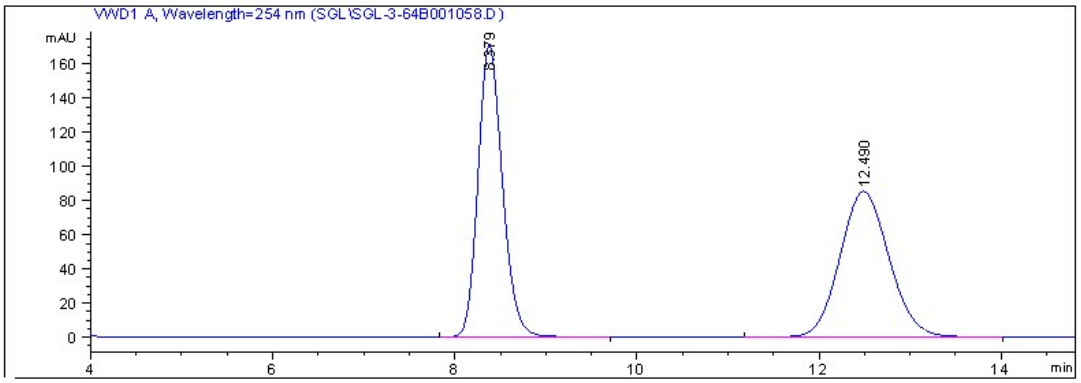
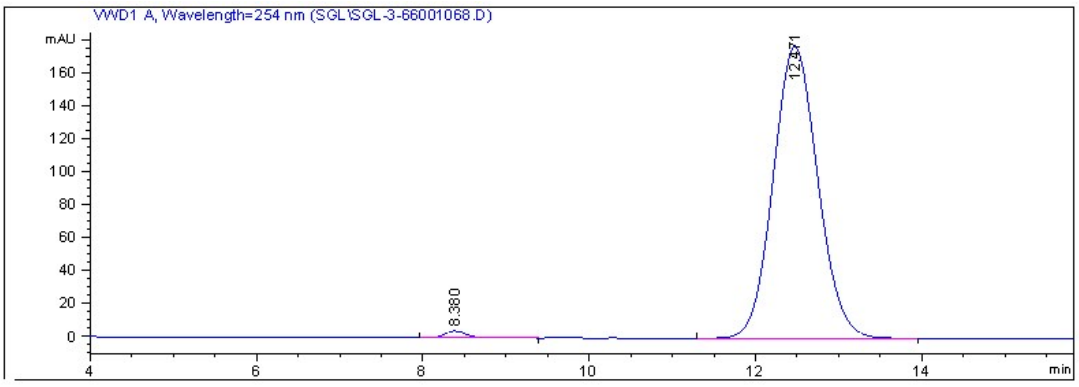
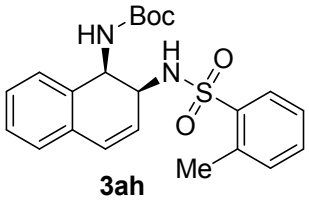


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.379	BB	0.2871	3171.75586	170.43819	49.8863
2	12.490	BB	0.5808	3186.21362	85.29987	50.1137

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.380	BB	0.2936	78.78133	4.11090	1.1770
2	12.471	BB	0.5807	6614.66455	177.10867	98.8230



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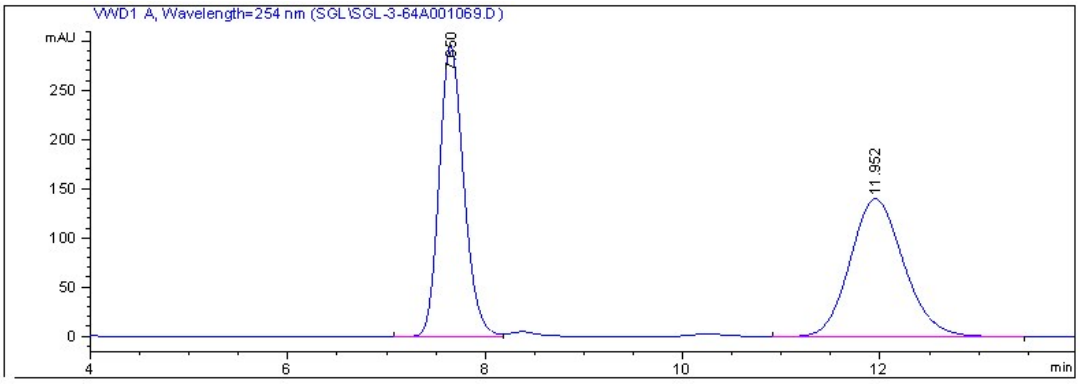
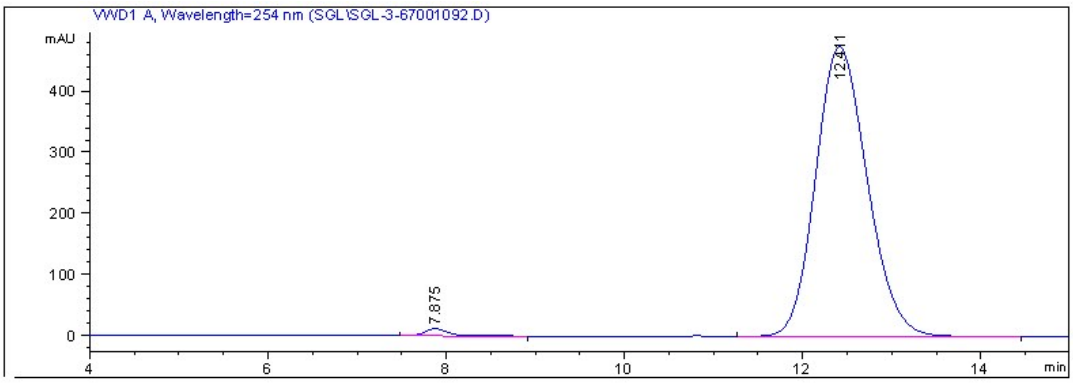
Racemic:

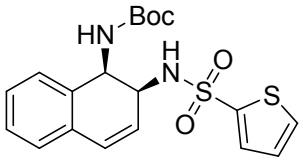


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.650	BV	0.2664	5109.42480	296.02634	49.2347
2	11.952	BB	0.5832	5268.26465	139.93561	50.7653

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.875	BB	0.2793	213.74620	11.74883	1.1090
2	12.411	BB	0.6244	1.90593e4	474.75104	98.8910



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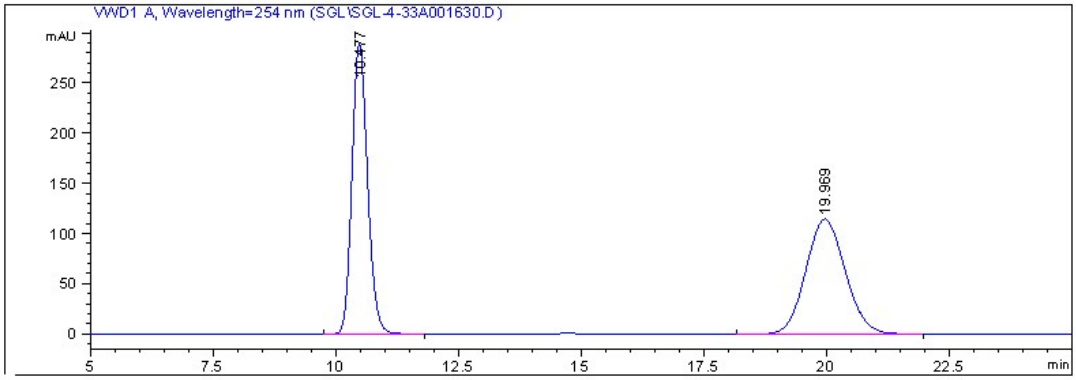
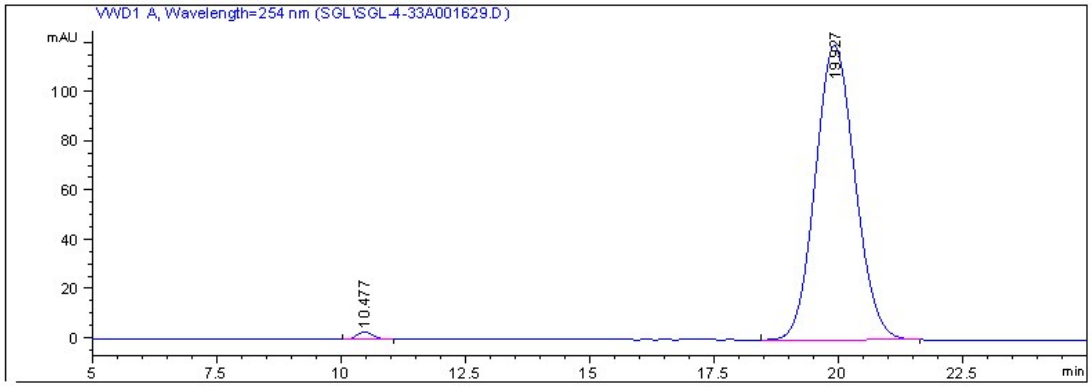
Racemic:

