Copper-Catalyzed Enantioselective Ring-Opening of Cyclic Diaryliodoniums and *O*-Alkylhydroxylamines

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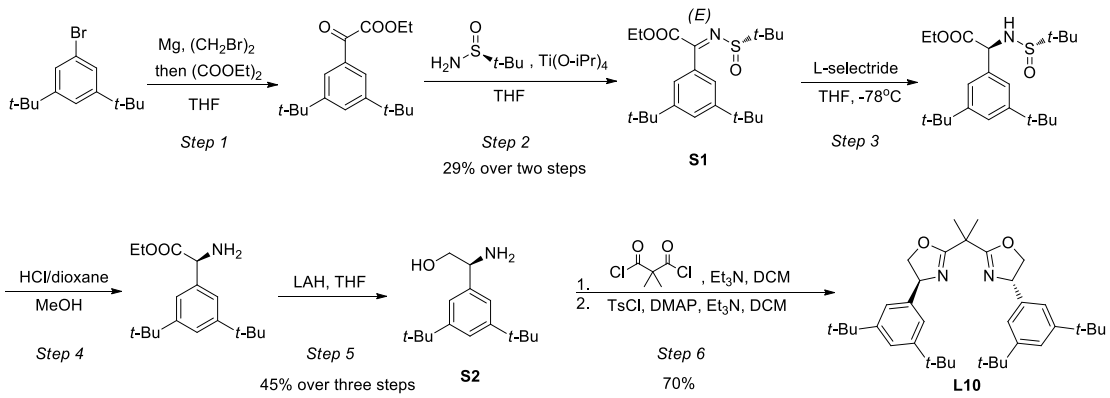
General Information

Nuclear magnetic resonances were recorded on Bruker-400 MHz instruments. Reference values for residual solvents were taken as δ = 7.26 ppm ($CDCl_3$), 2.50 ppm (DMSO-d_6) for ^{1}H NMR; δ = 77.00 ppm ($CDCl_3$), 39.52 ppm (DMSO-d_6) for ^{13}C NMR. High resolution mass spectral analysis (HRMS) was performed on Waters XEVO G2 Q-TOF (Waters Corporation). All reactions were performed under an inert atmosphere of dry nitrogen in flame-dried glassware, unless otherwise stated. Tetrahydrofuran were distilled over sodium in the presence of benzophenone under an atmosphere of nitrogen. Toluene and dichloroethane were distilled over calcium hydride under an atmosphere of nitrogen. The cyclic diaryliodoniums with triflate anion were synthesized according to the reported literature.[1]

Synthesis of L10



Step 1: Under nitrogen atmosphere, to a 200 mL Schleck flask containing magnesium (1.89 g, 78 mmol, 1.3 equiv) was added anhydrous THF (30 mL) followed by $(CH_2Br)_2$ (0.10 mL). To this mixture, a solution of 1-bromo-3,5-di-*tert*-butylbenzene (16.15 g, 60 mmol, 1.0 equiv) in THF (30 mL) was added dropwise under stirring at room temperature. Then the mixture was stirring at 65 ℃ for 1 h. Diethyl

oxalate (16.3 mL, 120 mmol) was dissolved in THF (30 mL) in a 250 mL round-bottom flask under nitrogen. The solution was cooled to –78 ℃, then the above Grignard reagent was added to the reaction mixture in 10 min. After stirring for 1 h at –78 ℃, the reaction was warmed up to room temperature and stirred for another 20 min. After the reaction completed, saturated $NaHCO_3$ solution (50 mL) was added. After stirring for 5 min, the mixture was filtered through a plug of Celite with ethyl acetate. The filtrate was extracted with ethyl acetate, washed with brine (50 mL x2), dried over anhydrous Na_2SO_4 and concentrated, then $(CO_2Et)_2$ was removed by distillation at 150 ℃, 0.01 Mpa to afford the crude product, which was used in next step without further purification.

Step 2: The crude product from Step 1 (11.32 g, about 39 mmol) and (*S*)-*tert*-butylsulfinamide (4.73 g, 39 mmol) were dissolved in anhydrous THF (60 mL), then $Ti(O\text{-}iPr)_4$ (17.3 mL, 58.5 mmol, 1.5 equiv) was added. After stirring at 65 ℃ for 12 h under nitrogen, the solution was cooled to room temperature, diluted with ethyl acetate (50 mL), quenched by aqueous NaOH solution (10% w/w, 30 mL) and the mixture was stirring for another 10 min before being filtered through a plug of Celite with ethyl acetate. The filtrate was then washed with brine (80 mL x2), dried over anhydrous sodium sulfate, concentrated and purified by flash column chromatography (silica gel, 5% ethyl acetate in hexanes) to afford the pure (*S*)-*N*-*tert*-butanesulfinyl ketimine ester **S1** (6.9 g, 17.53 mmol, 29% over two steps).

Step 3: In accordance with the literature procedure,[2] the (*S*)-*N*-*tert*-butanesulfinyl ketimine ester **S1** (17.53 mmol, 1.0 equiv) was dissolved in THF (50 mL) in a 200 mL round-bottom flask under nitrogen. The solution was cooled to –78 ℃ and stirred for 5 min, then L-Selectride in THF (19.3 mL, 1.0 M, 19.3 mmol, 1.1 equiv) was slowly added to the reaction mixture via a syringe at –78 ℃ over 40 min. The reaction was stirred at –78 ℃ for another 5 h, quenched with saturated NH_4Cl solution (30 mL) at –78 ℃, and warmed up to room temperature. The mixture was transferred to a separatory funnel containing brine (50 mL) and ethyl acetate (50 mL). The organic layer was separated and the aqueous layer was extracted with ethyl acetate (50 mL). The combined organic layers were washed with brine (50 mL x2), dried over sodium sulfate, and concentrated. The crude product was used in the next step without further purification.

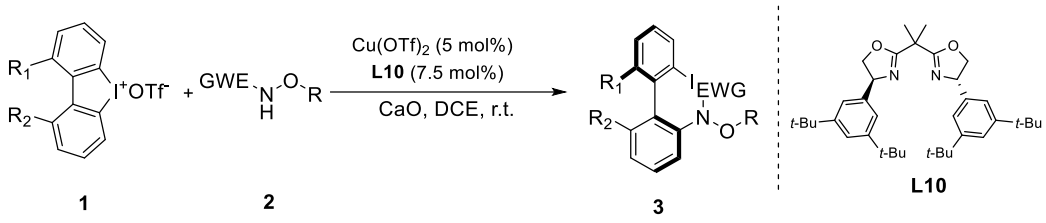
Step 4: The crude product from Step 3 was dissolved in methanol (40 mL) in a 200 mL round-bottom flask in open air. A solution of hydrogen chloride in dioxane (17 mL, 4.0 M, 68 mmol) was then added to the reaction mixture at 0 ℃ over 2 min. The reaction flask was capped with a rubber septum and stirred at room temperature for 1 h. Then aqueous NaOH solution (10% w/w, 30 mL) was added slowly and the solution was extracted with ethyl acetate (80 mL), washed with brine (80 mL x2), dried over sodium sulfate, and concentrated to afford the crude product. The ee value (95% ee) was determined after acetylation of the amino group in accordance with the literature.[3]

Step 5: The crude product from Step 4 was dissolved in anhydrous THF (50 mL) in a 200 mL round-bottom flask under nitrogen and the solution was cooled to 0 ℃. Lithium aluminum hydride (2

g, 52.7 mmol) was then added portionwise. The reaction was stirred at 0 ℃ overnight, and carefully quenched by sequential addition of 2 mL H_2O – 2 mL 15 wt% NaOH solution – 2 mL H_2O. The mixture was filtered through Celite with DCM, the filtrate was concentrated and purified by flash column chromatography on silica gel (DCM/ MeOH = 20/1) to afford the pure amino alcohol **S2** as white solid (1.98 g, 7.94 mmol, 29% over three steps).

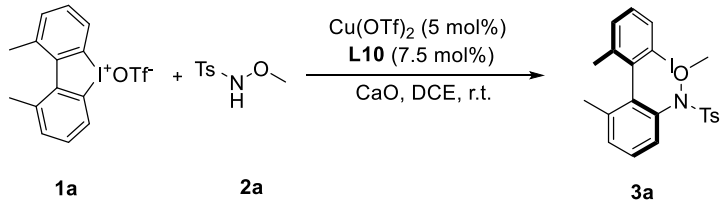
Step 6: To an ice-cooled 100 mL round-bottom flask containing **S2** (7.94 mmol, 2.0 equiv), DCM (30 mL) and triethylamine (2.2 mL, 15.88 mmol, 4.0 equiv) was added dimethylmalonyl dichloride (0.525 mL, 3.97 mmol, 1.0 equiv) dropwise. After stirring at room temperature for 2 h, tosyl chloride (1.81 g, 9.53 mmol, 2.4 equiv), DMAP (137 mg, 0.794 mmol, 0.2 equiv) and triethylamine (2.76 mL, 19.85 mmol, 5.0 equiv) were then added. The reaction was stirred for 2 d at room temperature before removing the solvent by evaporator, and the residue was purified by flash column chromatography on silica gel (hexanes/ ethyl acetate = 10/1) to afford **L10** as a white solid (1.55 g, 2.77 mmol, 70%). $[\alpha]_D^{20}$ = -48 (c = 1.21, $CHCl_3$). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 1.6 Hz, 2H), 7.10 (d, *J* = 1.6 Hz, 4H), 5.17 (dd, *J* = 10.0, 6.8 Hz, 2H), 4.64 (dd, *J* = 10.0, 8.4 Hz, 2H), 4.25 (dd, *J* = 8.4, 6.8 Hz, 2H), 1.69 (s, 6H), 1.29 (s, 36H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.00, 150.98, 141.69, 121.64, 121.00, 75.89, 70.10, 38.75, 34.83, 31.41, 24.61. HRMS (ESI): calcd for $C_{37}H_{55}N_2O_2$ $(M+H)^+$ 559.4264, found 559.4265.

General Procedure for the Synthesis of 3 (Procedure A)



To a Schlenk tube containing **1** (1.0 equiv), **2** (1.2 equiv), $Cu(OTf)_2$ (5.0 mol%), **L10** (7.5 mol%) and CaO (2.0 equiv) was added DCE (0.05 M) under nitrogen at room temperature. After being stirred for 3-12 h (monitored by TLC) at room temperature, the solvent was removed and the residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate) to afford the desired product **3.**

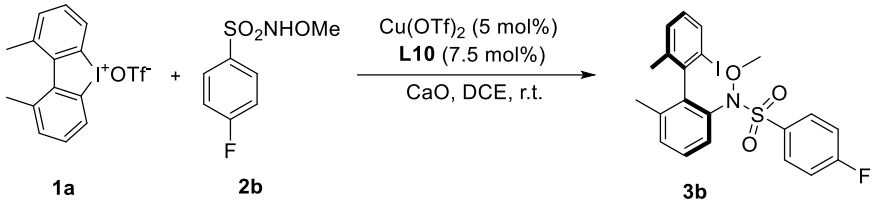
Compound **3a** was prepared following the **Procedure A**



The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2a** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at

room temperature for 3 h afforded **3a** (50.2 mg, 99%, 99% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +105 (c = 0.93, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.30 (dd, *J* = 12.2, 7.8 Hz, 4H), 7.15 (t, *J* = 7.8 Hz, 1H), 7.00 (t, *J* = 7.8 Hz, 1H), 6.63 (d, *J* = 8.4 Hz, 1H), 3.76 (s, 3H), 2.46 (s, 3H), 2.25 (s, 3H), 2.02 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.70, 143.16, 141.67, 139.84, 139.42, 137.69, 136.06, 132.10, 130.88, 130.09, 129.84, 129.16, 129.11, 127.95, 123.69, 101.12, 65.16, 21.78, 21.70, 20.16. HRMS (ESI): calcd for $C_{22}H_{22}INO_3SNa$ $(M+Na)^+$ 530.0263, found 530.0269.

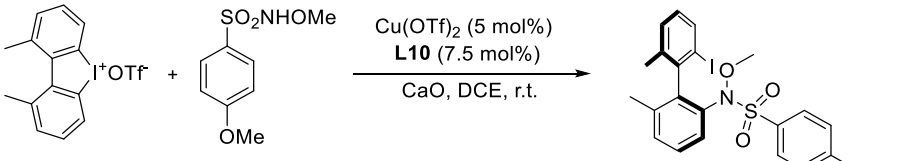
Compound **3b** was prepared following the **Procedure A**



1a **2b** **3b**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2b** (24.6 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3b** (51.7 mg, >99%, 99% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +73 (c = 0.99, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.8 Hz, 1H), 7.75 – 7.70 (m, 2H), 7.30 (t, *J* = 7.2 Hz, 2H), 7.16 (q, *J* = 8.0, 7.5 Hz, 3H), 7.00 (t, *J* = 7.8 Hz, 1H), 6.56 (d, *J* = 8.2 Hz, 1H), 3.81 (s, 3H), 2.23 (s, 3H), 2.02 (s, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -103.30. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.12, 164.57, 143.07, 141.43, 139.73, 139.15, 137.91, 136.10, 132.84 (d, *J* = 9.5 Hz), 131.03, 129.84, 129.22, 127.96, 123.39, 115.78 (d, *J* = 22.5 Hz), 101.04, 65.30, 21.76, 20.17. HRMS (ESI): calcd for $C_{21}H_{19}FINO_3SNa$ $(M+Na)^+$ 534.0012, found 534.0021.

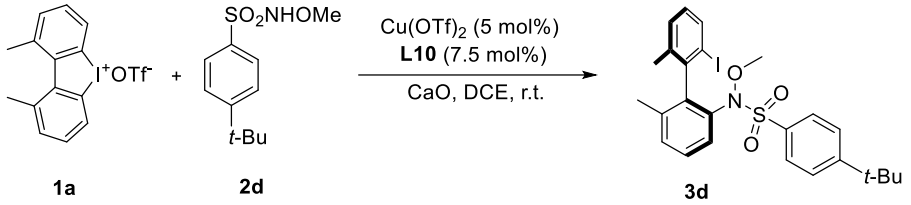
Compound **3c** was prepared following the **Procedure A**



1a **2c** **3c**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2c** (26.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3c** (54.1 mg, >99%, 99% ee). Rf = 0.4 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +106 (c = 0.98, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.68 – 7.62 (m, 2H), 7.30 (dd, *J* = 11.8, 7.6 Hz, 2H), 7.16 (t, *J* = 7.8 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.97 – 6.93 (m, 2H), 6.63 (d, *J* = 8.0 Hz, 1H), 3.89 (s, 3H), 3.77 (s, 3H), 2.25 (s, 3H), 2.02 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.77, 143.09, 141.67, 139.79, 139.57, 137.63, 136.03, 132.22, 130.81, 129.80, 129.12, 127.90, 126.39, 123.61, 113.64, 101.12, 65.16, 55.62, 21.76, 20.14. HRMS (ESI): calcd for $C_{22}H_{22}INO_4SNa$ $(M+Na)^+$ 546.0212, found 546.0212.

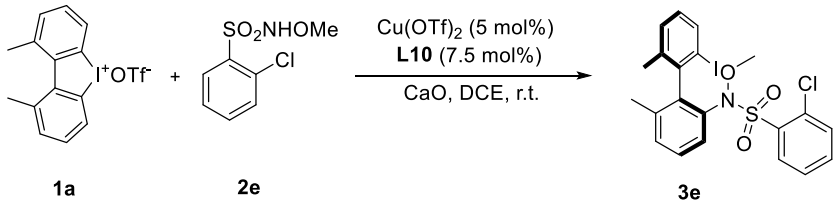
Compound **3d** was prepared following the **Procedure A**



1a **2d** **3d**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2d** (29.2 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3d** (54.7 mg, 99%, 99% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +82 (c = 0.78, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.67 – 7.63 (m, 2H), 7.52 – 7.47 (m, 2H), 7.30 (dd, *J* = 12.4, 7.6 Hz, 2H), 7.14 (t, *J* = 7.8 Hz, 1H), 7.00 (t, *J* = 7.8 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 3.75 (s, 3H), 2.25 (s, 3H), 2.02 (s, 3H), 1.36 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.71, 143.22, 141.69, 139.80, 139.38, 137.71, 136.07, 132.07, 130.91, 129.92, 129.85, 129.18, 127.93, 125.45, 123.73, 101.16, 65.14, 35.26, 31.07, 21.78, 20.16. HRMS (ESI): calcd for $C_{25}H_{28}INO_3SNa$ $(M+Na)^+$ 572.0732, found 572.0736.

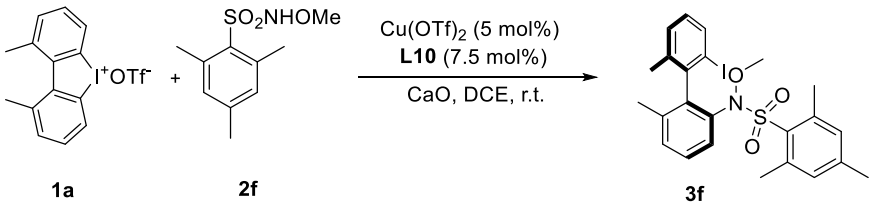
Compound **3e** was prepared following the **Procedure A**



1a **2e** **3e**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2e** (26.6 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 12 h afforded **3e** (45.7 mg, 86%, 88% ee). Rf = 0.4 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +37.6 (c = 0.85, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.76 (d, *J* = 7.6 Hz, 1H), 7.53 (ddd, *J* = 8.0, 7.2, 1.6 Hz, 1H), 7.47 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.41 – 7.33 (m, 2H), 7.32 – 7.27 (m, 2H), 7.26 – 7.22 (m, 1H), 6.99 (t, *J* = 7.8 Hz, 1H), 3.55 (s, 3H), 2.23 (s, 3H), 2.01 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.12, 141.74, 140.35, 138.31, 137.80, 136.07, 134.76, 134.65, 134.25, 133.63, 132.09, 131.32, 129.99, 129.16, 128.28, 126.62, 125.02, 101.12, 64.52, 21.86, 20.21. HRMS (ESI): calcd for $C_{21}H_{19}ClINO_3SNa$ $(M+Na)^+$ 549.9717, found 549.9711.

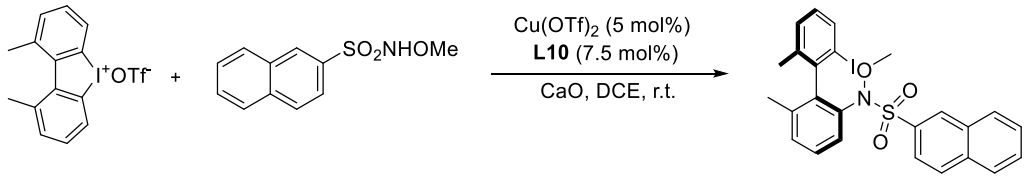
Compound **3f** was prepared following the **Procedure A**



1a **2f** **3f**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2f** (27.5 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3f** (50.1 mg, 94%, 96% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +22.7 (c = 0.74, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.68 (m, 2H), 7.42 – 7.33 (m, 2H), 7.26 (d, *J* = 8.0 Hz, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 6.89 (s, 2H), 3.23 (s, 3H), 2.31 (s, 6H), 2.28 (s, 3H), 2.22 (s, 3H), 2.00 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.50, 142.73, 142.51, 142.15, 140.56, 138.69, 137.23, 136.06, 131.69, 131.04, 130.97, 130.17, 129.06, 128.32, 126.10, 101.02, 63.80, 22.55, 21.91, 21.03, 20.21. HRMS (ESI): calcd for $C_{24}H_{26}INO_3SNa$ $(M+Na)^+$ 558.0576, found 558.0583.

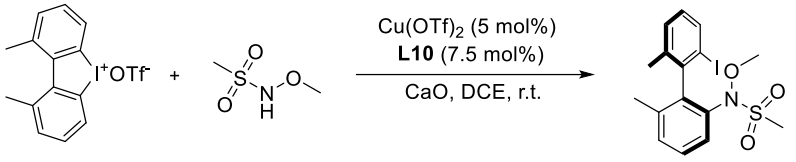
Compound **3g** was prepared following the **Procedure A**



1a **2g** **3g**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2g** (28.5 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3g** (mg, 93%, 94% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +83.6 (c = 1.07, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.97 – 7.88 (m, 3H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.71 – 7.59 (m, 3H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.8 Hz, 1H), 7.03 (t, *J* = 7.8 Hz, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 3.75 (s, 3H), 2.30 (s, 3H), 2.04 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.19, 141.68, 139.85, 139.13, 137.77, 136.10, 135.28, 132.39, 131.75, 131.01, 129.86, 129.44, 129.20, 128.43, 128.03, 127.87, 127.40, 124.79, 123.90, 101.16, 65.15, 21.82, 20.15. HRMS (ESI): calcd for $C_{25}H_{22}INO_3SNa$ $(M+Na)^+$ 566.0263, found 566.0270.

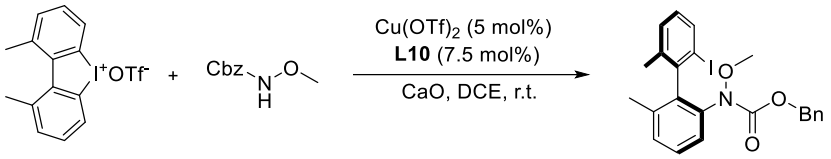
Compound **3h** was prepared following the **Procedure A**



1a **2h** **3h**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2h** (15.0 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 3 h afforded **3h** (37.9 mg, 88%, 96% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +49.5 (c = 1.10, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.0 Hz, 1H), 7.41 (dd, *J* = 7.2, 5.6 Hz, 2H), 7.37 – 7.34 (m, 1H), 7.27 – 7.24 (m, 1H), 6.97 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 3.00 (s, 3H), 2.12 (s, 3H), 2.01 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.33, 141.47, 139.57, 138.51, 138.36, 136.12, 131.17, 129.84, 129.25, 128.41, 123.45, 100.97, 64.97, 34.68, 21.62, 20.14. HRMS (ESI): calcd for $C_{16}H_{18}INO_3SNa$ $(M+Na)^+$ 453.9950, found 453.9955.

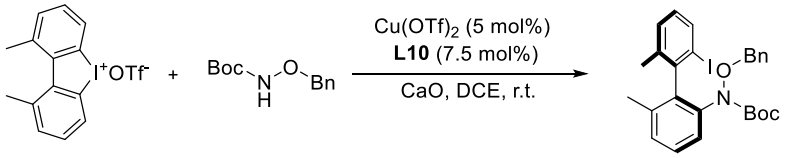
Compound **3i** was prepared following the **Procedure A**



1a **2i** **3i**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2i** (21.7 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3i** (42.1 mg, 86%, 93% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +0.7 (c = 0.89, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 10:90, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.5 Hz, 1H), 7.35 – 7.26 (m, 7H), 7.19 (d, *J* = 7.2 Hz, 1H), 7.15 (t, *J* = 4.6 Hz, 1H), 6.93 (t, *J* = 7.6 Hz, 1H), 5.19 (d, *J* = 12.4 Hz, 1H) , 5.16 (d, *J* = 12.4 Hz, 1H), 3.48 (s, 3H), 2.00 (s, 3H), 1.94 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 155.20, 142.22, 141.98, 138.25, 137.92, 136.81, 136.39, 135.99, 130.69, 129.68, 129.10, 128.37, 128.02, 127.97, 127.91, 126.67, 101.59, 67.52, 61.85, 21.15, 19.79. HRMS (ESI): calcd for $C_{23}H_{22}INO_3Na$ $(M+Na)^+$ 510.0542, found 510.0535.

Compound **3j** was prepared following the **Procedure A**

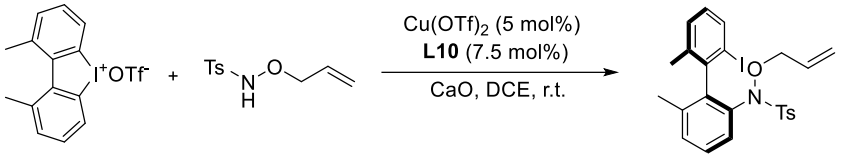


1a **2j** **3j**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2j** (26.8 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 12 h afforded **3j** (44.1 mg, 83%, 95% ee). Rf = 0.6 (PE/EA = 20:1). $[\alpha]_D^{20}$ = +9.5 (c = 1.01, $CHCl_3$). HPLC conditions: Chiralpak OD-H, isopropanol/hexane = 0.5:99.5, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.39 – 7.26 (m, 7H), 7.23 (d, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 6.95 (t, *J* = 8.0 Hz, 1H), 4.54 (d, *J* = 9.4 Hz, 1H), 4.50 (d, *J* = 9.4 Hz, 1H), 2.04 (s, 3H), 2.01 (s, 3H), 1.43 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 155.18, 142.41, 142.34, 138.66, 138.51, 137.59, 136.40, 135.48, 130.24, 129.68, 129.50, 129.06, 128.10, 128.05, 127.97, 126.84, 101.80, 81.63, 75.96, 28.22, 21.34, 19.91. HRMS (ESI): calcd for $C_{26}H_{28}INO_3Na$ $(M+Na)^+$ 552.1012, found 552.1016.

A reaction at 1 mmol scale: To a Schlenk tube containing **1a** (0.456 g, 1.0 mmol, 1.0 equiv), **2j** (0.268 g, 1.2 mmol, 1.2 equiv), $Cu(OTf)_2$ (18 mg, 5.0 mol%), **L10** (42 mg, 7.5 mol%) and CaO (0.112 g, 2.0 mmol, 2.0 equiv) was added DCE (10.0 mL, 0.10 M of **1a**) under nitrogen at room temperature and stirred at the same temperature. After complete consumption of starting material (12 h at room temperature), the solvent was removed by evaporation and the residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate = 40:1) to afford the desired product **3j** (0.481 g, 91%, 95% ee).

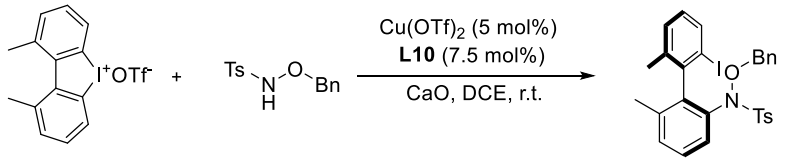
Compound **3k** was prepared following the **Procedure A**



1a **2k** **3k**

The reaction of **1a** (91.2 mg, 0.20 mmol, 1.0 equiv), **2k** (54.5 mg, 0.24 mmol, 1.2 equiv), $Cu(OTf)_2$ (3.6 mg, 5.0 mol%), **L10** (8.4 mg, 7.5 mol%) and CaO (22.4 mg, 0.40 mmol, 2.0 equiv) in DCE (4.0 mL) at room temperature for 6 h afforded **3k** (100.5 mg, 94%, 75% ee). Rf = 0.4 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +23.3 (c = 0.92, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 – 7.73 (m, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.34 – 7.26 (m, 4H), 7.13 (t, *J* = 8.0 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.60 (d, *J* = 7.6 Hz, 1H), 5.84 (ddt, *J* = 16.8, 10.4, 6.2 Hz, 1H), 5.29 – 5.13 (m, 2H), 4.70 (ddt, *J* = 11.0, 6.0, 1.2 Hz, 1H), 4.39 (ddt, *J* = 10.9, 6.4, 1.2 Hz, 1H), 2.46 (s, 3H), 2.26 (s, 3H), 2.01 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.71, 143.06, 141.59, 139.93, 139.60, 137.60, 136.04, 132.24, 131.68, 130.82, 130.27, 129.87, 129.17, 129.06, 127.91, 123.79, 119.35, 101.06, 78.44, 21.81, 21.71, 20.17. HRMS (ESI): calcd for $C_{24}H_{24}INO_3SNa$ $(M+Na)^+$ 556.0419, found 556.0419.

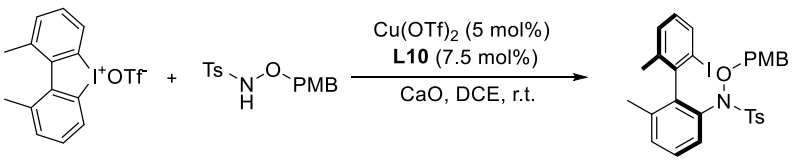
Compound **3l** was prepared following the **Procedure A**



1a **2l** **3l**

The reaction of **1a** (45.6 mg, 0.10 mmol, 1.0 equiv), **2l** (33.3 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3l** (61.8 mg, >99%, 98% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = -3.3 (c = 1.06, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.38 – 7.28 (m, 7H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.12 (t, *J* = 7.8 Hz, 1H), 7.03 (t, *J* = 7.8 Hz, 1H), 6.58 (d, *J* = 8.0 Hz, 1H), 5.34 (d, *J* = 9.2 Hz, 1H), 4.95 (d, *J* = 9.6 Hz, 1H), 2.43 (s, 3H), 2.31 (s, 3H), 2.05 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.63, 142.94, 141.59, 140.10, 139.72, 137.59, 136.10, 135.46, 131.44, 130.76, 130.31, 129.90, 129.19, 129.16, 129.02, 128.25, 127.84, 123.92, 101.12, 79.19, 21.88, 21.65, 20.20. HRMS (ESI): calcd for $C_{28}H_{26}INO_3SNa$ $(M+Na)^+$ 606.0576, found 606.0579.

Compound **3m** was prepared following the **Procedure A**

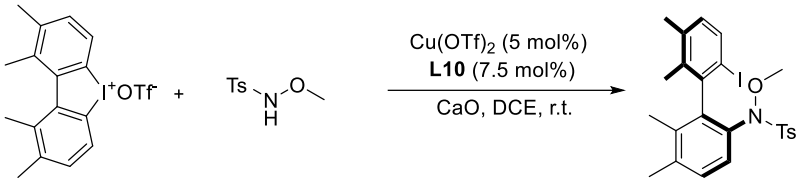


1a **2m** **3m**

The reaction of **1a** (91.2 mg, 0.20 mmol, 1.0 equiv), **2m** (73.8 mg, 0.24 mmol, 1.2 equiv), $Cu(OTf)_2$ (3.6 mg, 5.0 mol%), **L10** (8.4 mg, 7.5 mol%) and CaO (22.4 mg, 0.40 mmol, 2.0 equiv) in DCE (4.0 mL) at room temperature for 12 h afforded **3m** (107 mg, 87%, 93% ee). Rf = 0.4 (PE/EA =

10:1). $[\alpha]_D^{20}$ = -31.4 (c = 0.51, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.6 Hz, 1H), 7.60 – 7.53 (m, 2H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 6.4 Hz, 3H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.10 (t, *J* = 7.8 Hz, 1H), 7.02 (t, *J* = 7.8 Hz, 1H), 6.87 – 6.81 (m, 2H), 6.53 (d, *J* = 8.0 Hz, 1H), 5.25 (d, *J* = 9.2 Hz, 1H), 4.86 (d, *J* = 9.2 Hz, 1H), 3.80 (s, 3H), 2.42 (s, 3H), 2.29 (s, 3H), 2.03 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.70, 144.57, 143.00, 141.65, 140.12, 139.81, 137.61, 136.13, 131.58, 131.00, 130.73, 130.35, 129.93, 129.20, 129.03, 127.84, 127.67, 124.01, 113.67, 101.20, 78.87, 55.23, 21.91, 21.67, 20.22. HRMS (ESI): calcd for $C_{29}H_{28}INO_4SNa$ $(M+Na)^+$ 636.0681, found 636.0682.

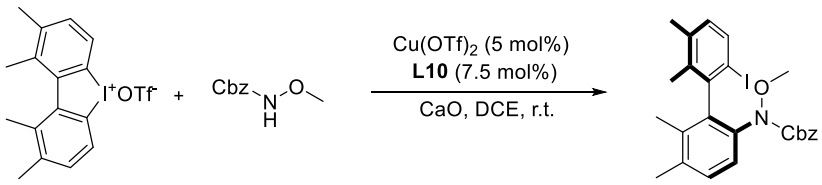
Compound **3n** was prepared following the **Procedure A**



1b **2a** **3n**

The reaction of **1b** (48.4 mg, 0.10 mmol, 1.0 equiv), **2a** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3n** (57 mg, >99%, 97% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +68.9 (c = 1.05, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.0 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 6.52 (d, *J* = 8.0 Hz, 1H), 3.73 (s, 3H), 2.46 (s, 3H), 2.32 (d, *J* = 2.4 Hz, 6H), 2.13 (s, 3H), 1.90 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.51, 143.90, 141.98, 138.68, 138.28, 137.18, 136.90, 136.00, 135.39, 132.38, 130.57, 130.03, 129.35, 129.03, 123.30, 97.96, 65.02, 21.67, 20.60, 20.27, 19.29, 16.56. HRMS (ESI): calcd for $C_{24}H_{26}INO_3SNa$ $(M+Na)^+$ 558.0576, found 558.0573.

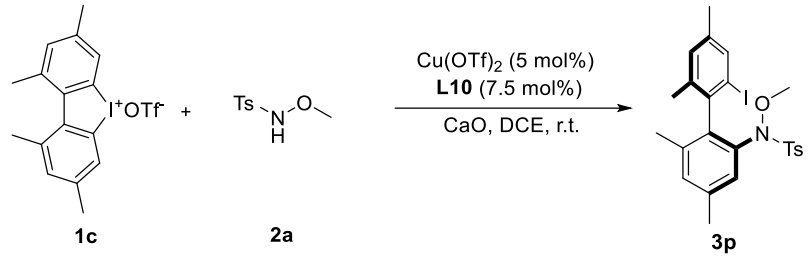
Compound **3o** was prepared following the **Procedure A**



1b **2i** **3o**

The reaction of **1b** (49.2 mg, 0.10 mmol, 1.0 equiv), **2i** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3o** (48.9 mg, 95%, 95% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +14.6 (c = 0.98, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.0 Hz, 1H), 7.35 – 7.26 (m, 5H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 5.18 (d, *J* = 12.4 Hz, 1H), 5.14 (d, *J* = 12.4 Hz, 1H), 3.45 (s, 3H), 2.35 (s, 3H), 2.19 (s, 3H), 1.87 (s, 3H), 1.82 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 155.23, 142.95, 142.04, 138.00, 137.00, 136.63, 136.32, 136.13, 135.69, 134.61, 130.57, 129.24, 128.32, 127.93, 126.23, 98.51, 67.35, 61.51, 20.60, 20.21, 18.03, 16.27. HRMS (ESI): calcd for $C_{25}H_{26}INO_3Na$ $(M+Na)^+$ 538.0855, found 538.0848.

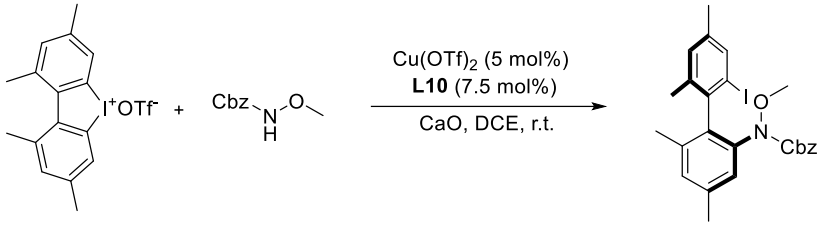
Compound **3p** was prepared following the **Procedure A**



1c **2a** **3p**

The reaction of **1c** (48.4 mg, 0.10 mmol, 1.0 equiv), **2a** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3p** (56.8 mg, >99%, 94% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +0.4 (c = 1.12, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.4 Hz, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.36 (s, 1H), 3.77 (s, 3H), 2.46 (s, 3H), 2.34 (s, 3H), 2.20 (s, 3H), 2.16 (s, 3H), 1.97 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.61, 140.22, 139.38, 139.24, 138.83, 138.75, 137.62, 137.44, 136.50, 132.16, 131.72, 130.80, 130.17, 128.93, 124.34, 101.42, 65.19, 21.71, 21.67, 20.96, 20.57, 20.11. HRMS (ESI): calcd for $C_{24}H_{26}INO_3SNa$ $(M+Na)^+$ 558.0576, found 558.0574.