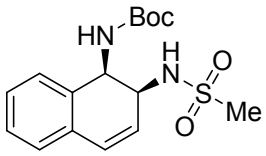


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.477	BB	0.3413	6369.62012	288.75998	49.8793
2	19.969	BB	0.8751	6400.43945	114.68821	50.1207

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.477	BB	0.3399	66.63047	3.03827	0.9916
2	19.927	BB	0.8600	6652.76758	119.59025	99.0084



3aj

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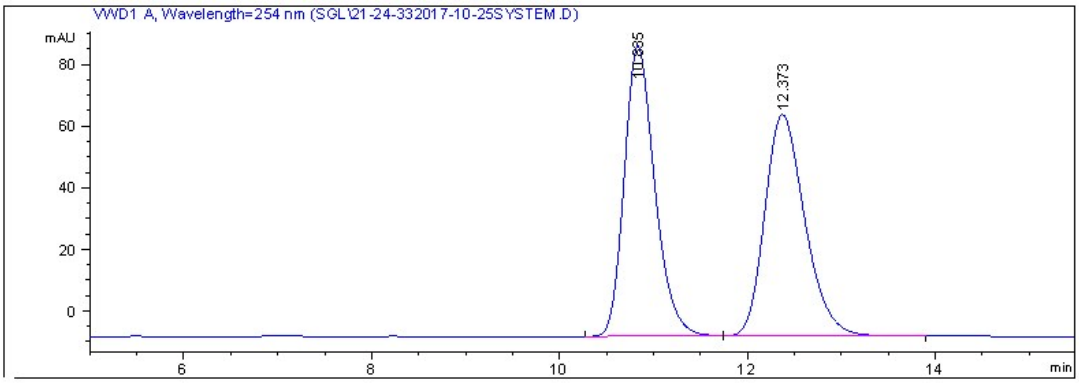
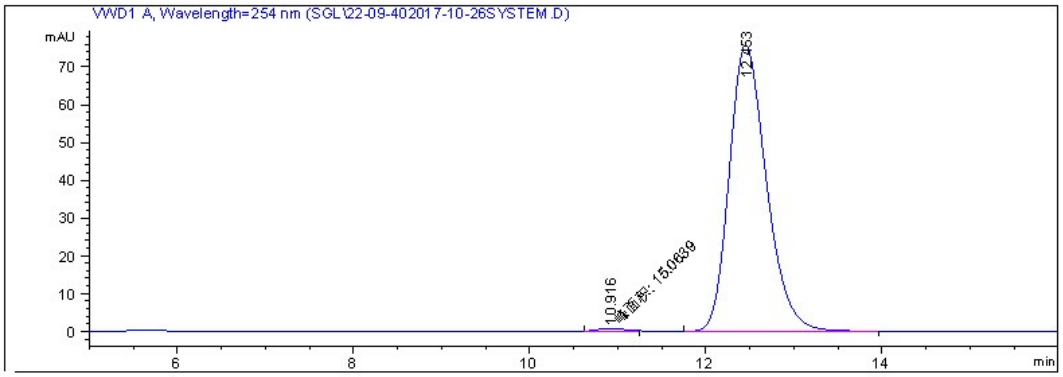
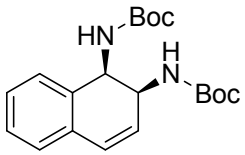


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.835	BB	0.3441	2124.20972	94.20878	49.9873
2	12.373	BB	0.4552	2125.28516	71.83150	50.0127

Enantioenriched:

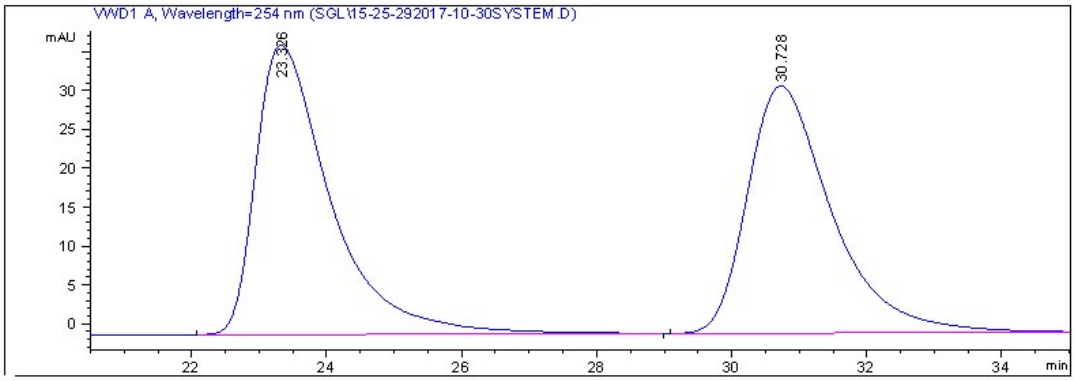


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.916	MM	0.3501	15.06394	7.17135e-1	0.6759
2	12.453	BB	0.4496	2213.61353	75.81379	99.3241



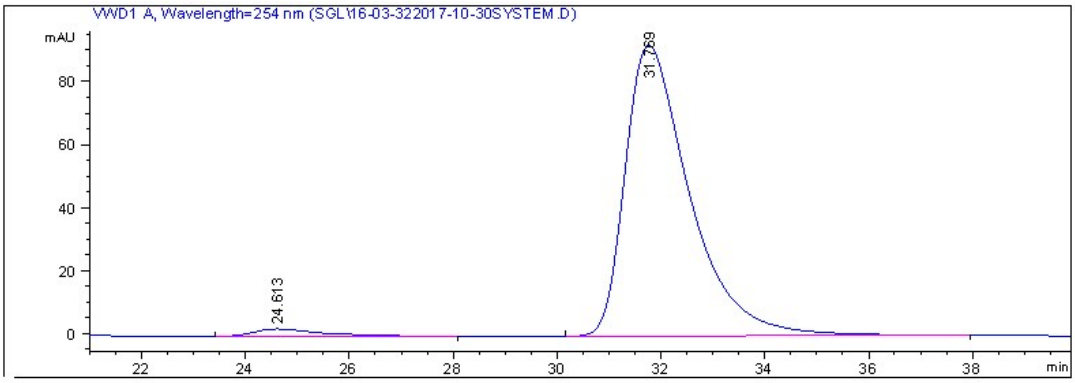
3ak

Racemic:

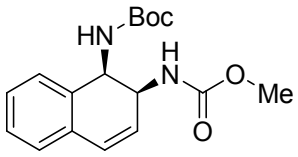


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.326	BB	1.1503	2866.37085	37.18521	50.9632
2	30.728	BBA	1.2952	2758.02563	31.75651	49.0368

Enantioenriched:

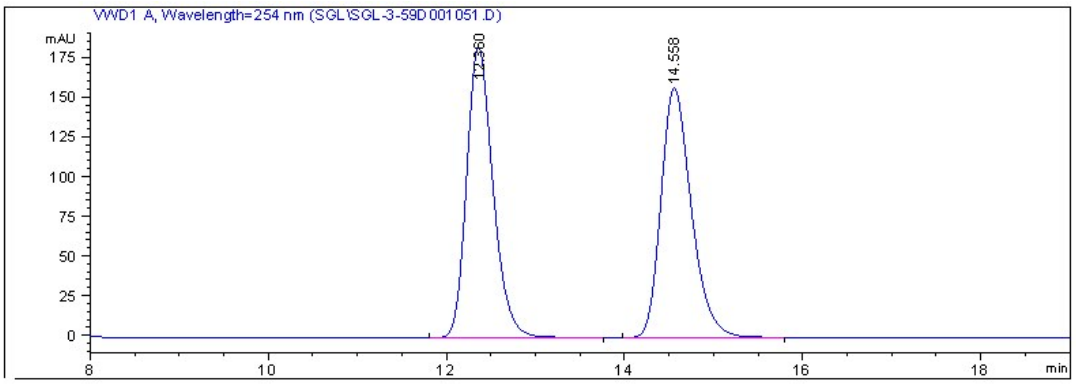


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.613	BB	1.0882	198.66302	2.31044	2.4307
2	31.769	BB	1.2962	7974.36621	92.28083	97.5693