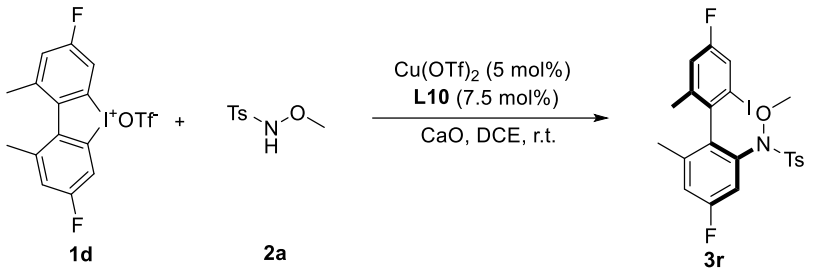
Compound **3q** was prepared following the **Procedure A**



1c **2i** **3q**

The reaction of **1c** (49.2 mg, 0.10 mmol, 1.0 equiv), **2i** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3q** (38.6 mg, 75%, 89% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +32.3 (c = 0.97, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.37 – 7.27 (m, 5H), 7.14 (s, 1H), 6.99 (s, 1H), 6.94 (s, 1H), 5.18 (s, 2H), 3.50 (s, 3H), 2.37 (s, 3H), 2.29 (s, 3H), 1.96 (s, 3H), 1.91 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 155.22, 139.23, 138.99, 138.72, 137.86, 137.70, 137.67, 136.83, 136.74, 136.10, 131.49, 130.63, 128.32, 127.96, 127.92, 127.20, 101.89, 67.38, 61.83, 21.12, 21.00, 20.55, 19.76. HRMS (ESI): calcd for $C_{25}H_{26}INO_3Na$ $(M+Na)^+$ 538.0855, found 538.0851.

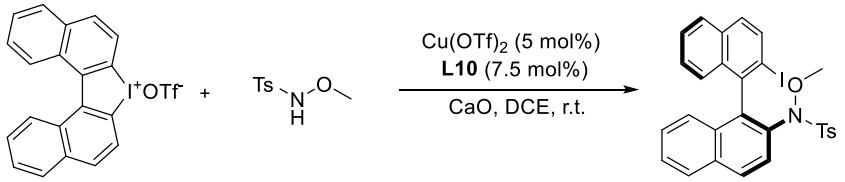
Compound **3r** was prepared following the **Procedure A**



1d **2a** **3r**

The reaction of **1d** (49.2 mg, 0.10 mmol, 1.0 equiv), **2a** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3r** (46 mg, 85%, 96% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +91.5 (c = 0.96, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.51 (dd, *J* = 7.8, 2.6 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.07 (dd, *J* = 9.2, 2.4 Hz, 1H), 7.01 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.25 (dd, *J* = 9.6, 2.4 Hz, 1H), 3.83 (s, 3H), 2.47 (s, 3H), 2.23 (s, 3H), 1.99 (s, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -112.49, -112.51, -112.54, -114.08, -114.10, -114.10, -114.13. **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.66, 160.18, 145.24, 141.63 (d, *J* = 7.9 Hz), 141.33 (d, *J* = 9.1 Hz), 139.81 (d, *J* = 8.8 Hz), 137.91 (d, *J* = 3.6 Hz), 136.89 (d, *J* = 3.4 Hz), 130.89, 130.20, 129.24, 123.24 (d, *J* = 23.5 Hz), 117.77 (d, *J* = 21.2 Hz), 116.89 (d, *J* = 20.6 Hz), 110.80 (d, *J* = 23.5 Hz), 100.52 (d, *J* = 7.9 Hz), 65.42, 22.13 (d, *J* = 1.5 Hz), 21.74, 20.35 (d, *J* = 1.8 Hz). HRMS (ESI): calcd for $C_{22}H_{20}F_2INO_3SNa$ $(M+Na)^+$ 566.0074, found 566.0078.

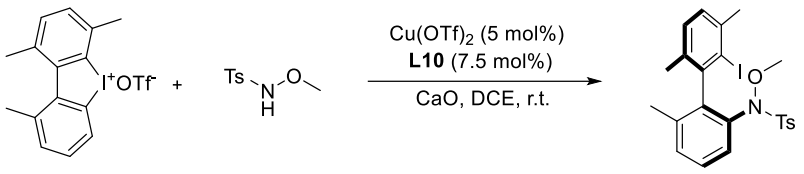
Compound **3s** was prepared following the **Procedure A**



1e **2a** **3s**

The reaction of **1e** (26.4 mg, 0.05 mmol, 1.0 equiv), **2a** (12 mg, 0.06 mmol, 1.2 equiv), $Cu(OTf)_2$ (0.9 mg, 5.0 mol%), **L10** (2.4 mg, 7.5 mol%) and CaO (5.6 mg, 0.10% mmol, 2.0 equiv) in DCE (1.0 mL) at room temperature for 12 h afforded **3s** (21.1 mg, 73%, >99% ee). Rf = 0.3 (PE/EA = 10:1). $[\alpha]_D^{20}$ = -24.2 (c = 0.38, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 10:90, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.8 Hz, 1H), 7.92 (dd, *J* = 8.2, 2.8 Hz, 2H), 7.84 (d, *J* = 8.8 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.53 (tdd, *J* = 8.0, 6.8, 1.2 Hz, 2H), 7.43 – 7.29 (m, 5H), 7.21 (dd, *J* = 8.4, 3.2 Hz, 3H), 7.03 (d, *J* = 8.8 Hz, 1H), 3.55 (s, 3H), 2.44 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.71, 139.89, 139.30, 137.17, 135.54, 134.27, 133.43, 132.62, 132.37, 132.15, 129.94, 129.54, 129.17, 129.14, 128.70, 128.02, 127.61, 127.20, 127.17, 126.98, 126.41, 126.23, 123.23, 100.64, 65.13, 21.70. HRMS (ESI): calcd for $C_{28}H_{22}INO_3SNa$ $(M+Na)^+$ 602.0263, found 602.0273.

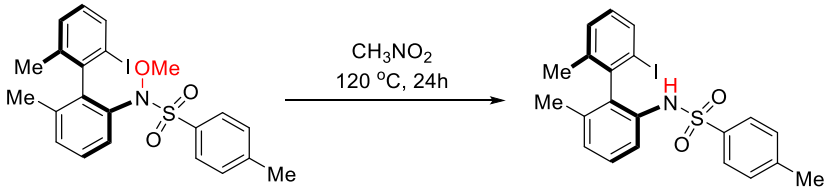
Compound **3t** was prepared following the **Procedure A**



1f **2a** **3t**

The reaction of **1f** (47.0 mg, 0.10 mmol, 1.0 equiv), **2a** (24.1 mg, 0.12 mmol, 1.2 equiv), $Cu(OTf)_2$ (1.8 mg, 5.0 mol%), **L10** (4.2 mg, 7.5 mol%) and CaO (11.2 mg, 0.20 mmol, 2.0 equiv) in DCE (2.0 mL) at room temperature for 6 h afforded **3t** (48.2 mg, 93%, 95% ee). Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +0.7 (c = 1.05, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 7.6 Hz, 3H), 7.22 – 7.17 (m, 2H), 7.15 (t, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 7.6 Hz, 1H), 3.70 (s, 3H), 2.49 (s, 3H), 2.46 (s, 3H), 2.21 (s, 3H), 2.01 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.64, 144.24, 142.16, 139.35, 138.92, 137.72, 136.57, 132.20, 130.90, 130.06, 129.58, 129.08, 128.67, 127.78, 123.74, 107.86, 65.12, 29.55, 21.69, 21.41, 20.14. HRMS (ESI): calcd for $C_{23}H_{24}INO_3SNa$ $(M+Na)^+$ 544.0419, found 544.0421.

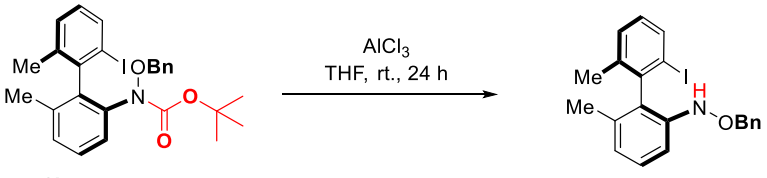
Synthesis of compound **4a**



3a, 98% ee **4a**, 71%, 99% ee

In a 10 mL schlenk tube capped with PTFE screw cap, **3a** (101 mg, 0.20 mmol, 98% ee) was dissolved in CH_3NO_2 (1.0 mL) under nitrogen. The mixture was heated to 120 ℃ and stirred for 24 h. After complete consumption of starting material, the reaction was cooled to room temperature, the solvent was removed and the residue was purified by column chromatography on silica gel (hexanes/ethyl acetate = 20:1) to afford the desired product **4a** (67.7 mg, 71%, 99% ee). Rf = 0.3 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +31.6 (c = 0.98, $CHCl_3$). HPLC conditions: Chiralpak ID-H, isopropanol/hexane = 5:95, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.77 – 7.71 (m, 2H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.23 (t, *J* = 8.6 Hz, 3H), 7.04 (t, *J* = 7.8 Hz, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 5.98 (s, 1H), 2.38 (s, 3H), 1.83 (s, 3H), 1.76 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.92, 139.51, 139.07, 137.46, 136.73, 136.60, 133.73, 132.53, 130.69, 130.40, 129.62, 128.67, 127.55, 125.44, 114.96, 101.38, 21.48, 20.77, 19.70. HRMS (ESI): calcd for $C_{21}H_{20}INO_2SNa$ $(M+Na)^+$ 500.0157, found 500.0156.

Synthesis of compound **5j**

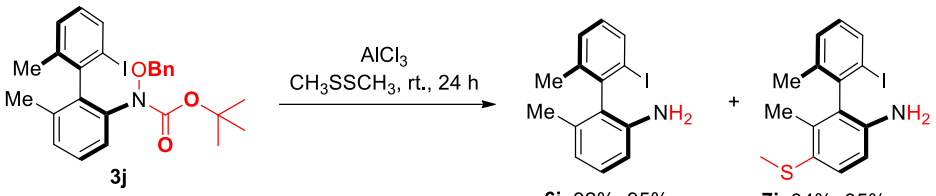


3j, 95%ee **5j**, 78%, 96% ee

To a 25 mL Schleck tube containing **3j** (106 mg, 0.20 mmol, 1.0equiv, 95% ee) and THF (1.0 mL) was added anhydrous $AlCl_3$ (80 mg, 0.60 mmol, 3.0 equiv) as one portion under nitrogen, and stirred for 24

h at room temperature. After complete consumption of starting material, the solution was quenched with 4M HCl solution (2 mL) and water (10 mL), extracted with ethyl acetate (10 mL x 2), dried over anhydrous sodium sulfate and purified by flash column chromatography on silica gel (hexanes/ethyl acetate = 50:1) to afford the desired product **5j** (66.4 mg, 78%, 96% ee). Rf = 0.7 (PE/EA = 20:1). $[\alpha]_D^{20}$ = +39.8 (c = 1.23, $CHCl_3$). HPLC conditions: Chiralpak OD-H, isopropanol/hexane = 1:99, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (t, *J* = 7.6 Hz, 1H), 7.43 – 7.26 (m, 7H), 7.15 (t, *J* = 9.2 Hz, 1H), 7.04 – 6.90 (m, 2H), 6.52 (d, *J* = 10.8 Hz, 1H), 4.80 (qd, *J* = 11.1, 8.4 Hz, 2H), 2.05 (d, *J* = 9.2 Hz, 3H), 1.92 (d, *J* = 9.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.75, 140.90, 138.90, 137.13, 136.50, 135.73, 130.30, 129.56, 129.19, 128.92, 128.68, 128.38, 128.14, 123.26, 112.13, 101.40, 77.35, 21.17, 19.32. HRMS (ESI): calcd for $C_{21}H_{20}INONa$ $(M+Na)^+$ 452.0487, found 452.0489.

Synthesis of compound **6j** and **7j**



3j
from 1.0 mmol scale reaction
91%, 95% ee

6j, 32%, 95% ee

7j, 34%, 95% ee

To a 25 mL Schleck tube containing **3j** (481.3 mg, 0.91 mmol, 1.0 equiv, 95% ee) and dimethyl disulfide (5.0 mL) was added anhydrous $AlCl_3$ (364 mg, 2.73 mmol, 3.0 equiv) as one portion under nitrogen, and stirred for 24 h at room temperature. After complete consumption of starting material, the solution was quenched with 4M HCl solution (4 mL) and water (10 mL), extracted with ethyl acetate (10 mL x 2), dried over anhydrous sodium sulfate and purified by flash column chromatography on silica gel (hexanes/ethyl acetate = 50:1) to afford product **6j** (92.6 mg, 32%, 95% ee) and **7j** (114.2 mg, 34%, 95% ee).

6j: Rf = 0.5 (PE/EA = 10:1). $[\alpha]_D^{20}$ = +34.1 (c = 0.74, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 1:99, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 6.65 (d, *J* = 8.0 Hz, 1H), 3.29 (s, 2H), 2.09 (s, 3H), 1.89 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.87, 142.04, 139.04, 137.14, 136.22, 130.31, 129.65, 129.35, 128.53, 120.01, 112.92, 102.08, 21.13, 19.71. HRMS (ESI): calcd for $C_{14}H_{14}INNa$ $(M+Na)^+$ 346.0069, found 346.0063.

7j: Rf = 0.4 (PE/EA = 10:1). $[\alpha]_D^{20}$ = -14.1 (c = 1.40, $CHCl_3$). HPLC conditions: Chiralpak AD-H, isopropanol/hexane = 2:98, flow: 1.0 mL/min, λ = 254 nm. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 6.8 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.66 (d, *J* = 8.4 Hz, 1H), 3.67 – 2.61 (broad, 2H), 2.40 (s, 3H), 2.07 (s, 3H), 1.99 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.09, 141.70, 139.00, 137.23, 136.52, 130.41, 130.37, 130.12, 129.45, 125.49, 113.51, 101.99, 21.16, 18.05, 17.00. HRMS (ESI): calcd for $C_{15}H_{17}INS$ $(M+H)^+$ 370.0126, found 370.0131.

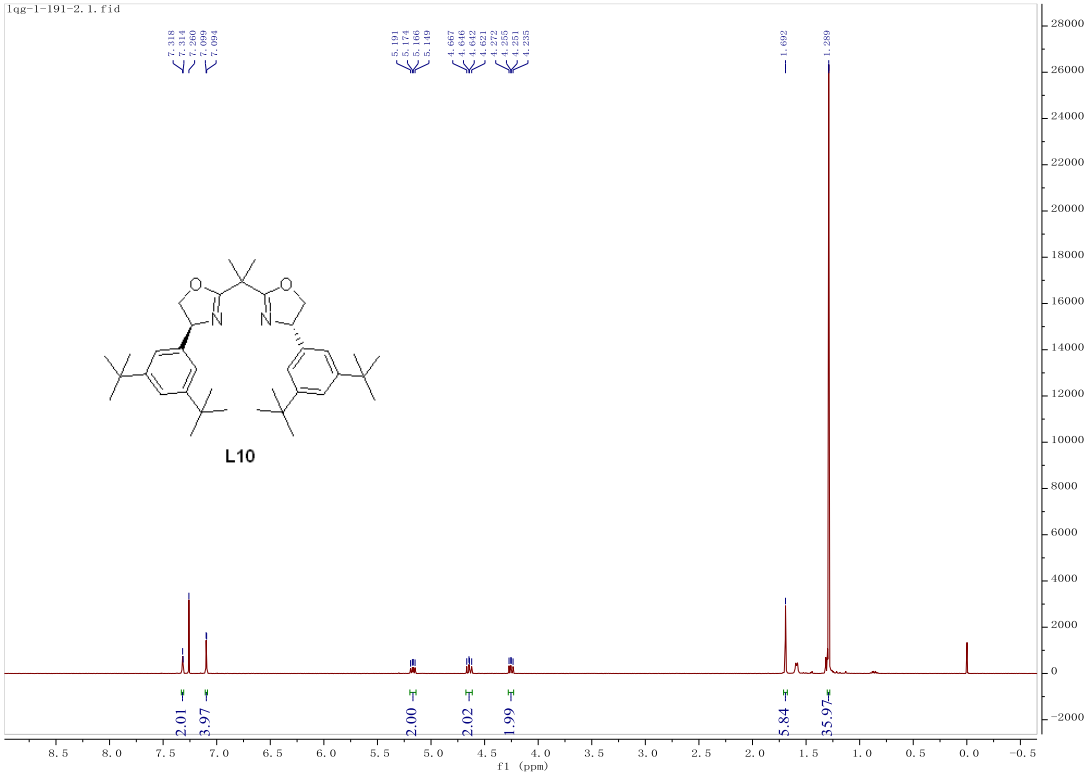
References

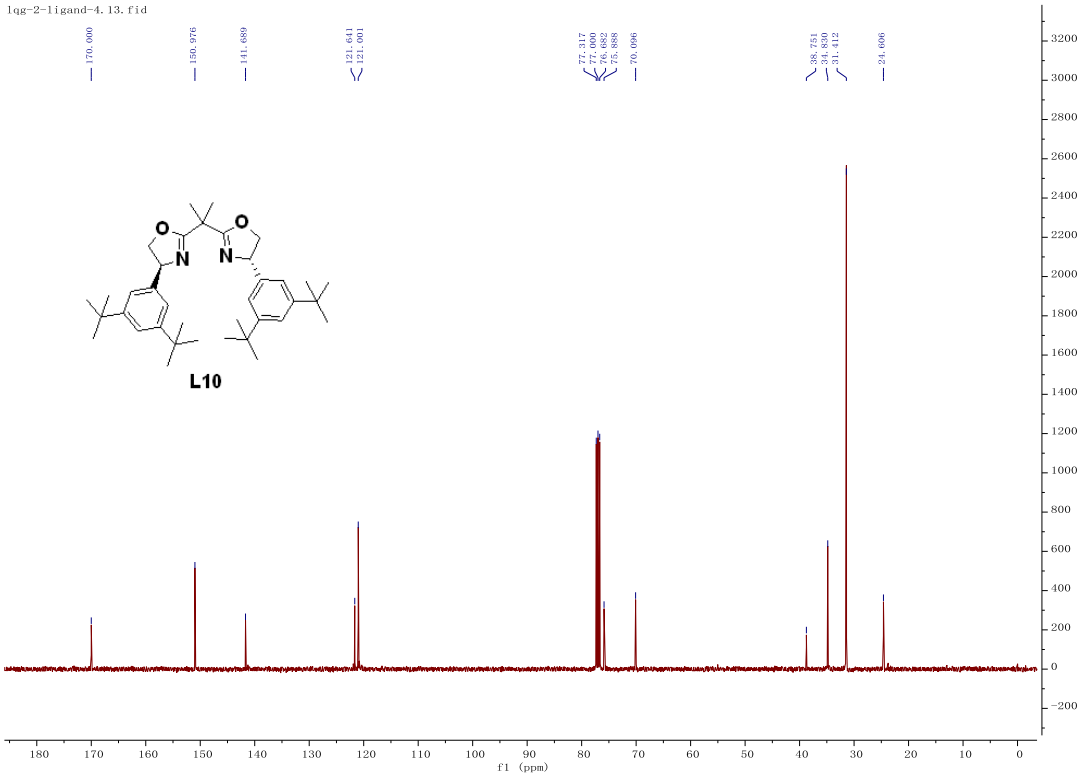
1. Zhao, K.; Duan, L.; Xu, S.; *J*iang, L.; Fu, Y.; Gu, Z. *Chem* **2018**, *4*, 599.