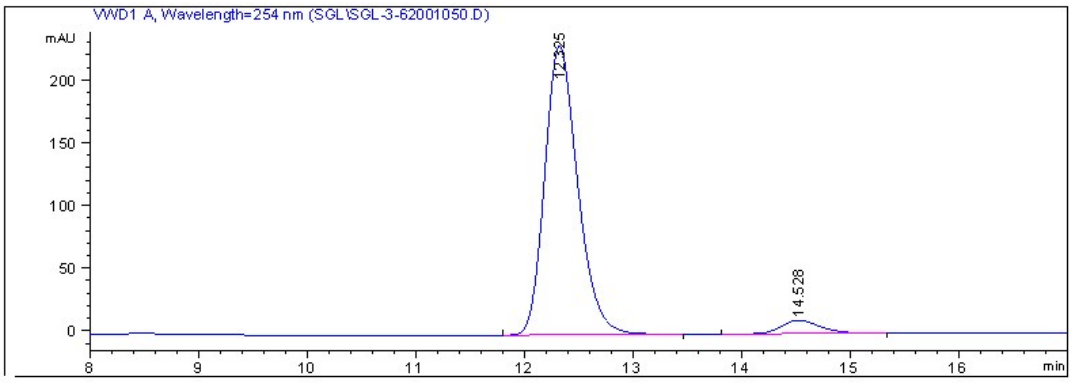
3al

Racemic:

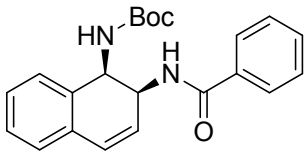


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.360	BB	0.3143	3750.72876	182.83127	50.2613
2	14.558	BB	0.3623	3711.73389	156.71568	49.7387

Enantioenriched:

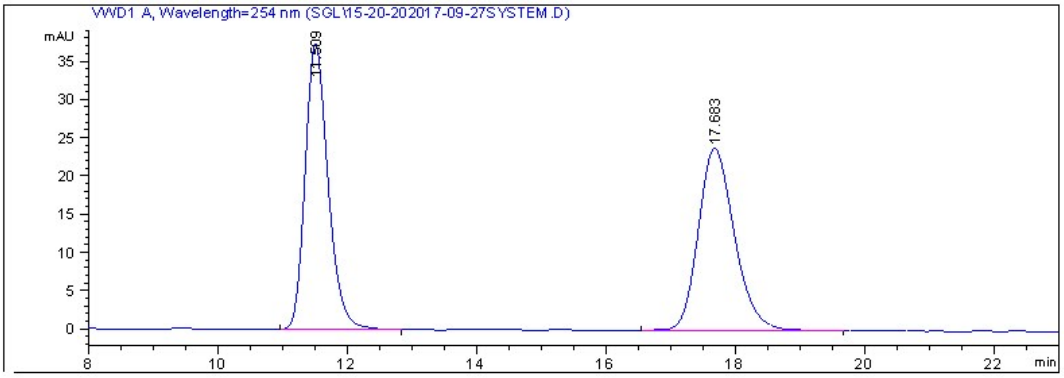


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.325	BB	0.3249	4872.92969	230.25255	94.9605
2	14.528	BB	0.3755	258.60477	10.56740	5.0395



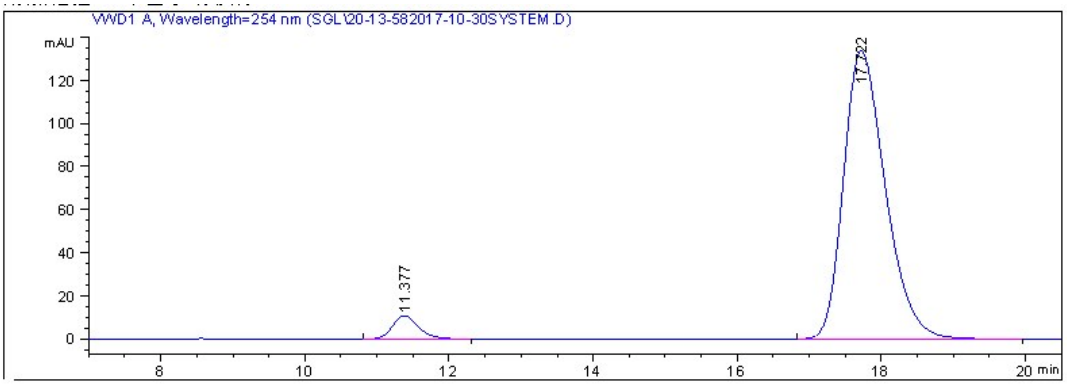
3am

Racemic:

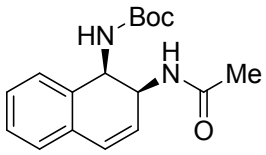


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.509	BB	0.3766	919.70013	37.31271	49.6280
2	17.683	BB	0.6022	933.48645	23.82291	50.3720

Enantioenriched:

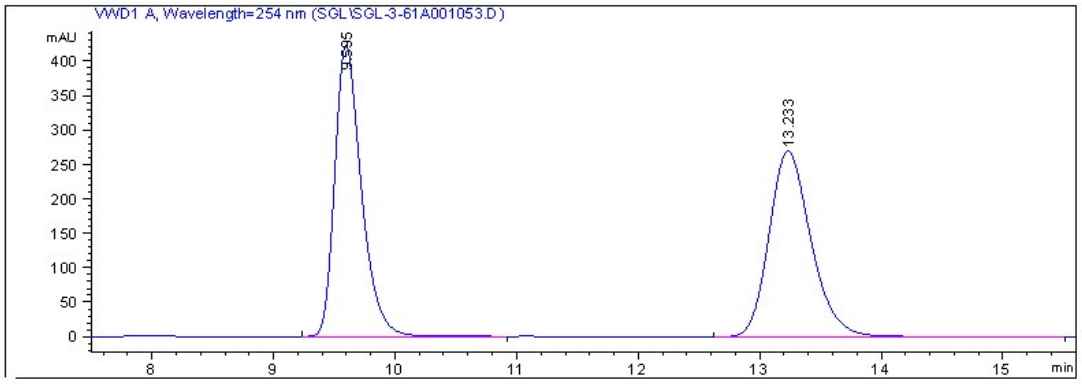


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.377	BB	0.3805	266.20831	10.72866	4.7132
2	17.722	BB	0.6192	5381.94531	133.81926	95.2868



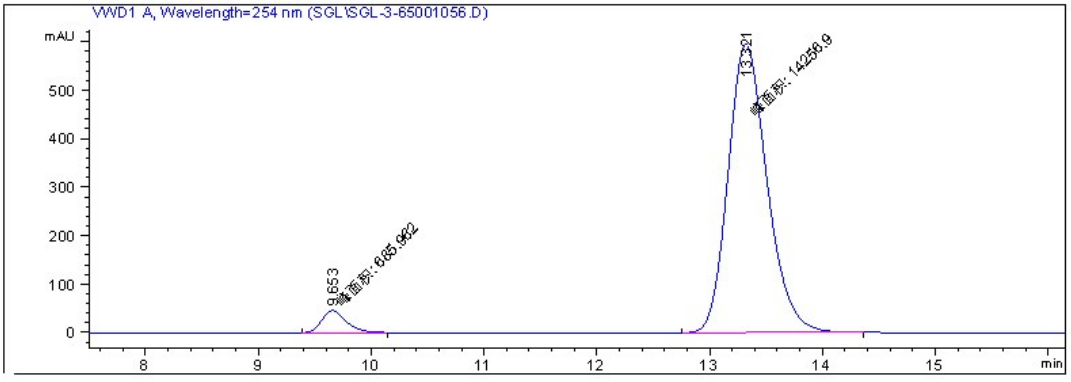
3an

Racemic:

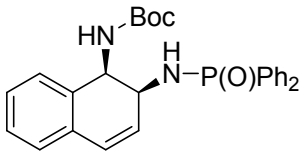


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.595	BB	0.2316	6426.71387	422.25473	49.8384
2	13.233	BB	0.3677	6468.38574	269.82254	50.1616

Enantioenriched:

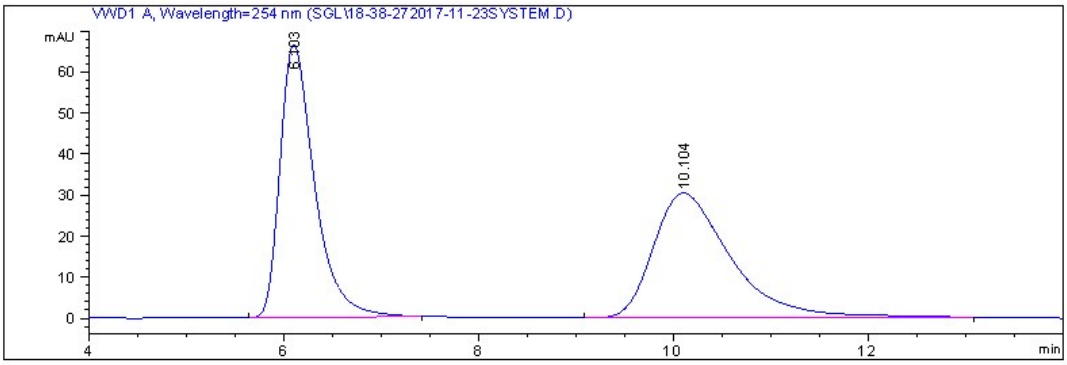


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.653	MM	0.2514	685.96185	45.47303	4.5906
2	13.321	MM	0.3991	1.42569e4	595.39813	95.4094



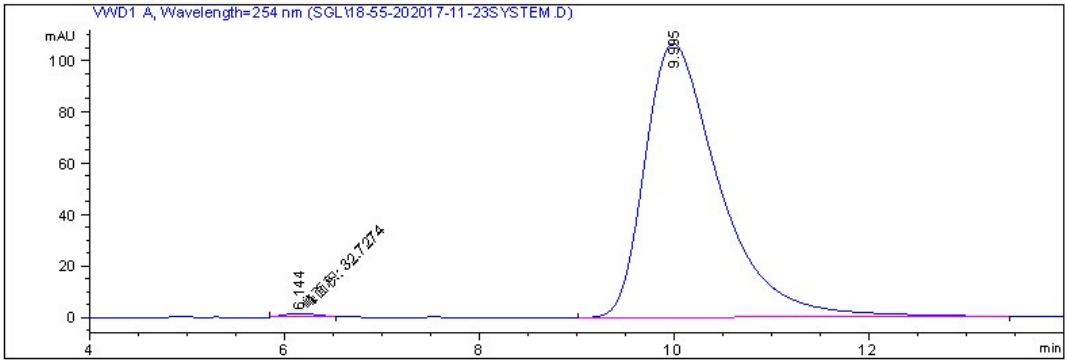
3ao

Racemic:

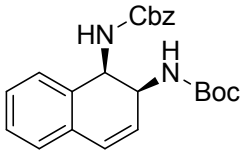


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.103	BB	0.3736	1641.77039	66.60519	49.4865
2	10.104	BB	0.8263	1675.84277	30.39302	50.5135

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.144	MM	0.3647	32.72740	1.49577	0.5748
2	9.995	BB	0.8008	5660.61963	106.54388	99.4252



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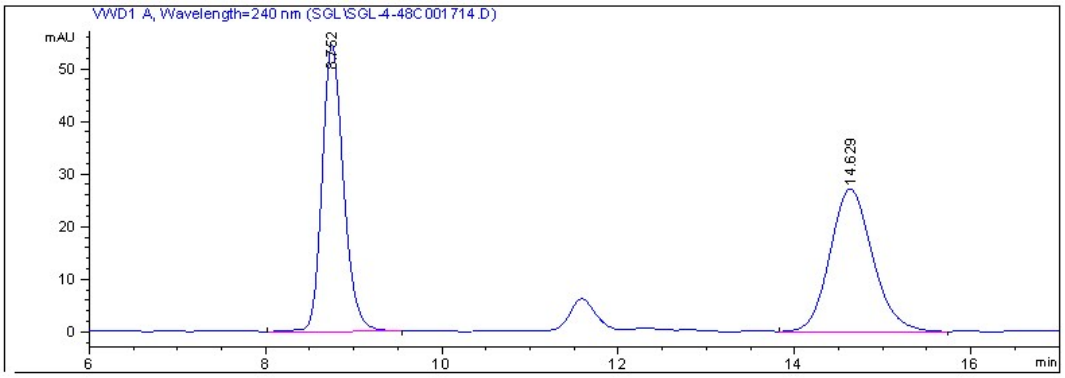
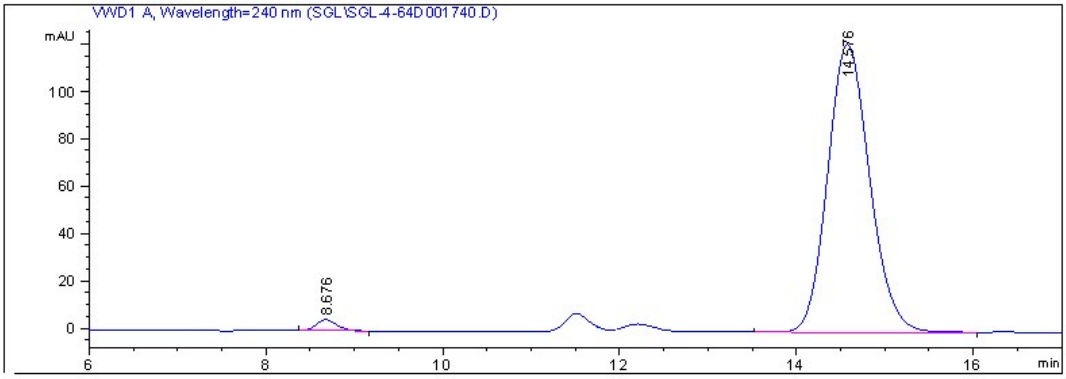
Racemic:

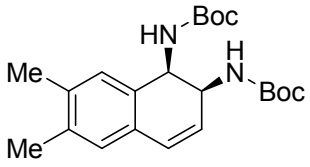


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.752	BB	0.2600	923.99994	54.45257	50.4136
2	14.629	BB	0.5169	908.83984	27.12661	49.5864

Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.676	BB	0.2475	75.07973	4.67161	1.8342
2	14.576	BB	0.5115	4018.25293	121.59698	98.1658



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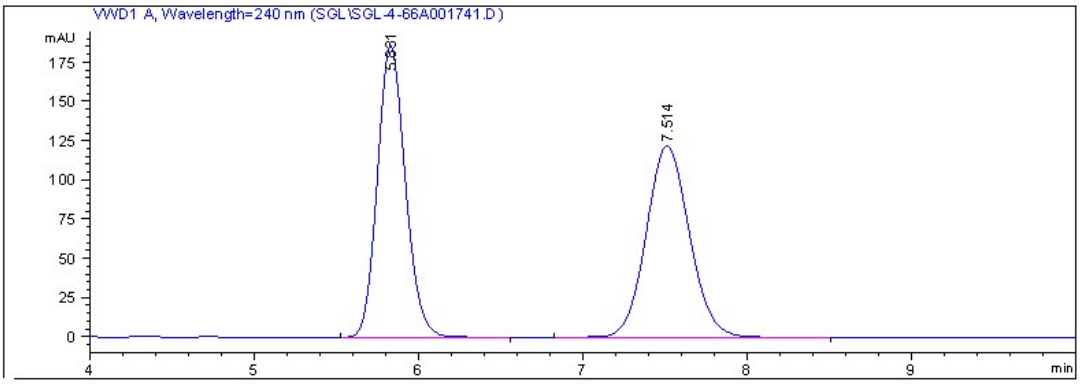
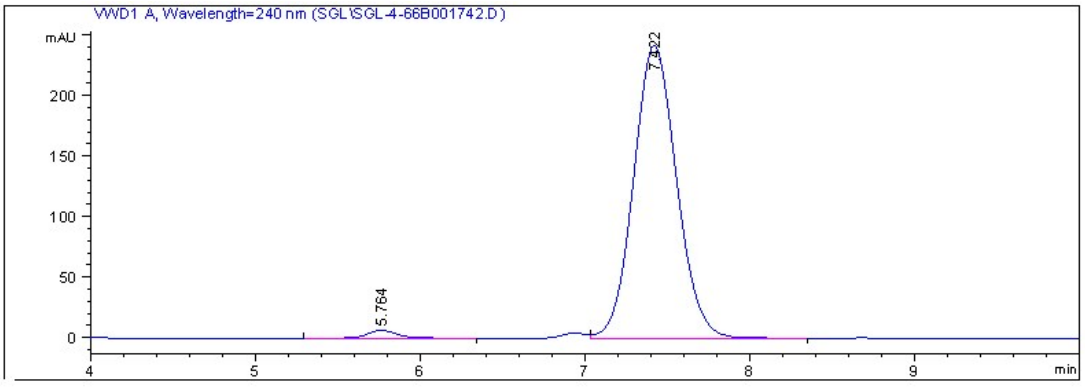
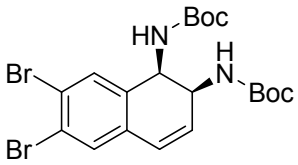