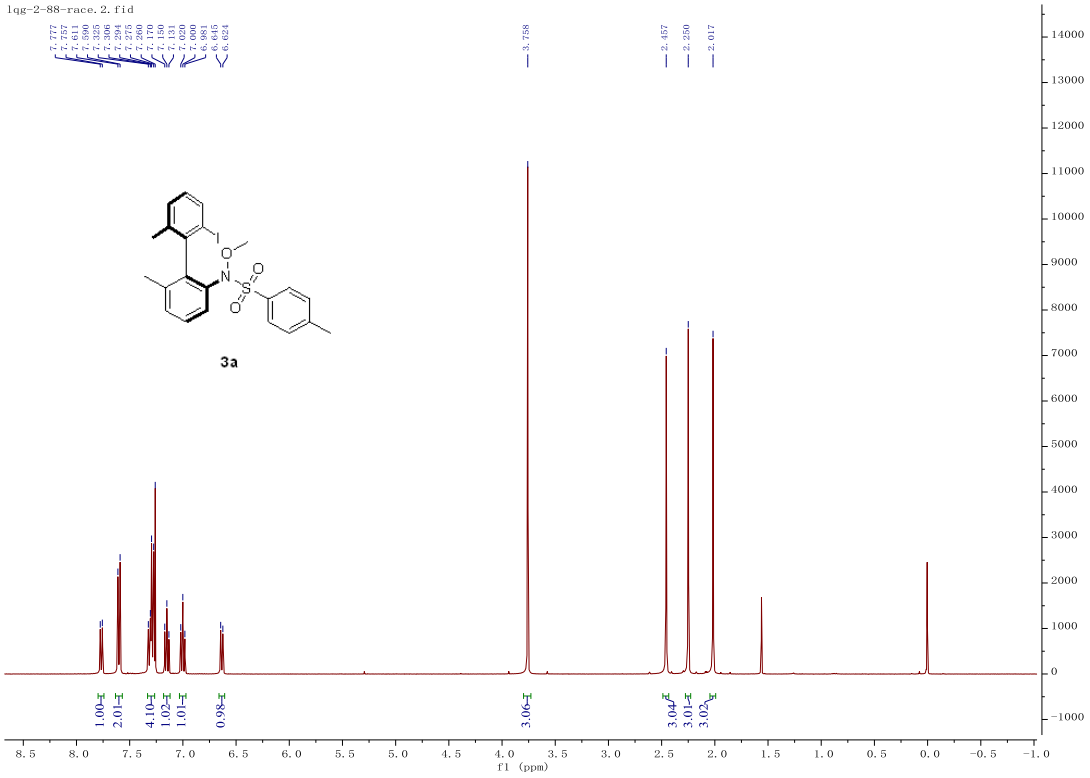
2. Reddy, L, R.; Gupta, A, P.; Liu, Y. *J. Org. Chem.* **2011**, *76*, 3409.

3. Zuo, Z.; Cong, H.; Li, W.; Choi, *J*.; Fu, G, C.; Macmillan, W, C. *J. Am. Chem. Soc.* **2016**, *138*, 1832.

Copies of NMR Spectroscopies



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6.645
6.624
3.758
2.457
2.250
2.017
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4.10
1.02
1.01
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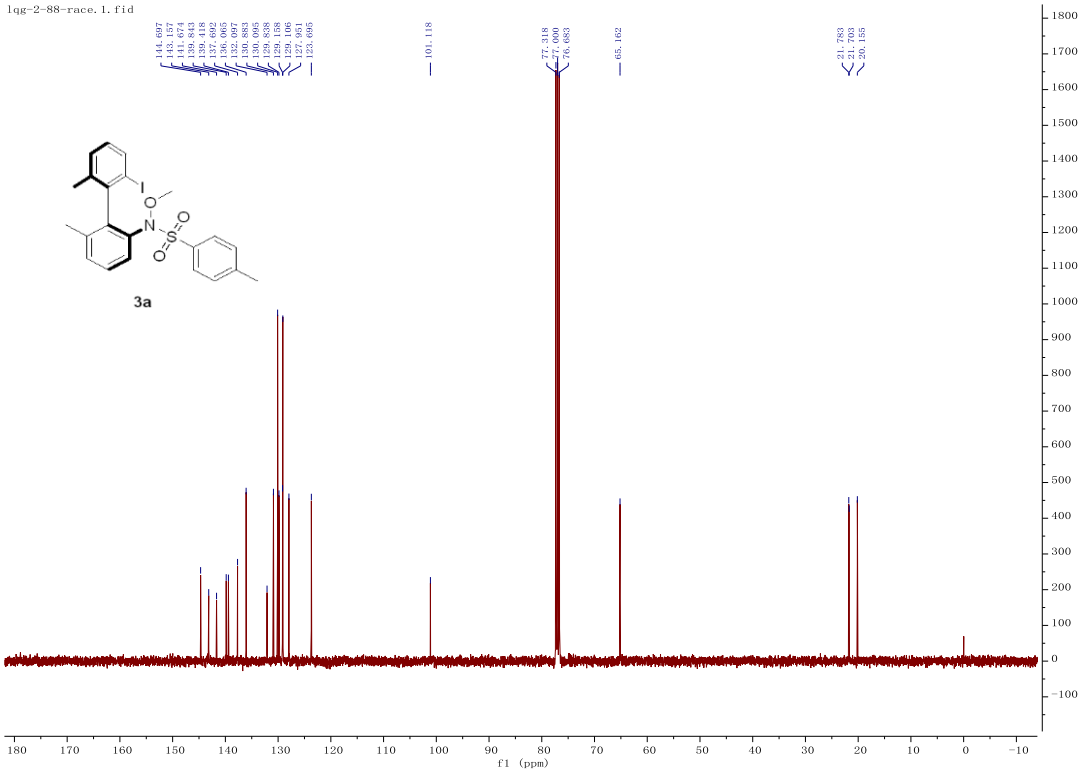
3.06
3.04
3.01
3.02
f1 (ppm)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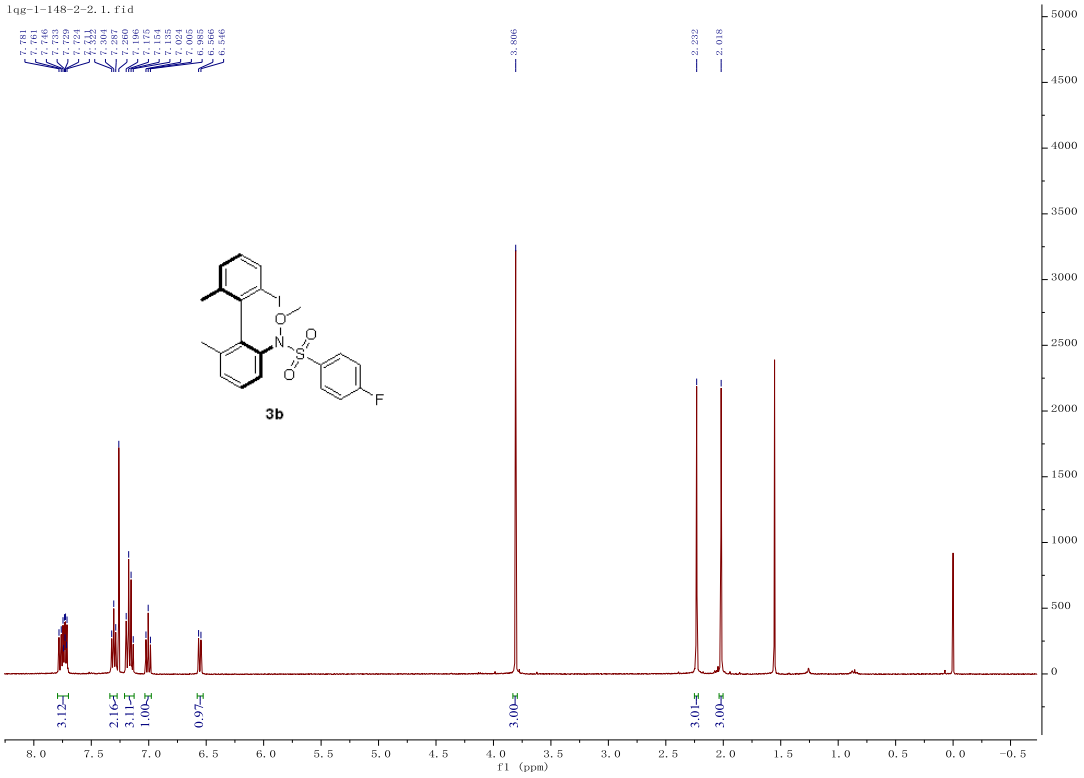
f1 (ppm)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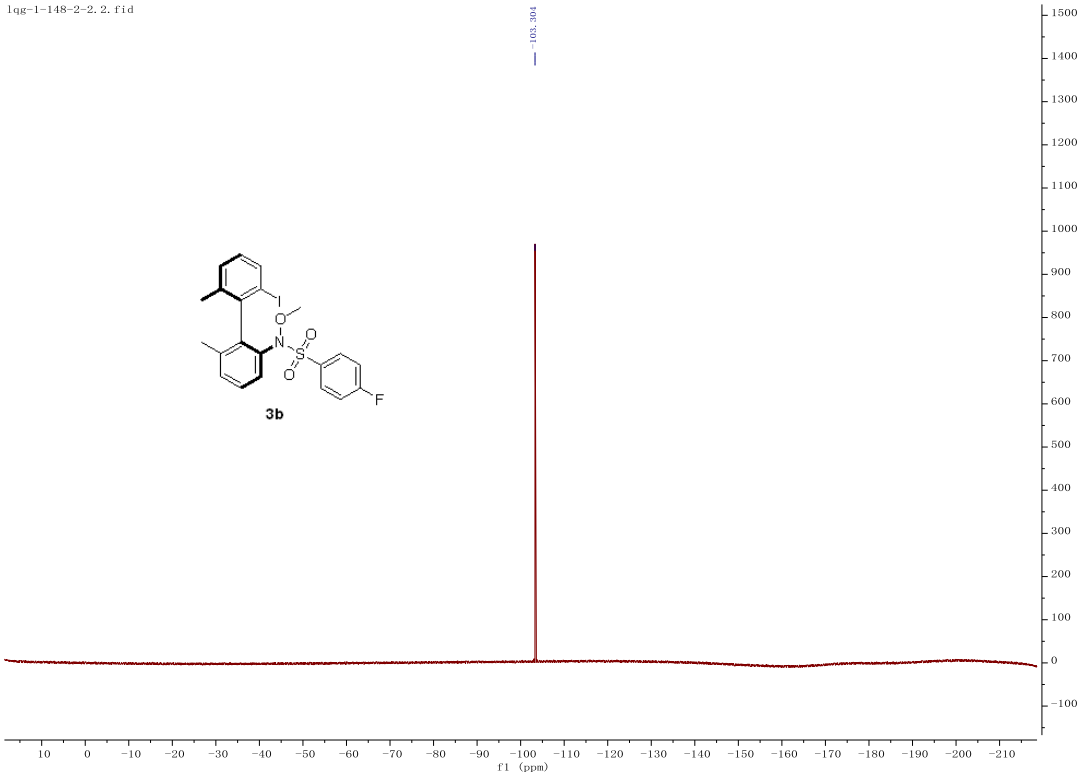
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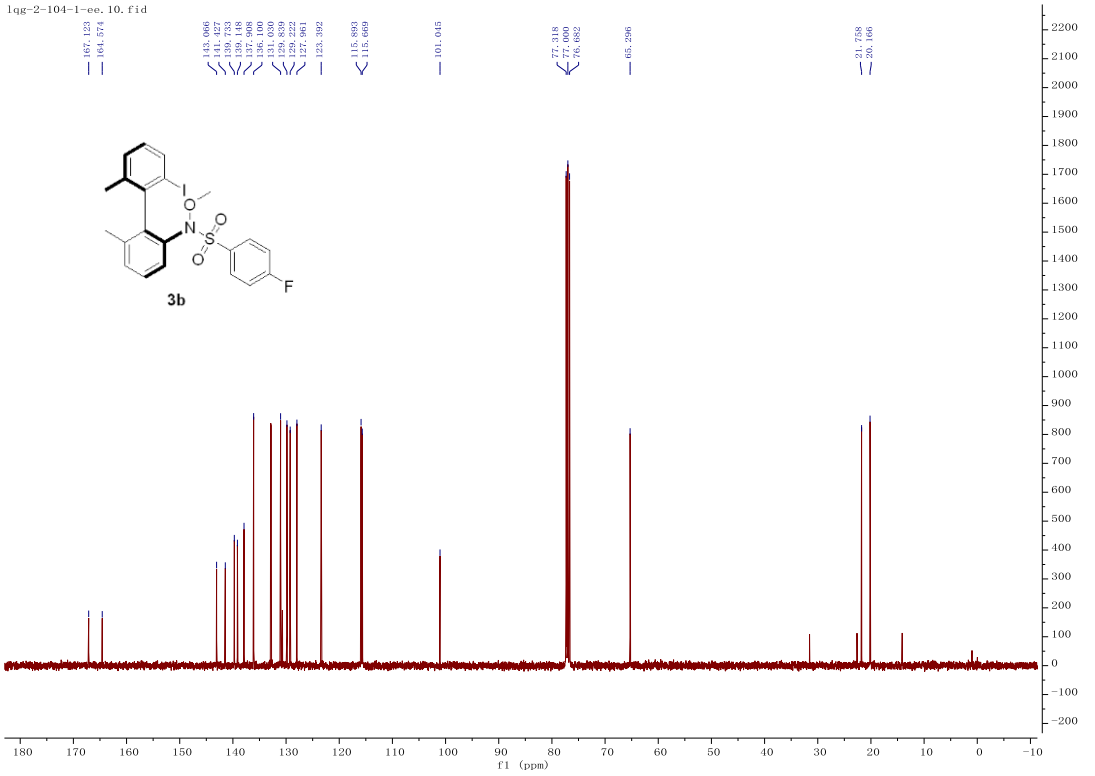
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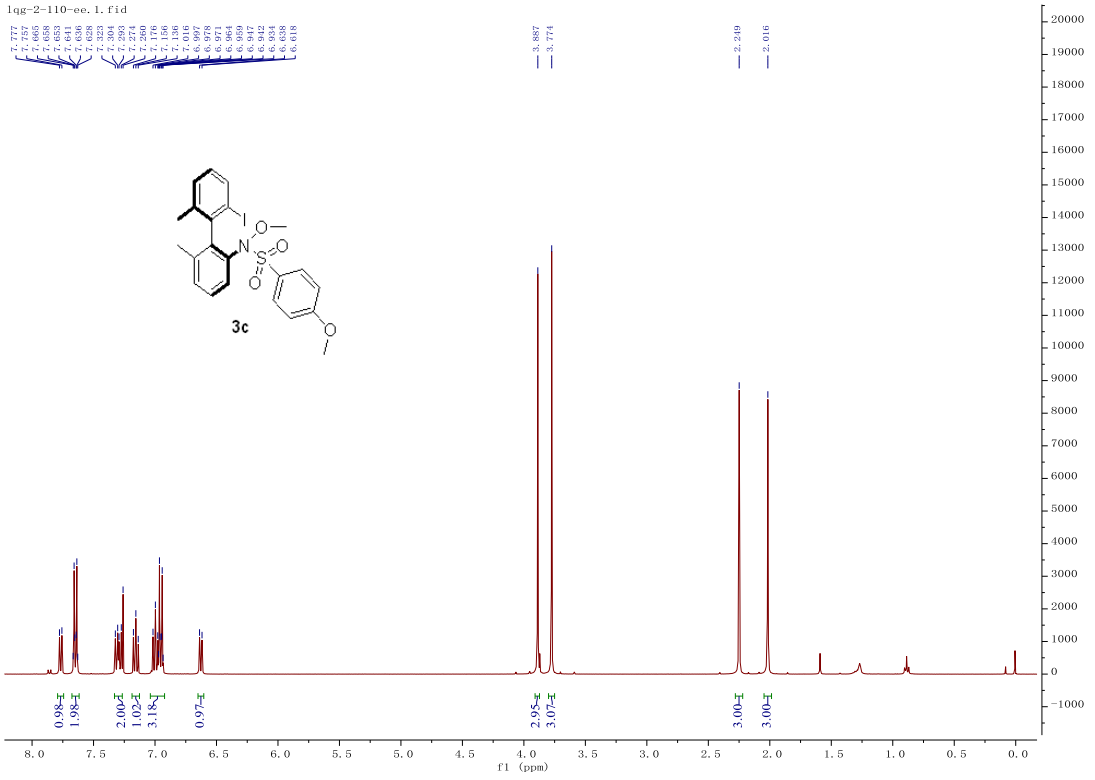
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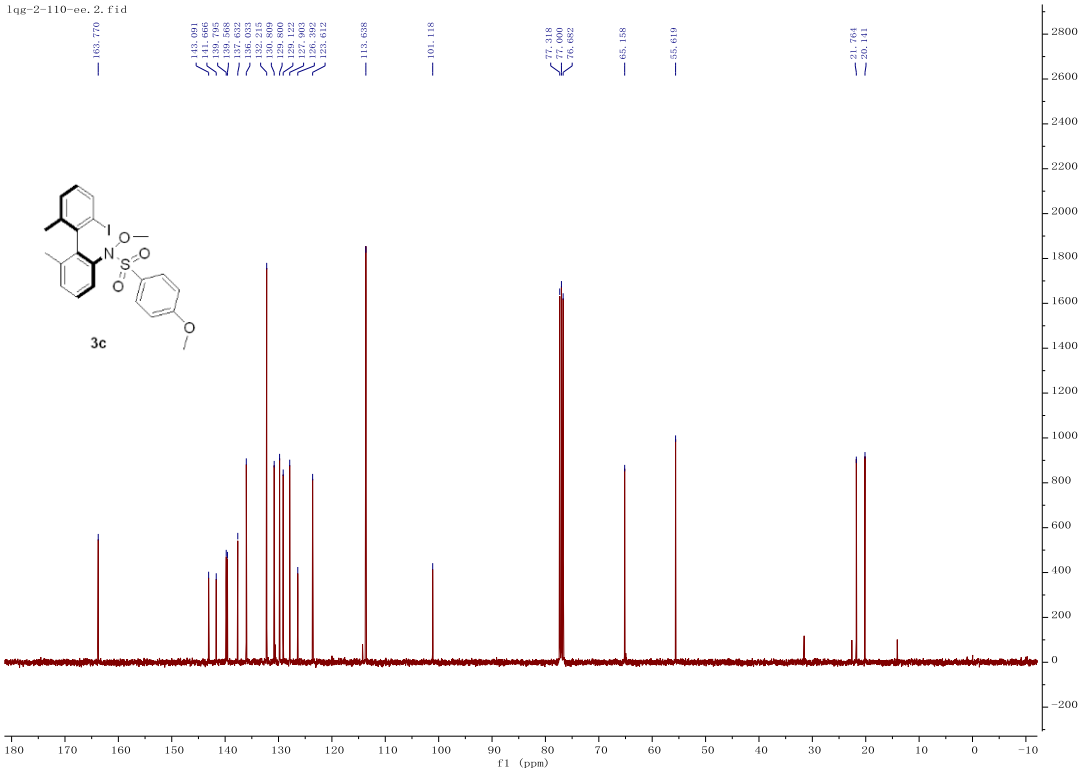
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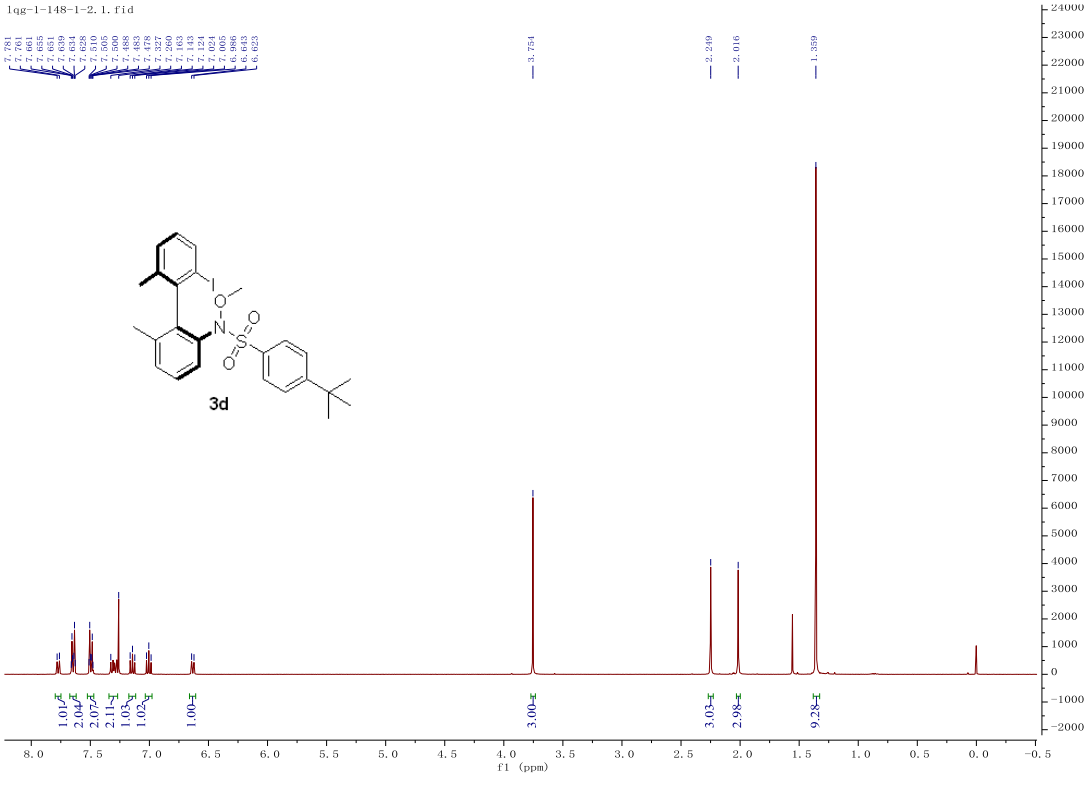
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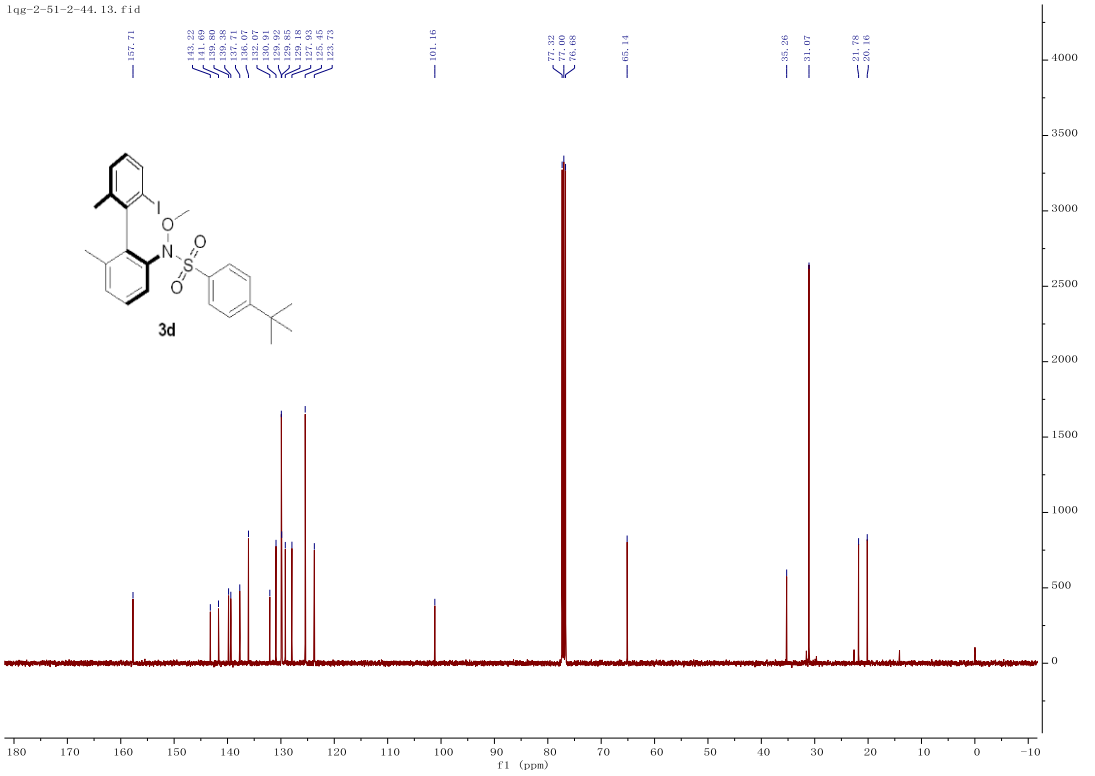
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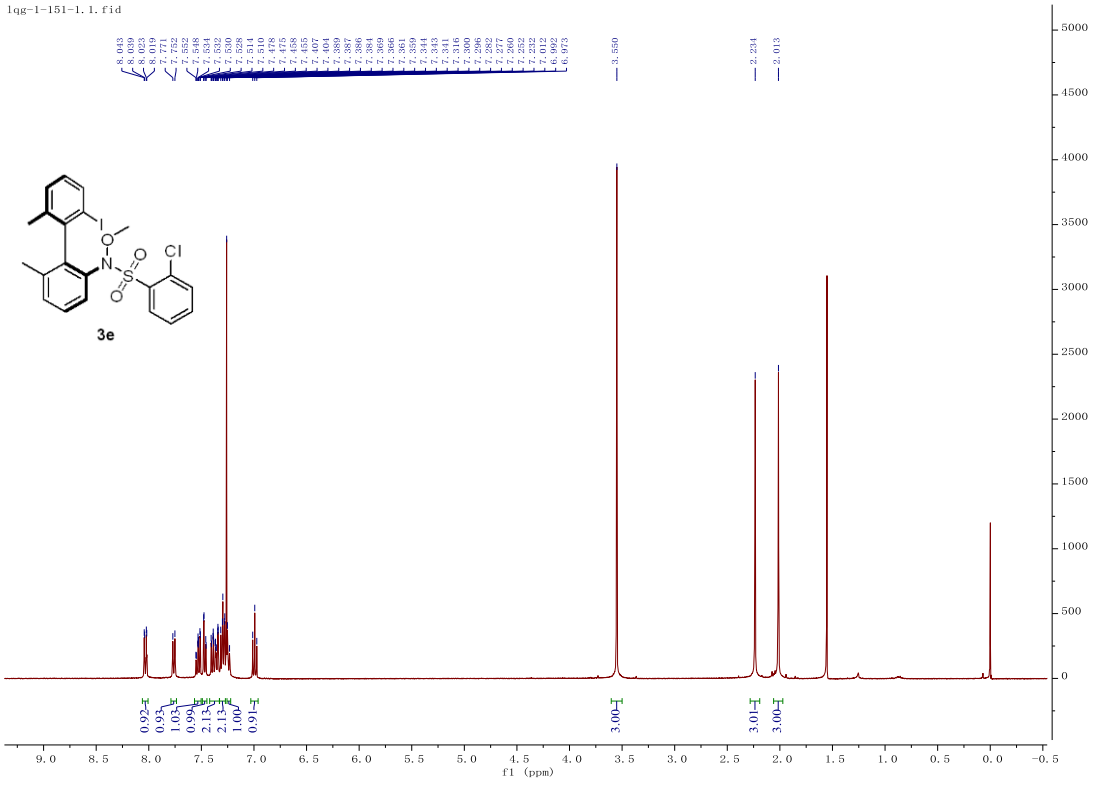
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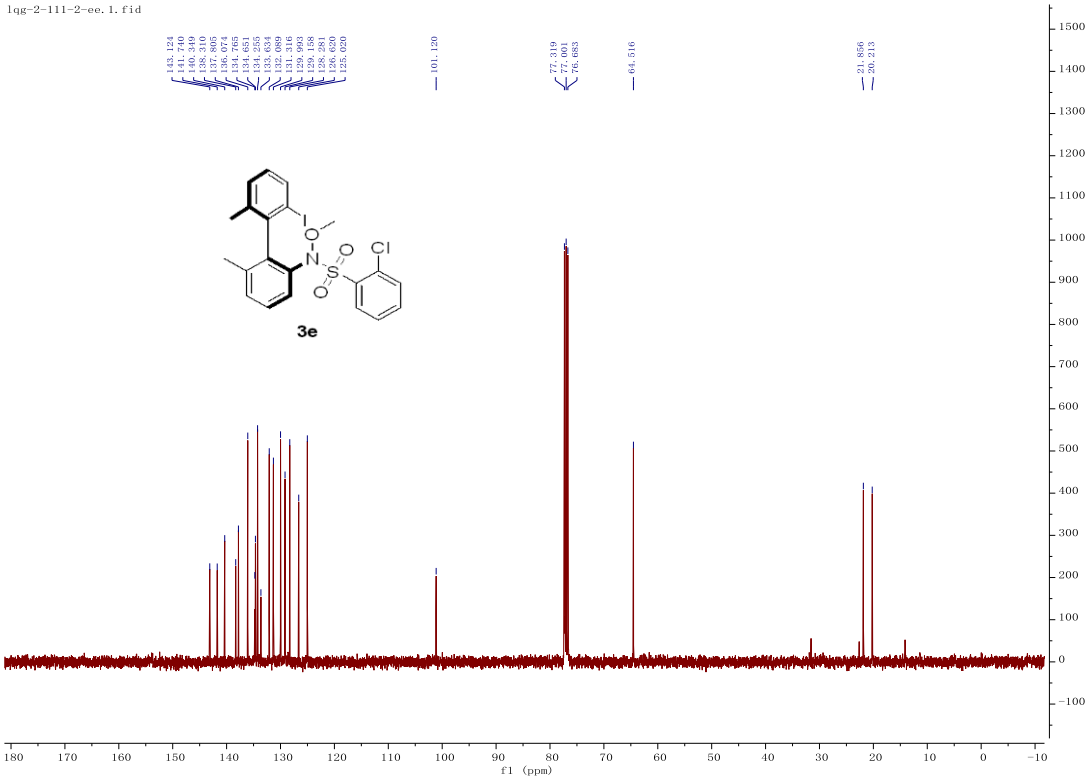
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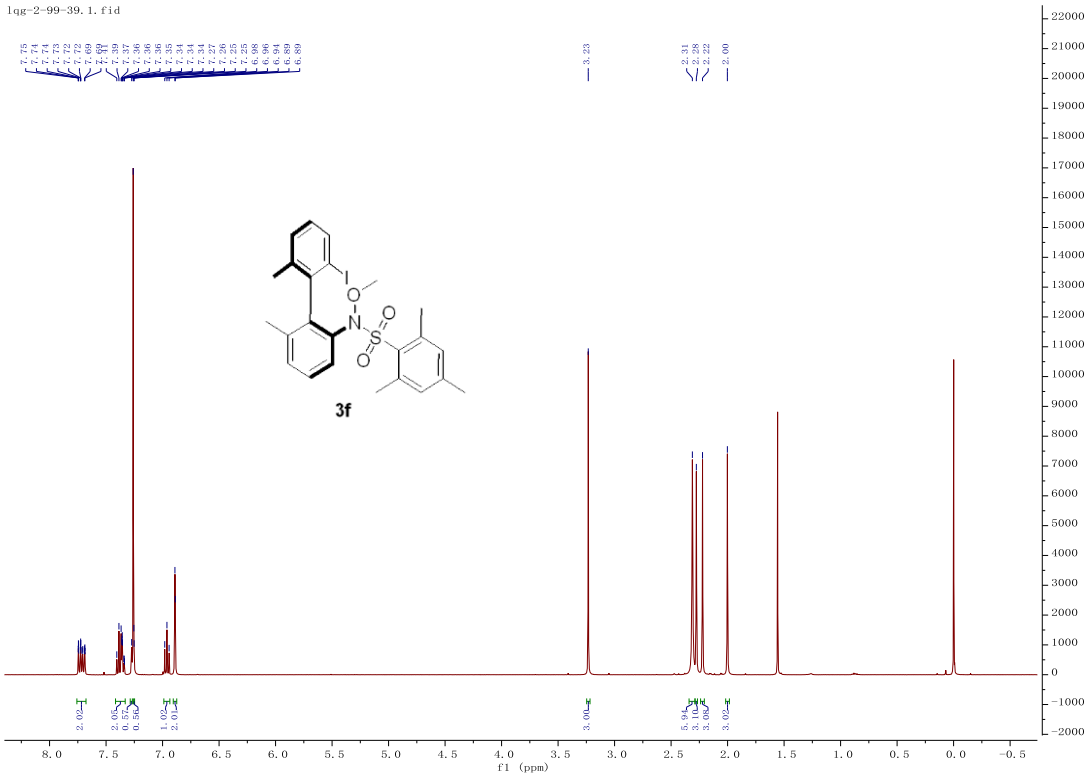
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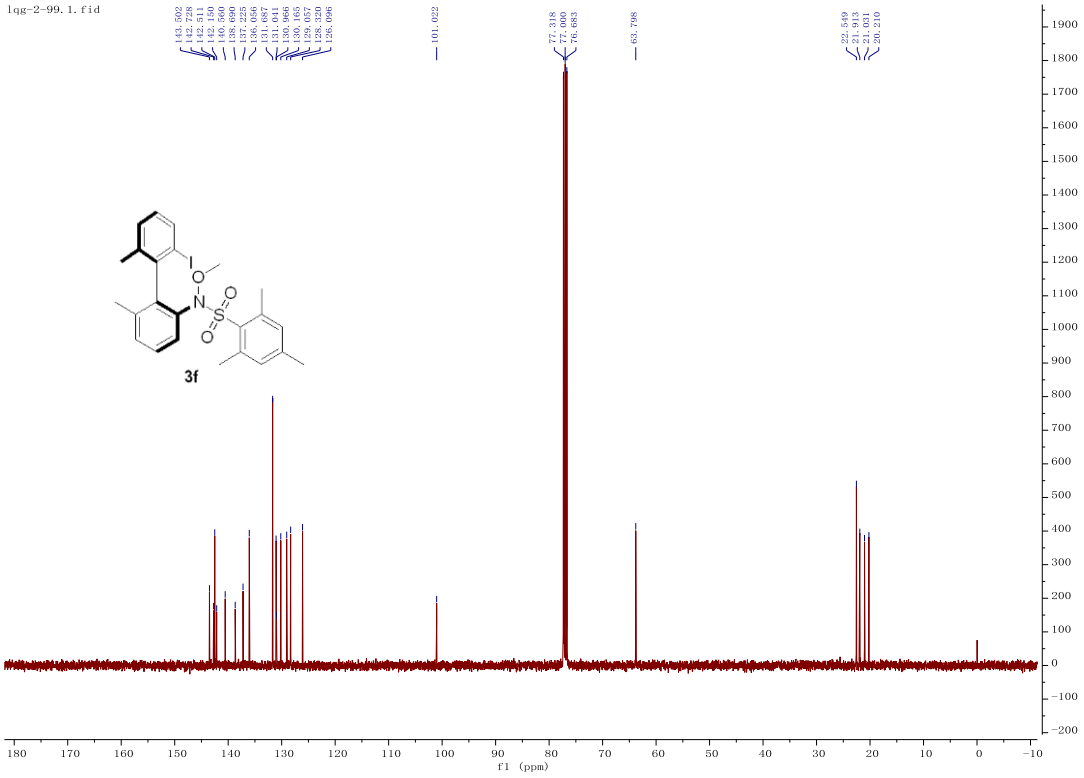
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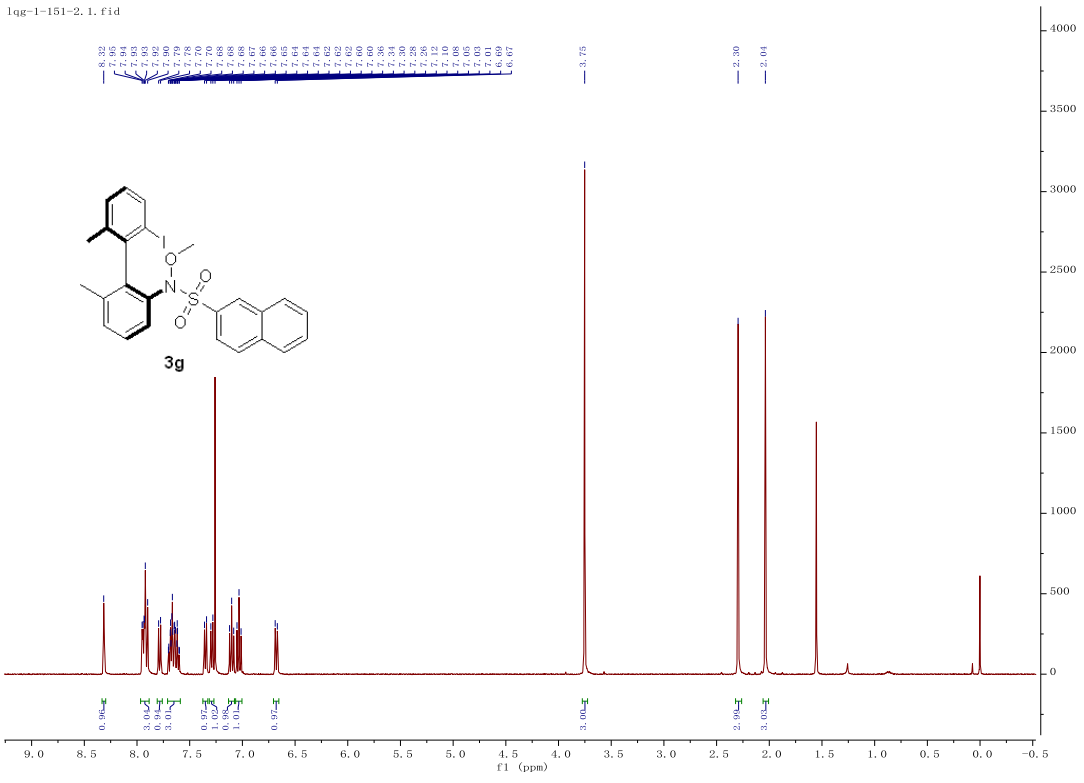
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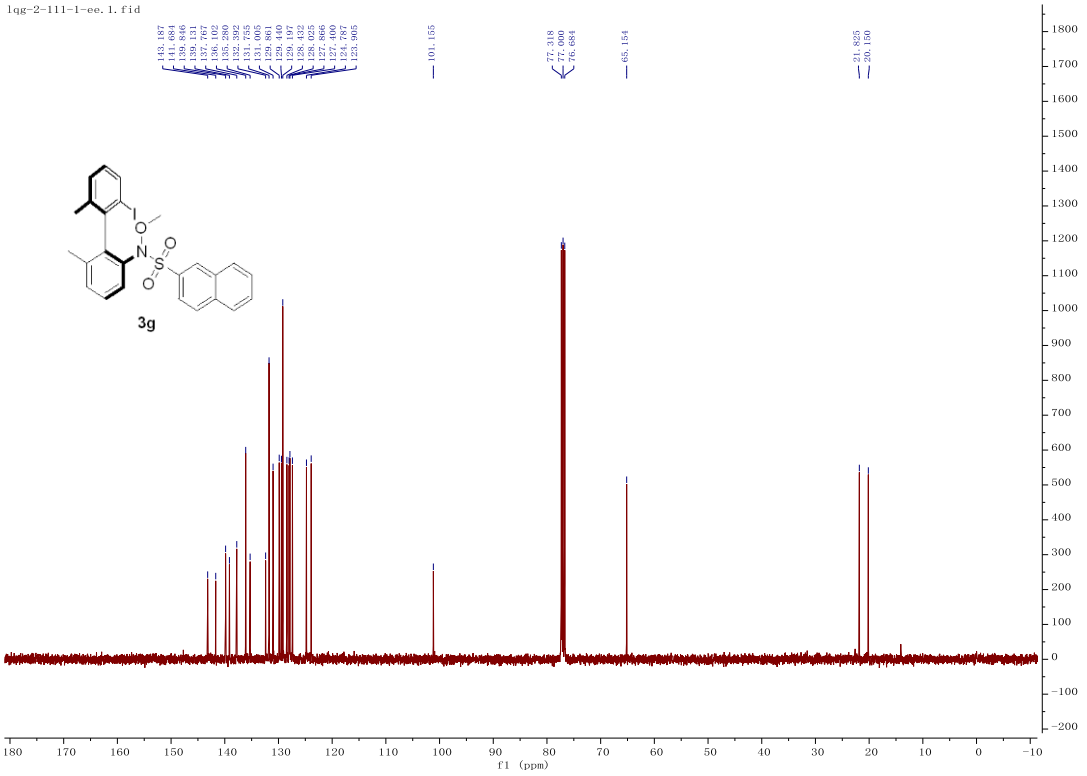
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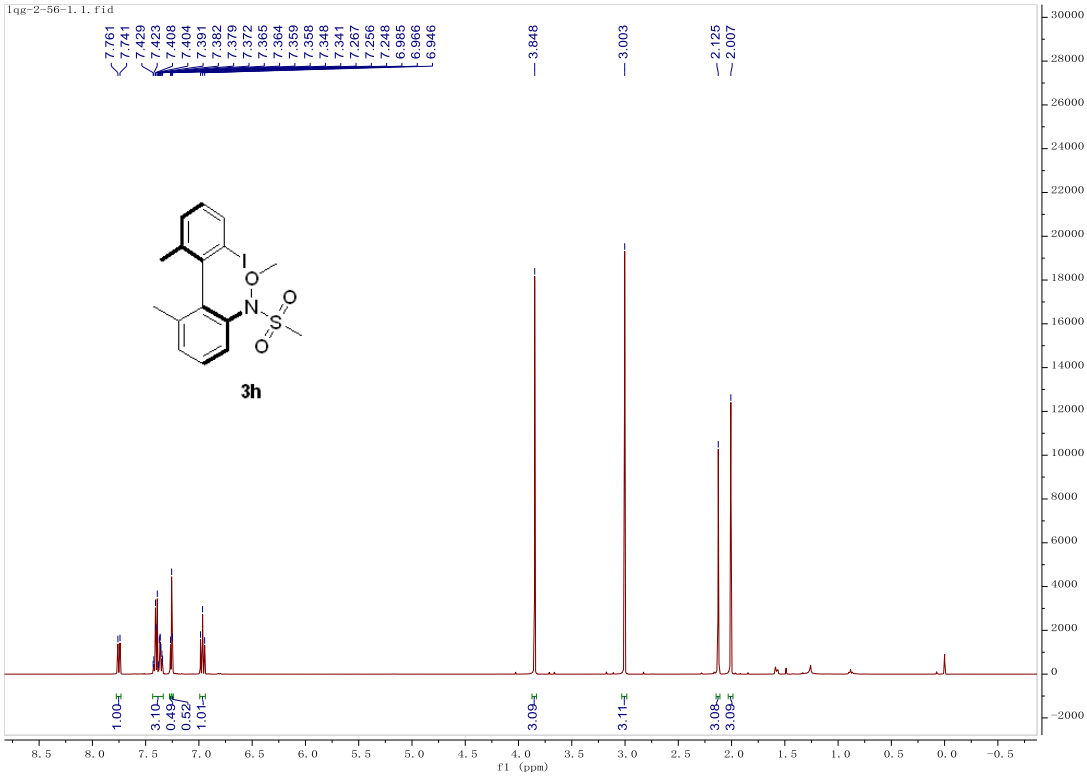
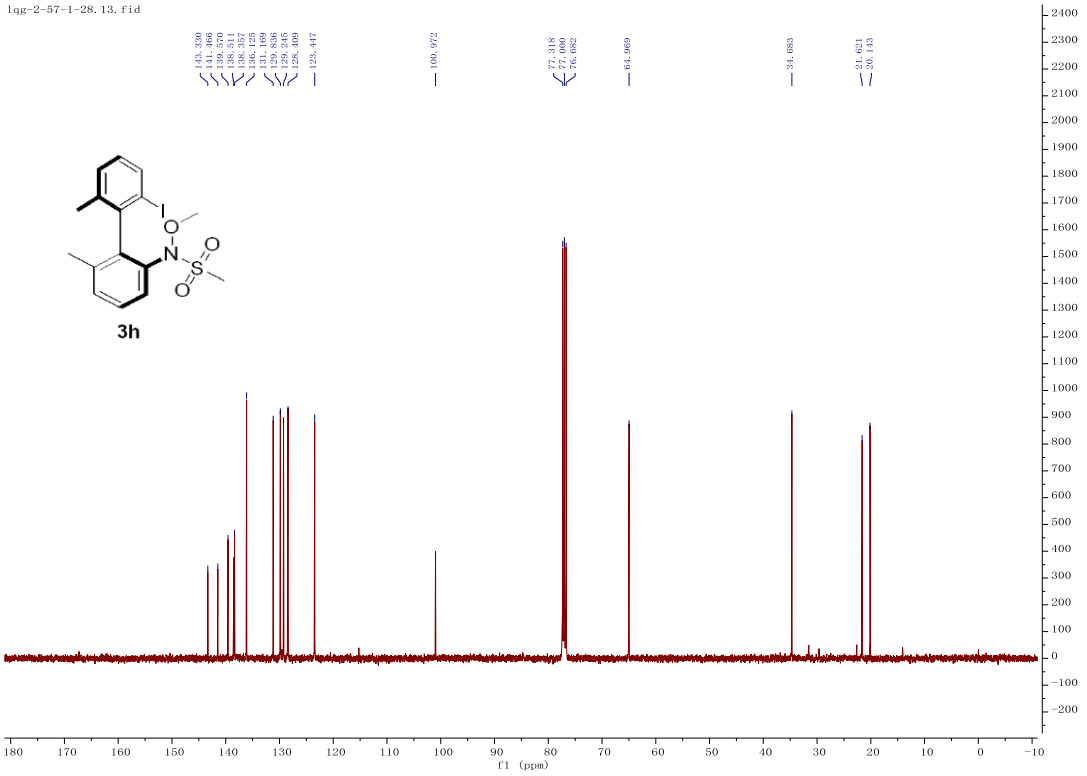
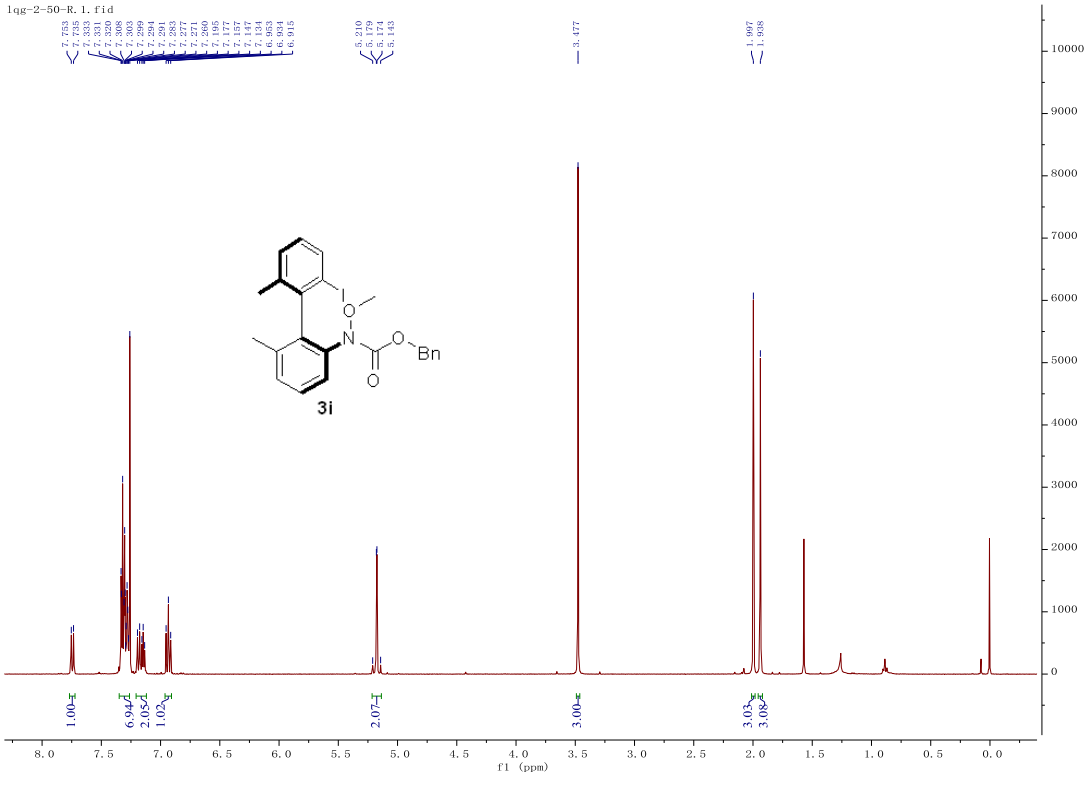
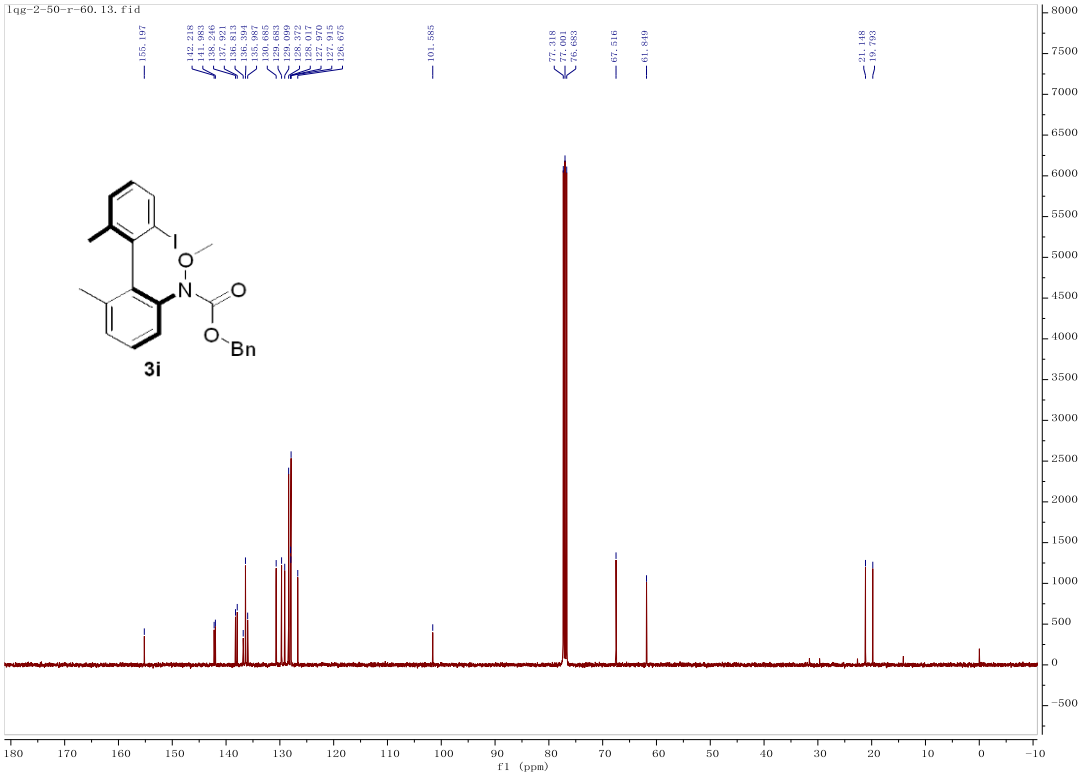
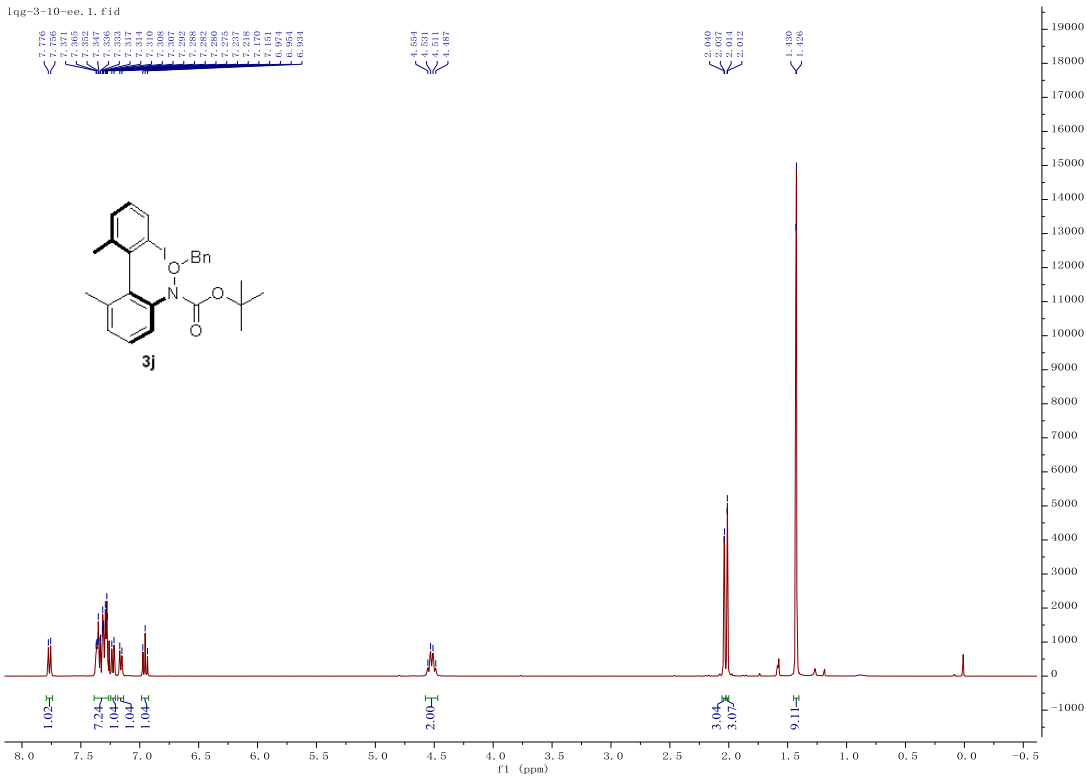
3g
f1 (ppm)