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.831	BB	0.1832	2201.65918	185.83861	49.5948
2	7.514	BB	0.2837	2237.63867	122.18274	50.4052

Enantioenriched:

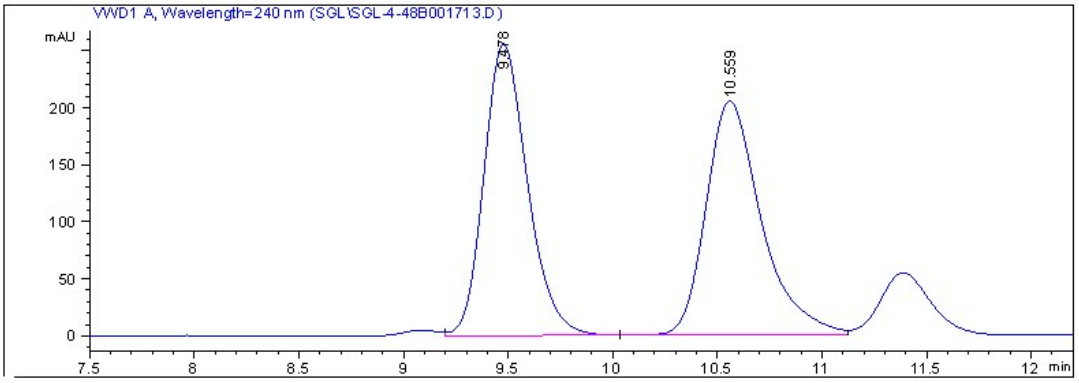


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.764	BB	0.1965	86.05535	6.62513	1.9219
2	7.422	VB	0.2820	4391.63428	241.71465	98.0781



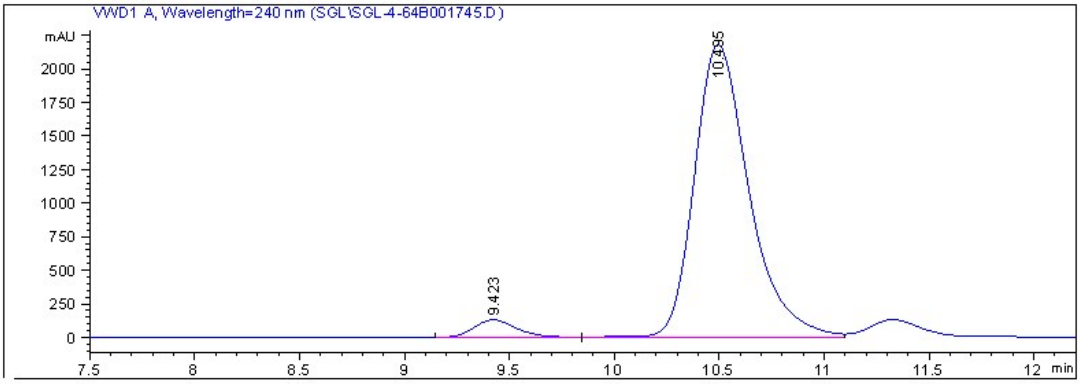
3dk

Racemic:

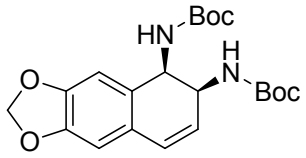


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.478	VB	0.2222	3693.61108	254.73824	49.3700
2	10.559	BV	0.2794	3787.87427	205.14586	50.6300

Enantioenriched:

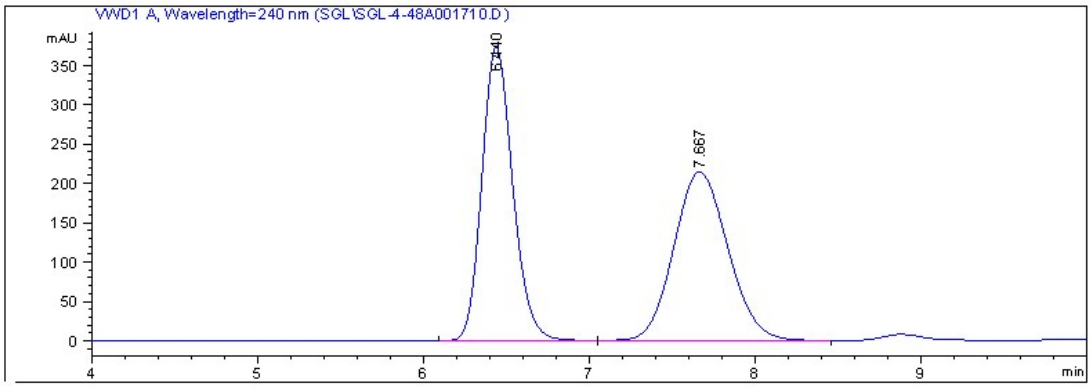


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.423	VV	0.2165	1827.50085	129.63821	4.4264
2	10.495	VV	0.2762	3.94593e4	2180.14307	95.5736



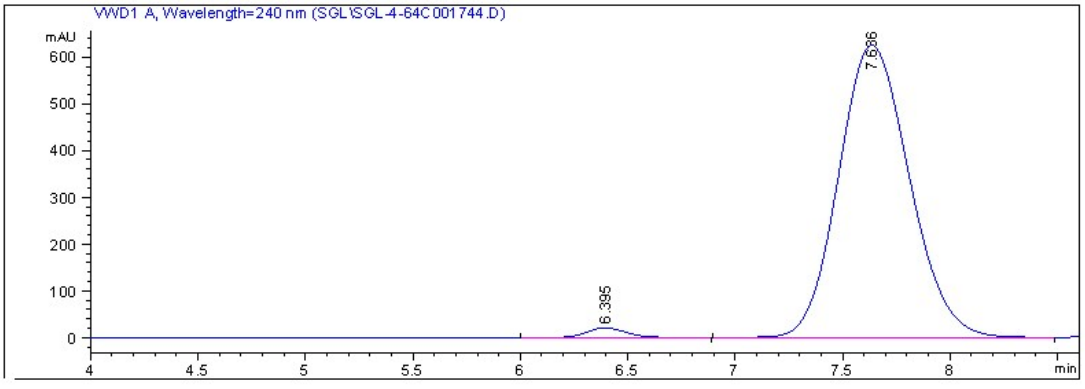
3ek

Racemic:

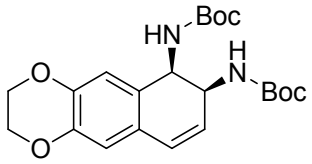


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.440	VB	0.1996	4831.85742	374.28995	49.8962
2	7.667	BB	0.3530	4851.96875	214.44601	50.1038

Enantioenriched:

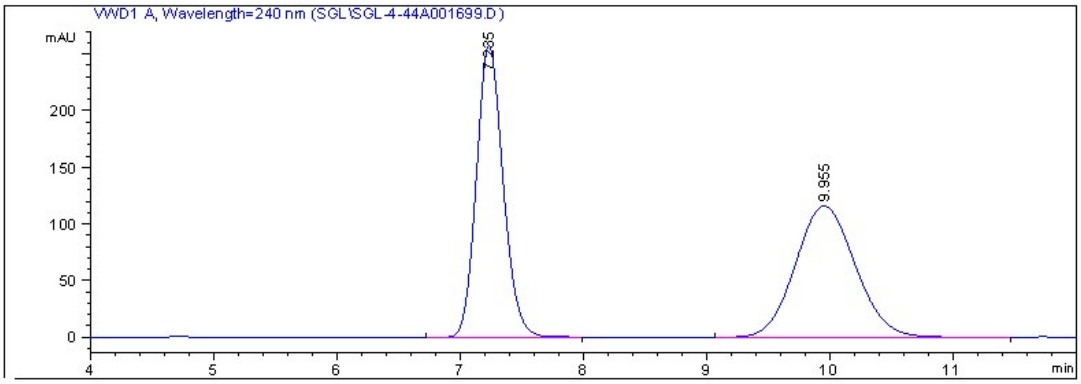


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.395	BB	0.1962	269.36316	21.06955	1.8441
2	7.636	BV	0.3574	1.43376e4	625.55518	98.1559



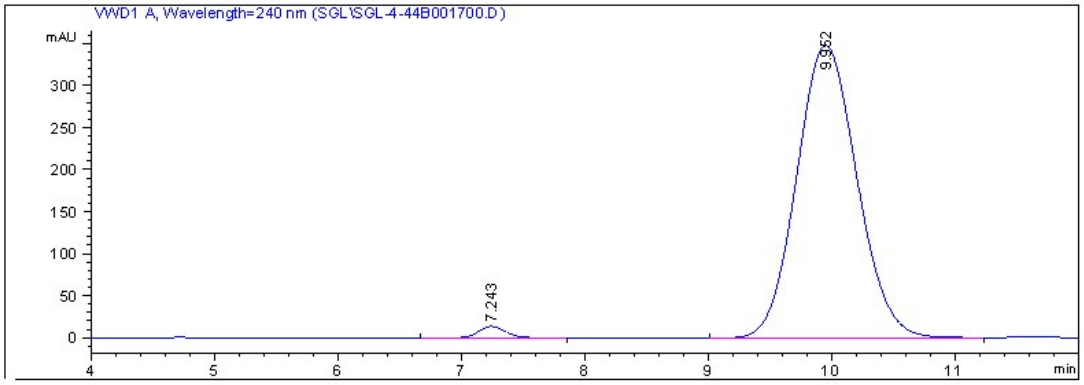
3fk

Racemic:

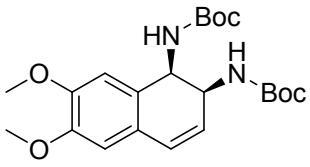


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.235	BB	0.2340	3886.33301	257.55435	49.7370
2	9.955	BB	0.5290	3927.44067	115.99681	50.2630

Enantioenriched:

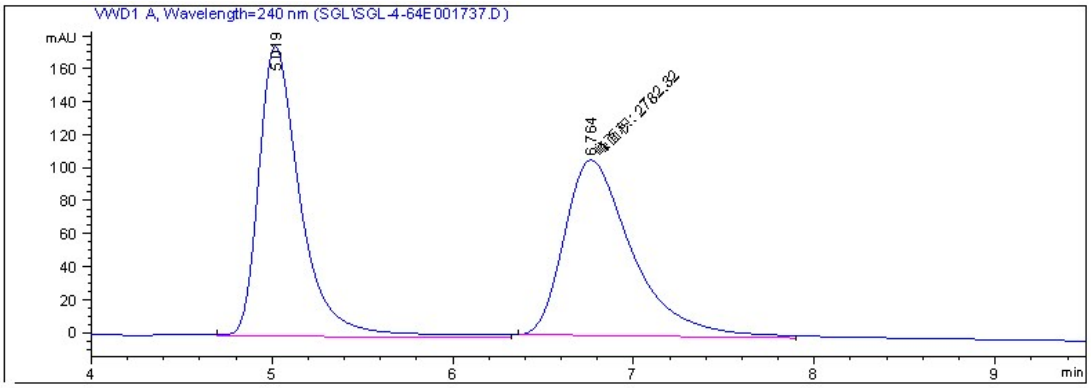


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.243	BB	0.2361	212.05009	13.80934	1.7668
2	9.952	BB	0.5281	1.17896e4	348.09711	98.2332



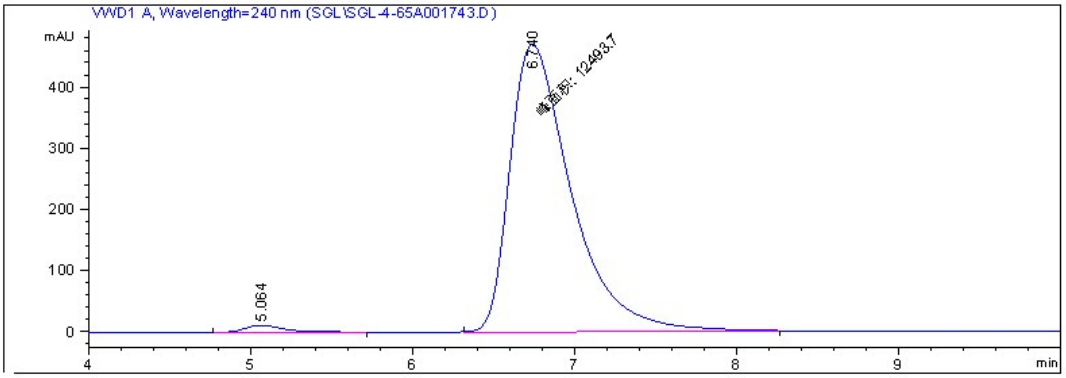
3gk

Racemic:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.019	VV	0.2421	2837.10791	175.93347	50.4875
2	6.764	MM	0.4364	2782.31665	106.26049	49.5125

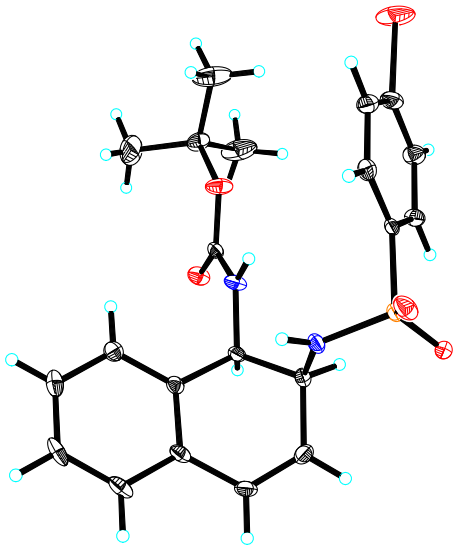
Enantioenriched:



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.064	BB	0.2471	187.90674	11.47426	1.4817
2	6.740	MM	0.4414	1.24937e4	471.76736	98.5183

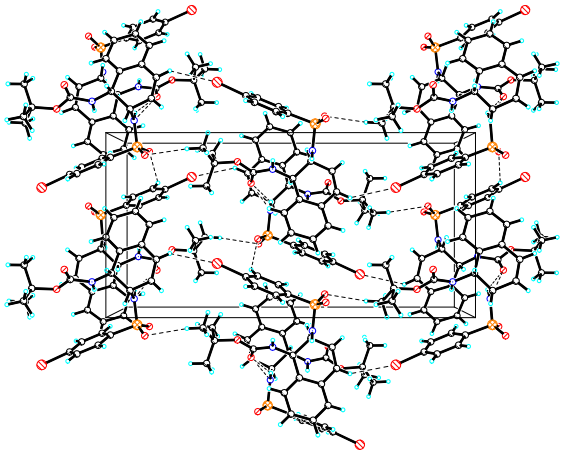
F: X-Ray Crystallography of Compound 3ad

Crystal data for cu_qsgl359b2_0m: $C_{21}H_{23}BrN_2O_4S$, M = 479.38, a = 9.4007(2) Å, b = 10.8468(2) Å, c = 21.7267(5) Å, α = 90°, β = 90°, γ = 90°, V = 2215.42(8) Å^3, T = 100(2) K, space group $P212121$, Z = 4, μ(CuKα) = 3.653 mm^{-1}, 13347 reflections measured, 3808 independent reflections (R_{int} = 0.0426). The final R_1 values were 0.0364 ($I>2\sigma(I)$). The final $wR(F^2)$ values were 0.0944 ($I> 2\sigma(I)$). The final R_1 values were 0.0365 (all data). The final $wR(F^2)$ values were 0.0946 (all data). The goodness of fit on F^2 was 1.087. Flack parameter = 0.089(6).



View of a molecule of qsgl359b2 with the atom-labelling scheme.

Displacement ellipsoids are drawn at the 30% probability level.



View of the pack drawing of qsgl359b2.

Hydrogen-bonds are shown as dashed lines.

Table 1.Crystal data and structure refinement for cu_qsgl359b2_0m.

Identification code	cu_qsgl359b2_0m	
Empirical formula	C21 H23 Br N2 O4 S	
Formula weight	479.38	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 9.4007(2) Å	α= 90°.
	b = 10.8468(2) Å	β= 90°.
	c = 21.7267(5) Å	γ = 90°.
Volume	2215.42(8) $Å^3$	
Z	4	
Density (calculated)	1.437 Mg/m^3	
Absorption coefficient	3.653 mm^{-1}	
F(000)	984	
Crystal size	0.710 x 0.250 x 0.220 mm^3	
Theta range for data collection	4.069 to 70.227°.	