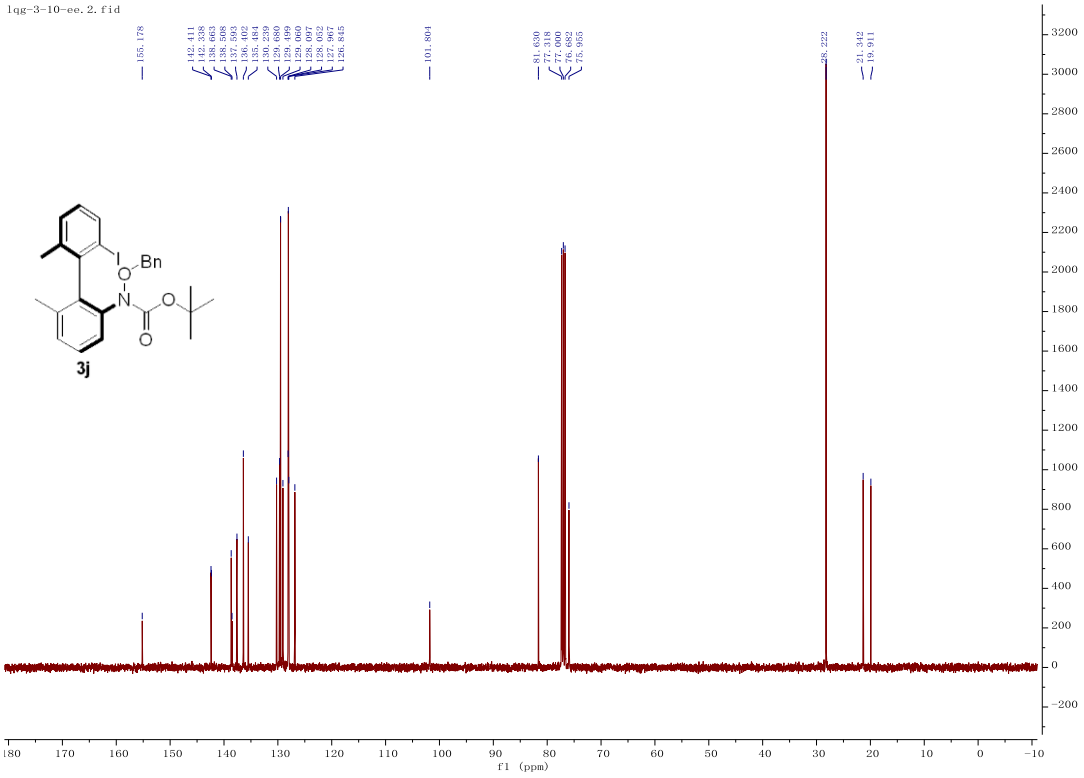

lqg-2-56-1.1.fid
7.761
7.741
7.429
7.423
7.408
7.404
7.391
7.382
7.379
7.372
7.365
7.364
7.359
7.358
7.348
7.341
7.267
7.256
7.248
6.985
6.966
6.946
3.848
3.003
2.125
2.007
1.00
3.10
0.49
0.52
1.01
3.09
3.11
3.08
3.09
3h
f1 (ppm)

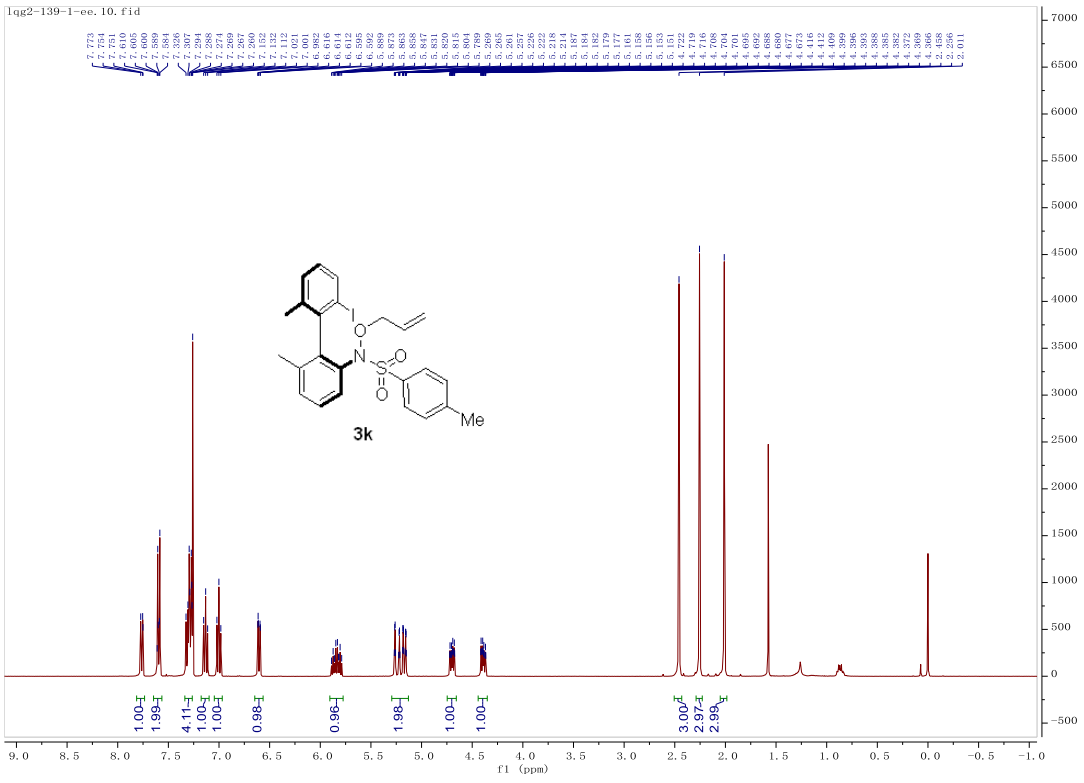

lqg-2-57-1-28.13.fid
143.330
141.466
139.570
138.511
138.357
136.125
131.169
129.836
129.245
128.409
123.447
100.972
77.318
77.000
76.682
64.969
34.683
21.621
20.143
3h
f1 (ppm)

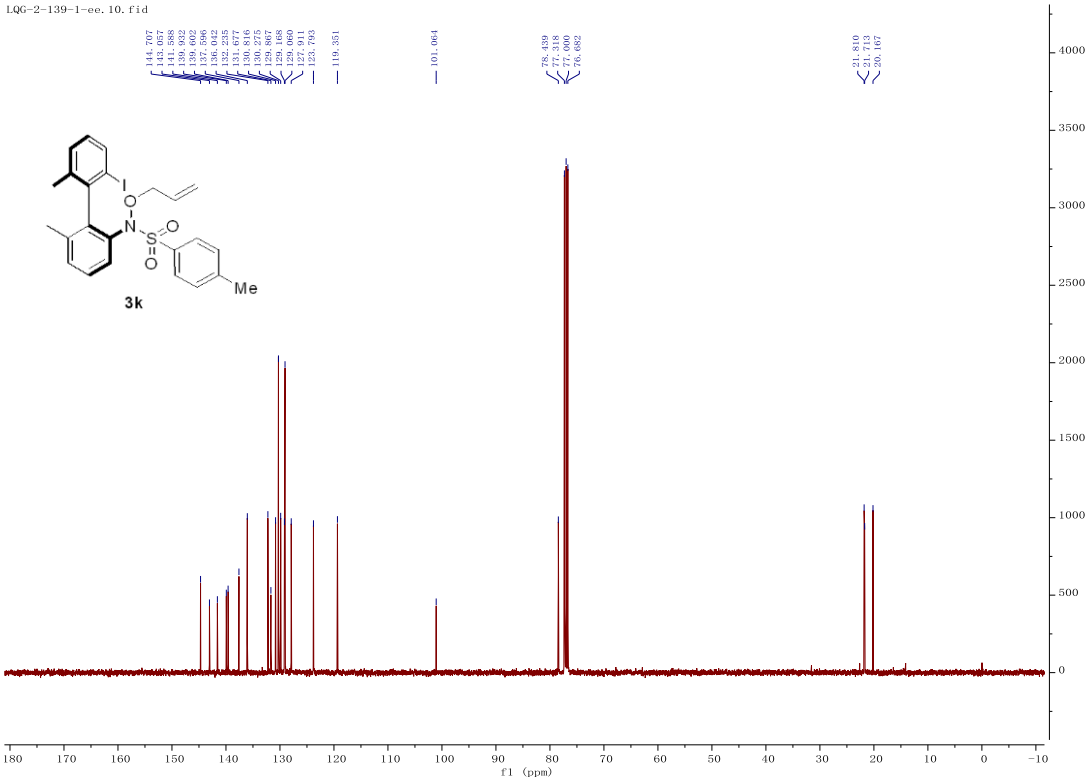

lqg-2-50-R.1.fid
7.753
7.738
7.393
7.331
7.320
7.308
7.303
7.298
7.294
7.291
7.283
7.277
7.271
7.260
7.195
7.177
7.157
7.147
7.134
6.953
6.934
6.915
5.210
5.179
5.174
5.143
3.477
1.997
1.958
Bn
3i
1.00
6.94
2.05
1.02
2.07
3.00
3.03
3.08
f1 (ppm)

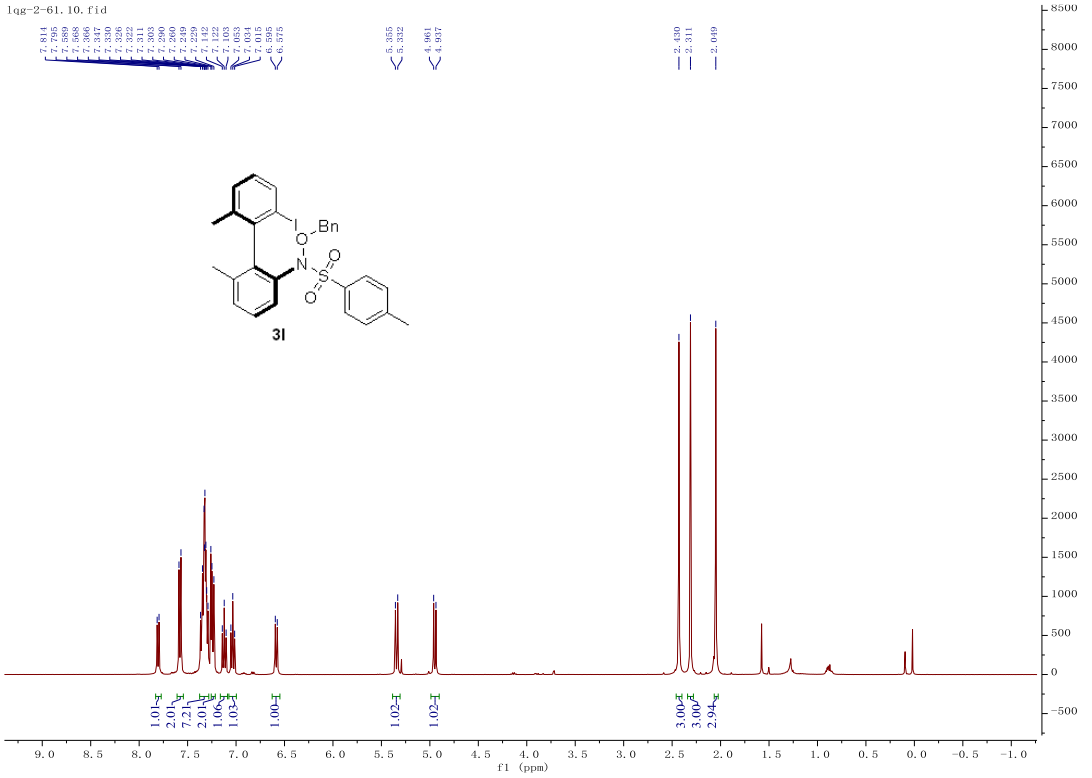

lqg-2-50-r-60.13.fid
155.197
142.218
141.983
139.566
137.921
136.813
136.394
135.697
135.085
129.683
129.099
128.372
128.017
127.970
127.915
126.675
101.585
77.318
77.001
76.683
67.516
61.849
21.148
19.793
I
O
N
O
O
Bn
3i
f1 (ppm)

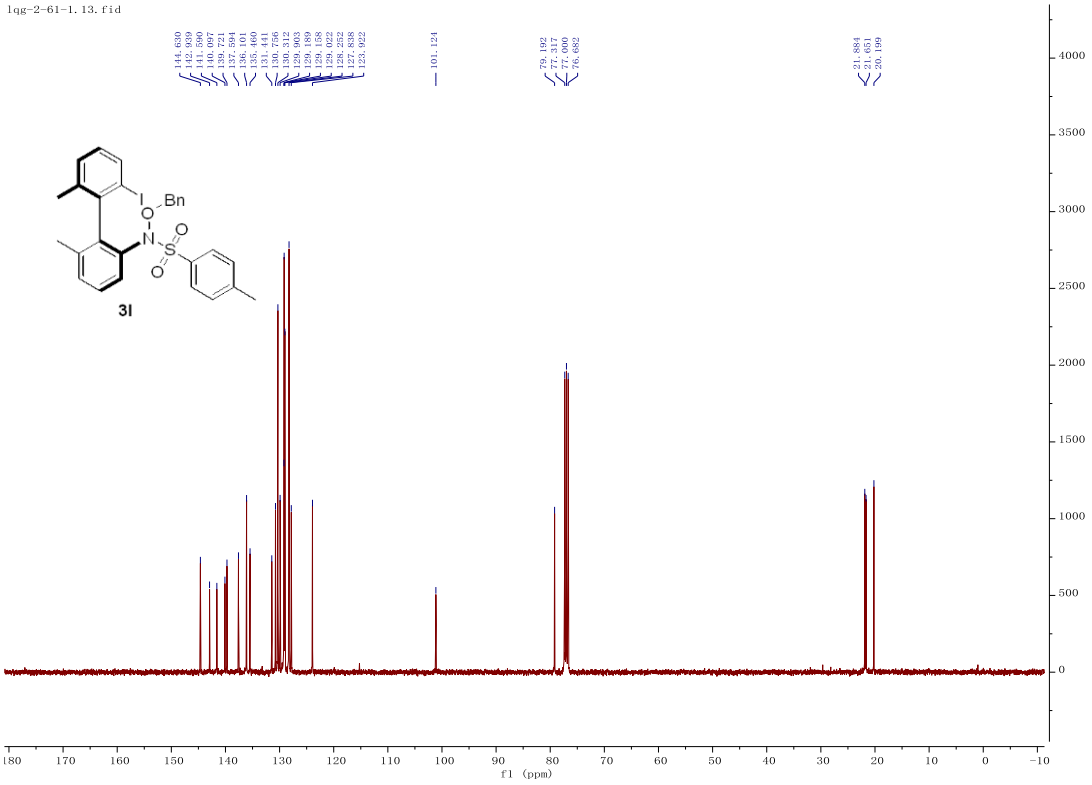

lqg-3-10-ee.1.fid
7.776
7.756
7.371
7.365
7.352
7.347
7.336
7.333
7.317
7.314
7.310
7.308
7.307
7.292
7.288
7.282
7.280
7.275
7.237
7.218
7.170
7.151
6.971
6.954
6.934
4.554
4.531
4.511
4.487
2.040
2.037
2.014
2.012
1.430
1.426
I
O
Bn
N
O
O
3j
1.02
7.24
1.04
1.04
1.04
2.00
3.04
3.07
9.11
f1 (ppm)

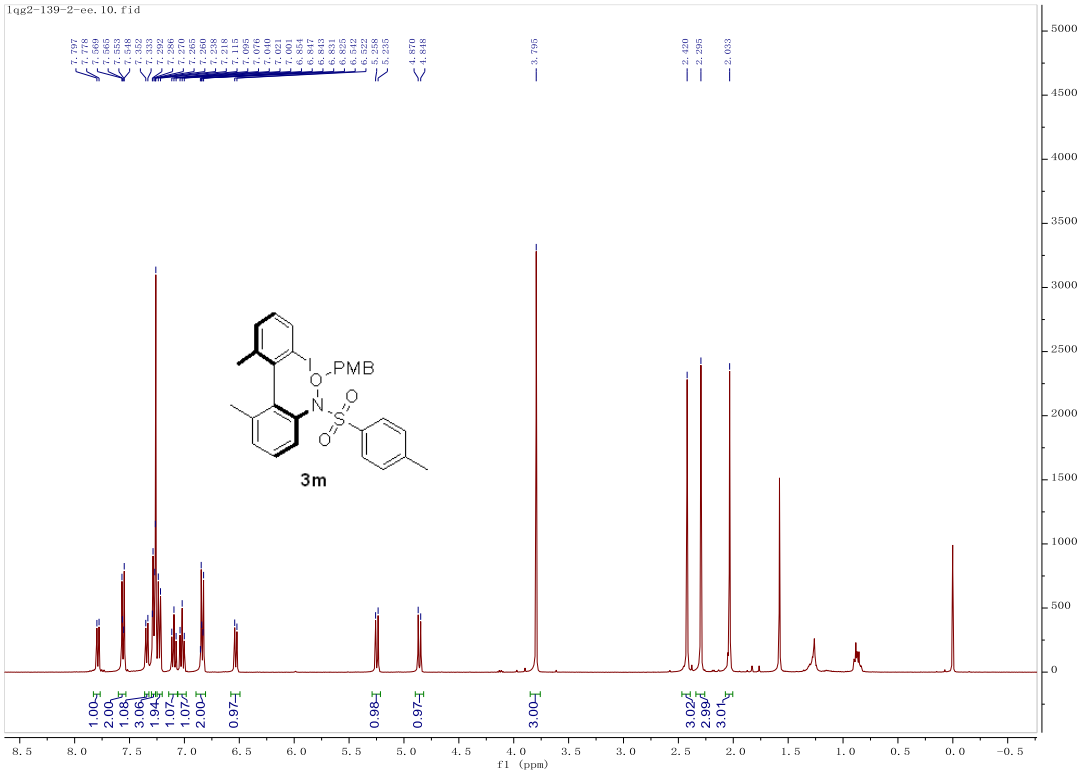

lqg-3-10-ee.2.fid
155.178
142.411
142.338
138.668
138.508
137.593
136.402
135.484
130.238
129.689
129.499
129.069
128.697
128.052
127.967
126.845
101.804
81.630
77.318
77.000
76.682
75.955
28.222
21.342
19.911
Bn
O
I
N
O
O
3j
f1 (ppm)