Index ranges	-9<=h<=11, -10<=k<=13, -25<=l<=25
Reflections collected	13347
Independent reflections	3808 [R(int) = 0.0426]
Completeness to theta = 67.679°	98.9 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3808 / 0 / 266
Goodness-of-fit on F^2	1.087
Final R indices [I>2sigma(I)]	R1 = 0.0364, wR2 = 0.0944
R indices (all data)	R1 = 0.0365, wR2 = 0.0946
Absolute structure parameter	0.089(6)
Extinction coefficient	0.0033(3)
Largest diff. peak and hole	0.904 and -0.872 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for cu_qsgl359b2_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	6621(1)	12883(1)	7969(1)	68(1)
S(1)	6869(1)	10605(1)	10679(1)	23(1)
O(1)	7991(3)	8556(3)	8503(1)	35(1)
O(2)	9835(3)	7407(2)	8861(1)	31(1)
O(3)	8119(3)	11123(2)	10956(1)	32(1)
O(4)	5506(3)	10798(2)	10954(1)	34(1)
N(1)	7066(3)	9132(2)	10604(1)	23(1)
N(2)	8098(3)	8146(3)	9491(1)	26(1)
C(1)	6708(4)	12146(3)	8761(2)	32(1)
C(2)	5472(4)	11678(4)	9013(2)	32(1)
C(3)	5524(4)	11182(3)	9600(2)	27(1)
C(4)	6807(4)	11183(3)	9919(2)	21(1)
C(5)	8516(4)	8606(3)	10568(2)	24(1)
C(6)	8616(4)	7637(3)	10060(2)	21(1)
C(7)	8747(4)	7981(3)	8939(2)	22(1)
C(8)	8369(4)	8493(4)	7846(2)	32(1)
C(9)	7210(6)	9287(7)	7559(2)	67(2)
C(10)	7988(4)	12144(4)	9065(2)	31(1)
C(11)	8032(4)	11656(3)	9656(2)	26(1)
C(12)	9783(6)	9110(7)	7749(2)	65(2)
C(13)	8308(12)	7206(5)	7628(3)	95(3)

C(14)	7892(4)	6440(3)	10243(2)	21(1)
C(15)	7981(4)	6080(3)	10864(2)	24(1)
C(16)	8698(4)	6882(3)	11301(2)	30(1)
C(17)	8952(4)	8064(4)	11178(2)	29(1)
C(18)	7364(4)	4960(3)	11042(2)	31(1)
C(19)	6679(4)	4223(3)	10619(2)	34(1)
C(20)	6615(4)	4575(3)	10008(2)	32(1)
C(21)	7217(4)	5681(3)	9821(2)	26(1)

Table 3. Bond lengths [Å] and angles [°] for cu_qsgl359b2_0m.

Br(1)-C(1)	1.899(4)
S(1)-O(4)	1.430(3)
S(1)-O(3)	1.435(3)
S(1)-N(1)	1.617(3)
S(1)-C(4)	1.767(3)
O(1)-C(7)	1.338(4)
O(1)-C(8)	1.471(4)
O(2)-C(7)	1.209(4)
N(1)-C(5)	1.479(5)
N(1)-H(2)	0.8800
N(2)-C(7)	1.358(5)
N(2)-C(6)	1.439(5)
N(2)-H(11)	0.8800
C(1)-C(10)	1.373(6)
C(1)-C(2)	1.381(6)
C(2)-C(3)	1.386(6)
C(2)-H(21)	0.9500
C(3)-C(4)	1.390(5)
C(3)-H(22)	0.9500
C(4)-C(11)	1.384(5)
C(5)-C(17)	1.505(5)
C(5)-C(6)	1.528(5)
C(5)-H(19)	1.0000
C(6)-C(14)	1.519(5)
C(6)-H(18)	1.0000
C(8)-C(13)	1.475(7)
C(8)-C(12)	1.504(7)

C(8)-C(9)	1.523(7)
C(9)-H(1)	0.9800
C(9)-H(6)	0.9800
C(9)-H(7)	0.9800
C(10)-C(11)	1.390(6)
C(10)-H(24)	0.9500
C(11)-H(23)	0.9500
C(12)-H(5)	0.9800
C(12)-H(3)	0.9800
C(12)-H(4)	0.9800
C(13)-H(8)	0.9800
C(13)-H(10)	0.9800
C(13)-H(9)	0.9800
C(14)-C(21)	1.385(5)
C(14)-C(15)	1.407(5)
C(15)-C(18)	1.401(5)
C(15)-C(16)	1.455(6)
C(16)-C(17)	1.332(6)
C(16)-H(13)	0.9500
C(17)-H(12)	0.9500
C(18)-C(19)	1.377(6)
C(18)-H(17)	0.9500
C(19)-C(20)	1.382(6)
C(19)-H(16)	0.9500
C(20)-C(21)	1.387(5)
C(20)-H(15)	0.9500
C(21)-H(14)	0.9500
O(4)-S(1)-O(3)	120.09(17)

O(4)-S(1)-N(1)	106.85(16)
O(3)-S(1)-N(1)	109.60(16)
O(4)-S(1)-C(4)	108.05(18)
O(3)-S(1)-C(4)	106.24(17)
N(1)-S(1)-C(4)	105.05(15)
C(7)-O(1)-C(8)	122.4(3)
C(5)-N(1)-S(1)	119.5(2)
C(5)-N(1)-H(2)	120.3
S(1)-N(1)-H(2)	120.3
C(7)-N(2)-C(6)	123.8(3)
C(7)-N(2)-H(11)	118.1
C(6)-N(2)-H(11)	118.1
C(10)-C(1)-C(2)	123.1(3)
C(10)-C(1)-Br(1)	118.3(3)
C(2)-C(1)-Br(1)	118.5(3)
C(1)-C(2)-C(3)	118.6(4)
C(1)-C(2)-H(21)	120.7
C(3)-C(2)-H(21)	120.7
C(2)-C(3)-C(4)	119.2(3)
C(2)-C(3)-H(22)	120.4
C(4)-C(3)-H(22)	120.4
C(11)-C(4)-C(3)	121.1(3)
C(11)-C(4)-S(1)	119.3(3)
C(3)-C(4)-S(1)	119.6(3)
N(1)-C(5)-C(17)	110.9(3)
N(1)-C(5)-C(6)	111.1(3)
C(17)-C(5)-C(6)	110.5(3)
N(1)-C(5)-H(19)	108.1
C(17)-C(5)-H(19)	108.1

C(6)-C(5)-H(19)	108.1
N(2)-C(6)-C(14)	113.6(3)
N(2)-C(6)-C(5)	109.6(3)
C(14)-C(6)-C(5)	111.9(3)
N(2)-C(6)-H(18)	107.1
C(14)-C(6)-H(18)	107.1
C(5)-C(6)-H(18)	107.1
O(2)-C(7)-O(1)	126.2(3)
O(2)-C(7)-N(2)	124.8(3)
O(1)-C(7)-N(2)	109.0(3)
O(1)-C(8)-C(13)	110.2(4)
O(1)-C(8)-C(12)	109.2(4)
C(13)-C(8)-C(12)	114.2(6)
O(1)-C(8)-C(9)	101.5(3)
C(13)-C(8)-C(9)	112.1(5)
C(12)-C(8)-C(9)	108.9(4)
C(8)-C(9)-H(1)	109.5
C(8)-C(9)-H(6)	109.5
H(1)-C(9)-H(6)	109.5
C(8)-C(9)-H(7)	109.5
H(1)-C(9)-H(7)	109.5
H(6)-C(9)-H(7)	109.5
C(1)-C(10)-C(11)	118.1(4)
C(1)-C(10)-H(24)	121.0
C(11)-C(10)-H(24)	121.0
C(4)-C(11)-C(10)	119.9(3)
C(4)-C(11)-H(23)	120.1
C(10)-C(11)-H(23)	120.1
C(8)-C(12)-H(5)	109.5

C(8)-C(12)-H(3)	109.5
H(5)-C(12)-H(3)	109.5
C(8)-C(12)-H(4)	109.5
H(5)-C(12)-H(4)	109.5
H(3)-C(12)-H(4)	109.5
C(8)-C(13)-H(8)	109.5
C(8)-C(13)-H(10)	109.5
H(8)-C(13)-H(10)	109.5
C(8)-C(13)-H(9)	109.5
H(8)-C(13)-H(9)	109.5
H(10)-C(13)-H(9)	109.5
C(21)-C(14)-C(15)	119.8(3)
C(21)-C(14)-C(6)	122.7(3)
C(15)-C(14)-C(6)	117.4(3)
C(18)-C(15)-C(14)	118.7(4)
C(18)-C(15)-C(16)	122.0(3)
C(14)-C(15)-C(16)	119.3(3)
C(17)-C(16)-C(15)	121.8(3)
C(17)-C(16)-H(13)	119.1
C(15)-C(16)-H(13)	119.1
C(16)-C(17)-C(5)	120.3(3)
C(16)-C(17)-H(12)	119.8
C(5)-C(17)-H(12)	119.8
C(19)-C(18)-C(15)	120.9(4)
C(19)-C(18)-H(17)	119.6
C(15)-C(18)-H(17)	119.6
C(18)-C(19)-C(20)	120.0(3)
C(18)-C(19)-H(16)	120.0
C(20)-C(19)-H(16)	120.0

C(19)-C(20)-C(21)	120.2(4)
C(19)-C(20)-H(15)	119.9
C(21)-C(20)-H(15)	119.9
C(14)-C(21)-C(20)	120.4(4)
C(14)-C(21)-H(14)	119.8
C(20)-C(21)-H(14)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cu_qsgl359b2_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	66(1)	97(1)	40(1)	44(1)	-16(1)	-22(1)
S(1)	37(1)	14(1)	18(1)	0(1)	1(1)	2(1)
O(1)	39(2)	50(2)	16(1)	6(1)	3(1)	15(1)
O(2)	36(1)	31(1)	26(1)	3(1)	6(1)	11(1)
O(3)	52(2)	20(1)	23(1)	0(1)	-11(1)	-4(1)
O(4)	49(2)	25(1)	28(1)	4(1)	12(1)	9(1)
N(1)	29(2)	16(1)	25(1)	2(1)	2(1)	-2(1)
N(2)	30(2)	27(1)	20(1)	7(1)	3(1)	10(1)
C(1)	39(2)	31(2)	28(2)	11(2)	-4(2)	-2(2)
C(2)	33(2)	31(2)	30(2)	4(2)	-10(2)	-3(2)
C(3)	29(2)	22(2)	30(2)	2(2)	0(2)	-6(1)
C(4)	33(2)	12(1)	17(2)	0(1)	0(2)	2(1)
C(5)	27(2)	16(1)	27(2)	0(1)	-3(2)	-1(1)
C(6)	27(2)	17(1)	19(2)	4(1)	-1(1)	4(1)
C(7)	31(2)	15(1)	20(2)	3(1)	1(1)	-1(1)
C(8)	37(2)	43(2)	16(2)	-1(2)	3(2)	2(2)
C(9)	60(3)	116(5)	25(2)	12(3)	0(2)	28(4)
C(10)	33(2)	34(2)	27(2)	6(2)	1(2)	-4(2)
C(11)	25(2)	26(2)	26(2)	5(2)	-3(2)	0(2)
C(12)	52(3)	110(5)	32(2)	30(3)	-1(2)	-20(3)
C(13)	194(9)	49(3)	42(3)	-16(3)	-27(5)	-14(5)
C(14)	25(2)	17(1)	22(2)	0(1)	2(1)	5(1)
C(15)	31(2)	19(2)	23(2)	5(1)	6(2)	8(1)
C(16)	43(2)	28(2)	18(2)	4(2)	-1(2)	10(2)

C(17)	34(2)	29(2)	25(2)	-7(2)	-8(2)	5(2)
C(18)	38(2)	21(2)	34(2)	10(2)	14(2)	11(2)
C(19)	30(2)	13(1)	60(3)	8(2)	11(2)	2(1)
C(20)	28(2)	20(2)	49(2)	-6(2)	2(2)	-1(1)
C(21)	27(2)	22(2)	29(2)	-2(2)	0(2)	7(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for cu_qsgl359b2_0m.

	x	y	z	U(eq)
H(2)	6317	8647	10584	28
H(11)	7316	8590	9502	31
H(21)	4605	11696	8788	38
H(22)	4693	10844	9783	32
H(19)	9191	9289	10467	28
H(18)	9648	7449	10000	26
H(1)	6275	8984	7689	100
H(6)	7281	9244	7109	100
H(7)	7327	10143	7693	100
H(24)	8821	12466	8876	38
H(23)	8900	11648	9880	31
H(5)	9821	9874	7990	97
H(3)	9904	9302	7312	97
H(4)	10547	8557	7883	97
H(8)	9155	6762	7770	143
H(10)	8274	7196	7178	143
H(9)	7454	6806	7793	143
H(13)	8994	6553	11686	35
H(12)	9411	8567	11475	35
H(17)	7419	4705	11459	37
H(16)	6251	3472	10747	41

H(15)	6158	4058	9716	39
H(14)	7165	5920	9401	31

Table 6.Torsion angles [°] for cu_qsgl359b2_0m.

O(4)-S(1)-N(1)-C(5)	-156.0(3)
O(3)-S(1)-N(1)-C(5)	-24.4(3)
C(4)-S(1)-N(1)-C(5)	89.4(3)
C(10)-C(1)-C(2)-C(3)	0.0(6)
Br(1)-C(1)-C(2)-C(3)	-177.6(3)
C(1)-C(2)-C(3)-C(4)	0.7(6)
C(2)-C(3)-C(4)-C(11)	-0.8(5)
C(2)-C(3)-C(4)-S(1)	177.6(3)
O(4)-S(1)-C(4)-C(11)	149.2(3)
O(3)-S(1)-C(4)-C(11)	19.1(3)
N(1)-S(1)-C(4)-C(11)	-97.0(3)
O(4)-S(1)-C(4)-C(3)	-29.3(3)
O(3)-S(1)-C(4)-C(3)	-159.4(3)
N(1)-S(1)-C(4)-C(3)	84.5(3)
S(1)-N(1)-C(5)-C(17)	100.9(3)
S(1)-N(1)-C(5)-C(6)	-135.9(3)
C(7)-N(2)-C(6)-C(14)	-97.2(4)
C(7)-N(2)-C(6)-C(5)	136.8(3)
N(1)-C(5)-C(6)-N(2)	52.4(4)
C(17)-C(5)-C(6)-N(2)	175.9(3)
N(1)-C(5)-C(6)-C(14)	-74.5(4)
C(17)-C(5)-C(6)-C(14)	48.9(4)
C(8)-O(1)-C(7)-O(2)	3.8(6)
C(8)-O(1)-C(7)-N(2)	-176.4(3)
C(6)-N(2)-C(7)-O(2)	-1.0(6)
C(6)-N(2)-C(7)-O(1)	179.2(3)
C(7)-O(1)-C(8)-C(13)	61.4(6)

C(7)-O(1)-C(8)-C(12)	-64.8(5)
C(7)-O(1)-C(8)-C(9)	-179.7(4)
C(2)-C(1)-C(10)-C(11)	-0.7(6)
Br(1)-C(1)-C(10)-C(11)	177.0(3)
C(3)-C(4)-C(11)-C(10)	0.2(5)
S(1)-C(4)-C(11)-C(10)	-178.3(3)
C(1)-C(10)-C(11)-C(4)	0.6(6)
N(2)-C(6)-C(14)-C(21)	22.6(5)
C(5)-C(6)-C(14)-C(21)	147.3(3)
N(2)-C(6)-C(14)-C(15)	-160.4(3)
C(5)-C(6)-C(14)-C(15)	-35.6(4)
C(21)-C(14)-C(15)-C(18)	-0.8(5)
C(6)-C(14)-C(15)-C(18)	-177.9(3)
C(21)-C(14)-C(15)-C(16)	179.7(3)
C(6)-C(14)-C(15)-C(16)	2.5(5)
C(18)-C(15)-C(16)-C(17)	-162.1(4)
C(14)-C(15)-C(16)-C(17)	17.4(6)
C(15)-C(16)-C(17)-C(5)	-0.9(6)
N(1)-C(5)-C(17)-C(16)	91.1(4)
C(6)-C(5)-C(17)-C(16)	-32.5(5)
C(14)-C(15)-C(18)-C(19)	-0.1(5)
C(16)-C(15)-C(18)-C(19)	179.4(3)
C(15)-C(18)-C(19)-C(20)	1.1(6)
C(18)-C(19)-C(20)-C(21)	-1.2(6)
C(15)-C(14)-C(21)-C(20)	0.7(5)
C(6)-C(14)-C(21)-C(20)	177.7(3)
C(19)-C(20)-C(21)-C(14)	0.3(6)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cu_qsgl359b2_0m [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
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C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7

C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
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N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7
C(12)-H(4)...O(2)	0.98	2.55	3.042(6)	110.8
C(13)-H(8)...O(2)	0.98	2.55	3.047(7)	111.1
N(1)-H(2)...O(2)#3	0.88	2.17	2.922(4)	143.3
C(9)-H(6)...O(3)#2	0.98	2.57	3.525(6)	166.1
C(9)-H(7)...Br(1)	0.98	3.10	4.040(7)	160.3
C(10)-H(24)...O(4)#1	0.95	2.49	3.253(5)	137.7

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y+5/2,-z+2 #2 -x+3/2,-y+2,z-1/2 #3 x-1/2,-y+3/2,-z+2