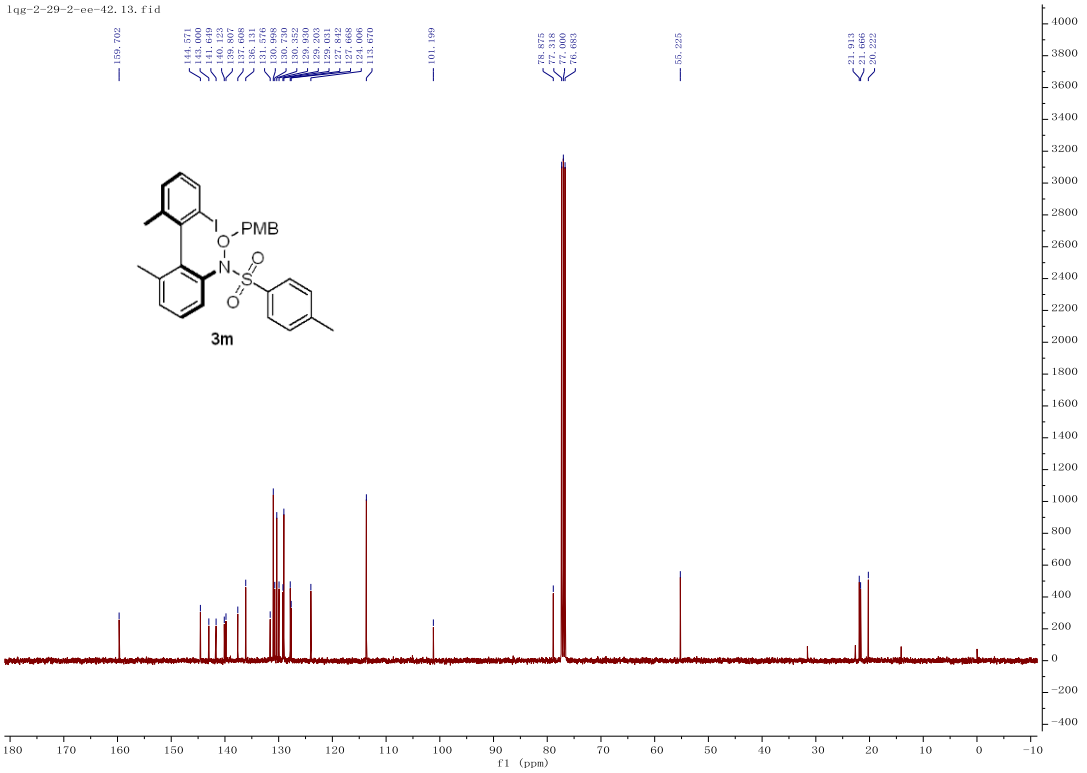

lqg2-139-1-ee.10.fid
7.773
7.754
7.751
7.610
7.605
7.600
7.589
7.584
7.326
7.307
7.304
7.288
7.274
7.269
7.267
7.260
7.152
7.132
7.112
7.001
7.001
6.982
6.616
6.614
6.612
6.595
6.592
6.589
5.873
5.863
5.858
5.847
5.841
5.820
5.815
5.804
5.799
5.789
5.269
5.261
5.257
5.226
5.222
5.218
5.214
5.187
5.184
5.182
5.179
5.177
5.161
5.158
5.156
5.153
5.151
4.722
4.719
4.716
4.708
4.704
4.701
4.695
4.692
4.688
4.680
4.677
4.673
4.416
4.412
4.409
4.406
4.398
4.393
4.388
4.385
4.382
4.372
4.369
4.366
4.258
4.256
2.011
O
I
N
O
O
S
Me
3k
1.00
1.99
4.11
1.00
1.00
0.98
0.96
1.98
1.00
1.00
3.00
2.97
2.99
f1 (ppm)

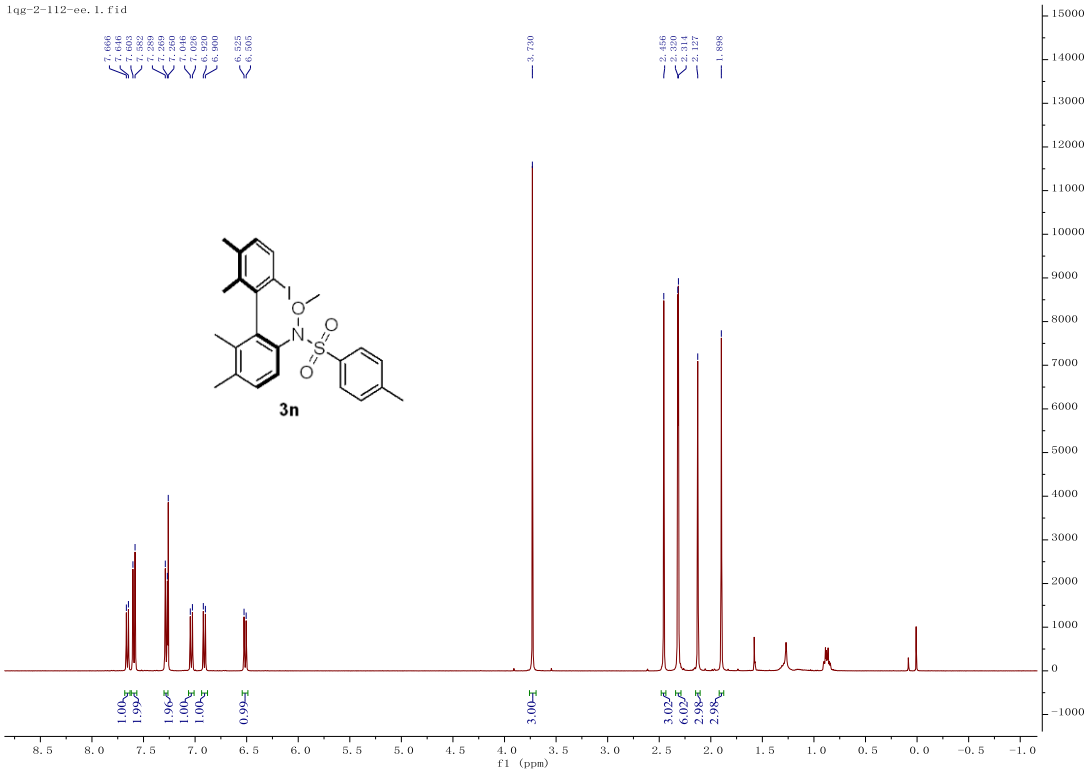

LQG-2-139-1-ee.10.fid
144.707
143.057
141.398
140.952
139.602
137.596
136.042
132.235
131.677
130.816
130.275
129.987
129.168
129.060
127.911
123.793
119.351
101.064
78.439
77.318
77.000
76.682
21.810
21.713
20.467
3k
Me
f1 (ppm)

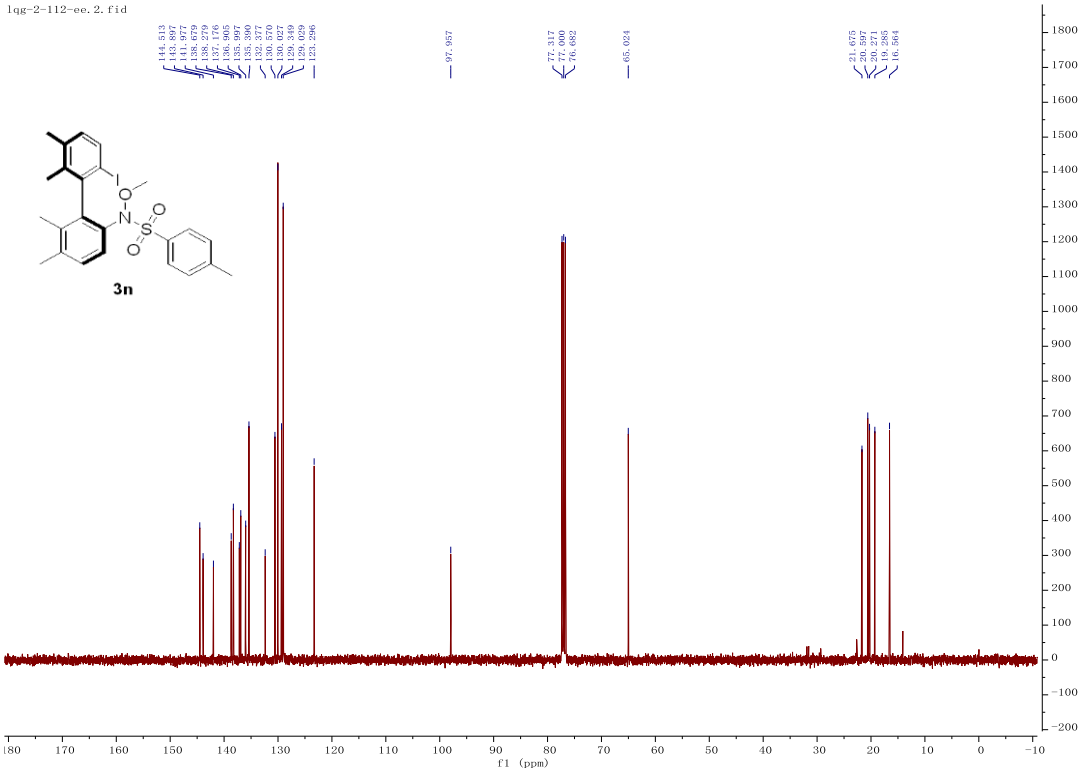

lqg-2-61.10.fid
7.814
7.795
7.589
7.568
7.366
7.347
7.330
7.326
7.322
7.311
7.303
7.290
7.260
7.249
7.229
7.142
7.122
7.103
7.053
7.034
7.015
6.595
6.575
5.355
5.332
4.961
4.937
2.430
2.311
2.049
3l
Bn
1.01
2.01
7.21
2.01
1.06
1.03
1.00
1.02
1.02
3.00
3.00
2.94
f1 (ppm)

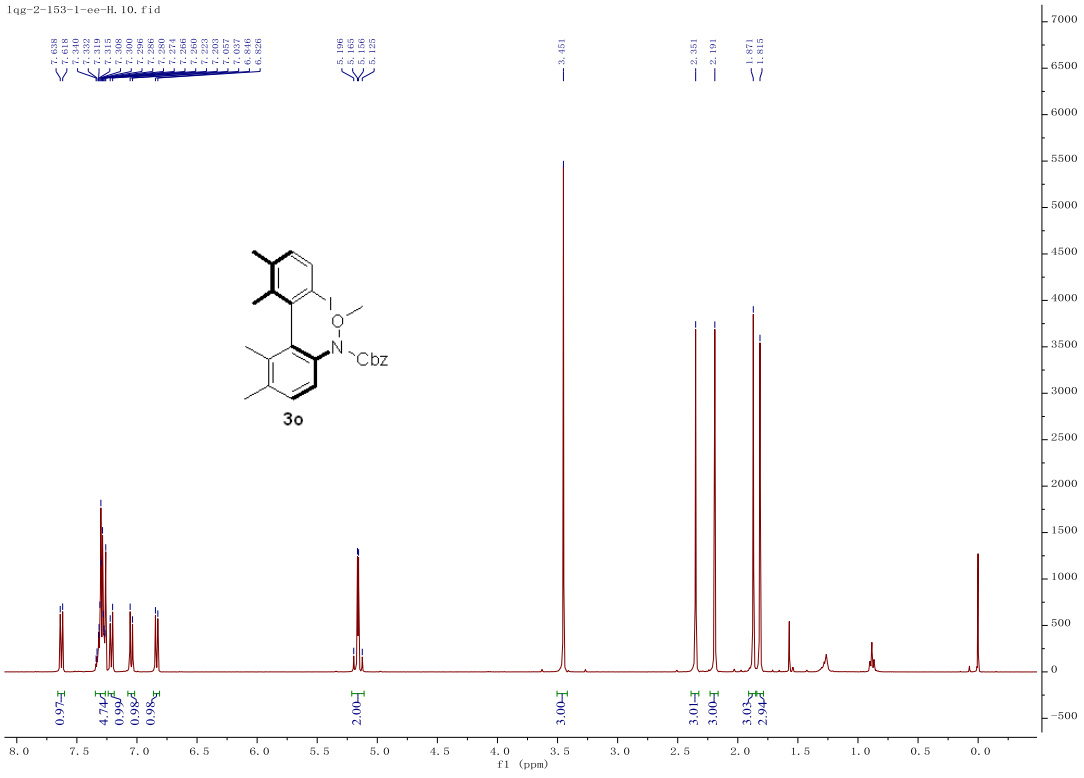

lqg-2-61-1.13.fid
144.630
142.939
141.590
140.667
139.721
137.594
136.101
135.460
131.141
130.756
130.312
129.993
129.189
129.158
129.022
128.252
127.858
123.922
101.124
79.192
77.317
77.000
76.682
21.884
21.651
20.199
Bn
O
N
S
O
O
3l
f1 (ppm)


lqg2-139-2-ee.10.fid
7.797
7.778
7.569
7.565
7.553
7.548
7.352
7.333
7.292
7.286
7.270
7.265
7.260
7.238
7.218
7.115
7.095
7.076
7.060
7.021
7.001
6.854
6.847
6.841
6.831
6.825
6.542
6.522
5.258
5.235
4.870
4.848
3.795
2.420
2.295
2.033
PMB
O
N
S
O
O
3m
1.00
2.00
1.08
3.06
1.94
1.07
1.07
2.00
0.97
0.98
0.97
3.00
3.02
2.99
3.01
f1 (ppm)


lqg-2-29-2-ee-42.13.fid
PMB
3m
f1 (ppm)


lqg-2-112-ee.1.fid
3n
f1 (ppm)


lqg-2-112-ee.2.fid
144.513
143.897
141.977
141.177
138.679
138.279
137.176
136.495
135.997
135.390
132.377
130.579
130.057
129.349
129.029
122.296
97.957
77.317
77.000
76.682
65.024
21.675
20.597
20.271
18.965
16.564
3n
f1 (ppm)


lqg-2-153-1-ee-H.10.fid
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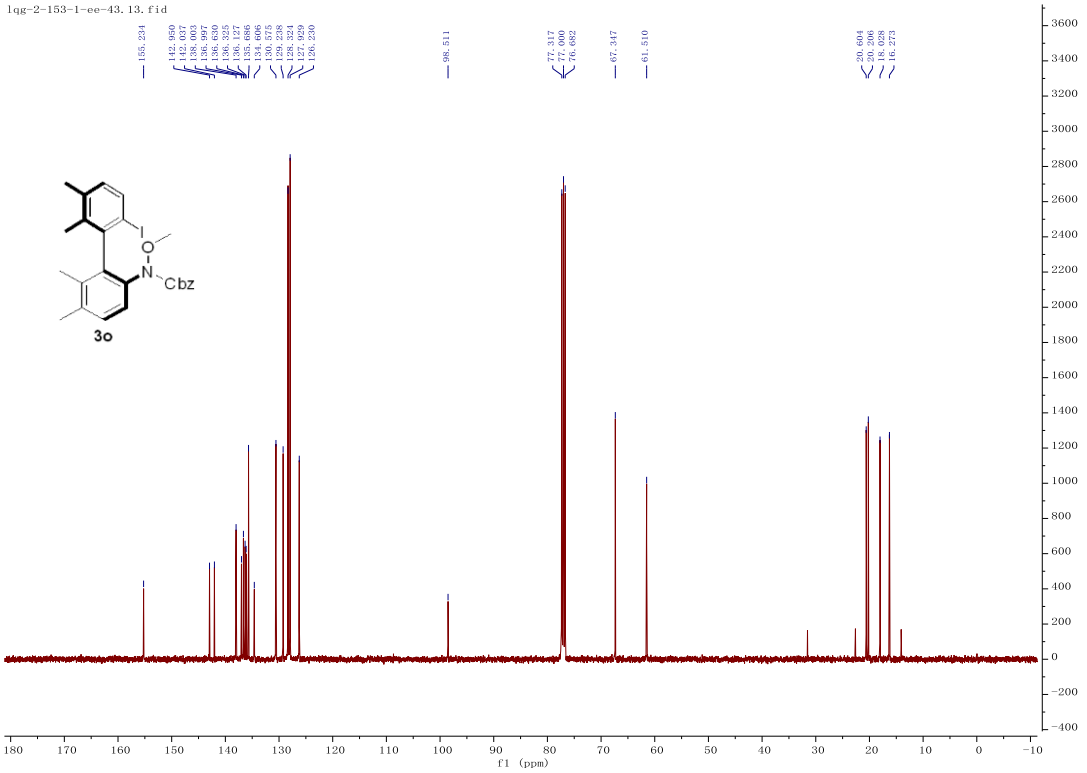
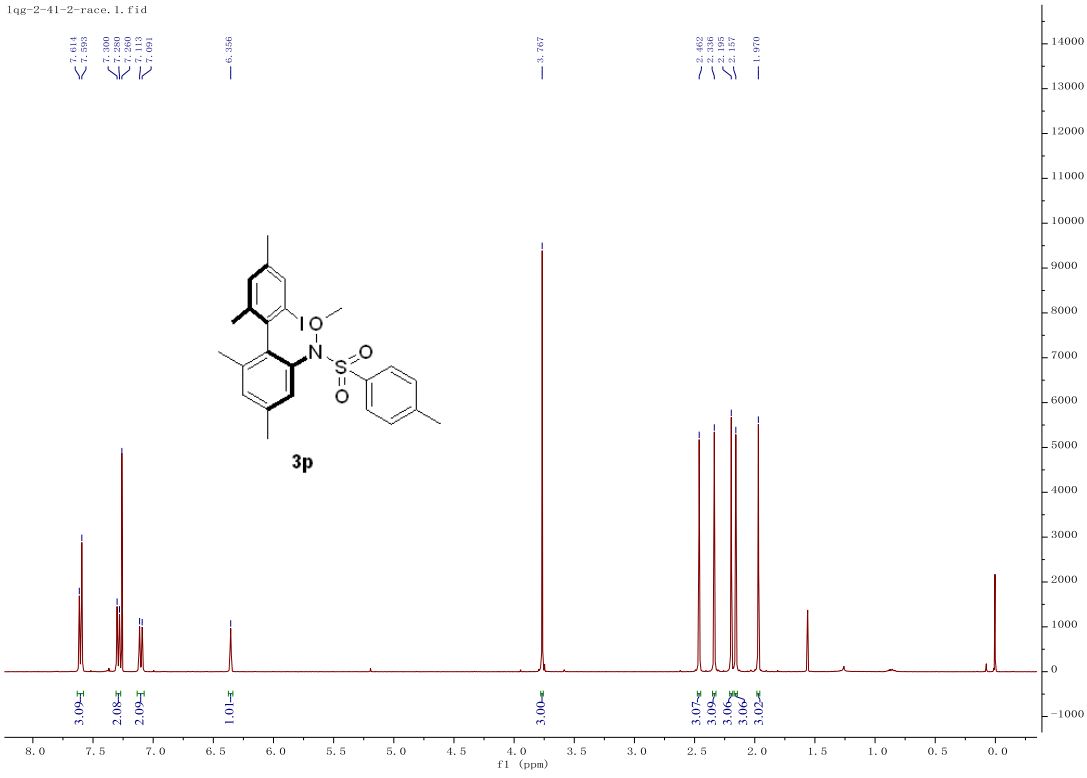
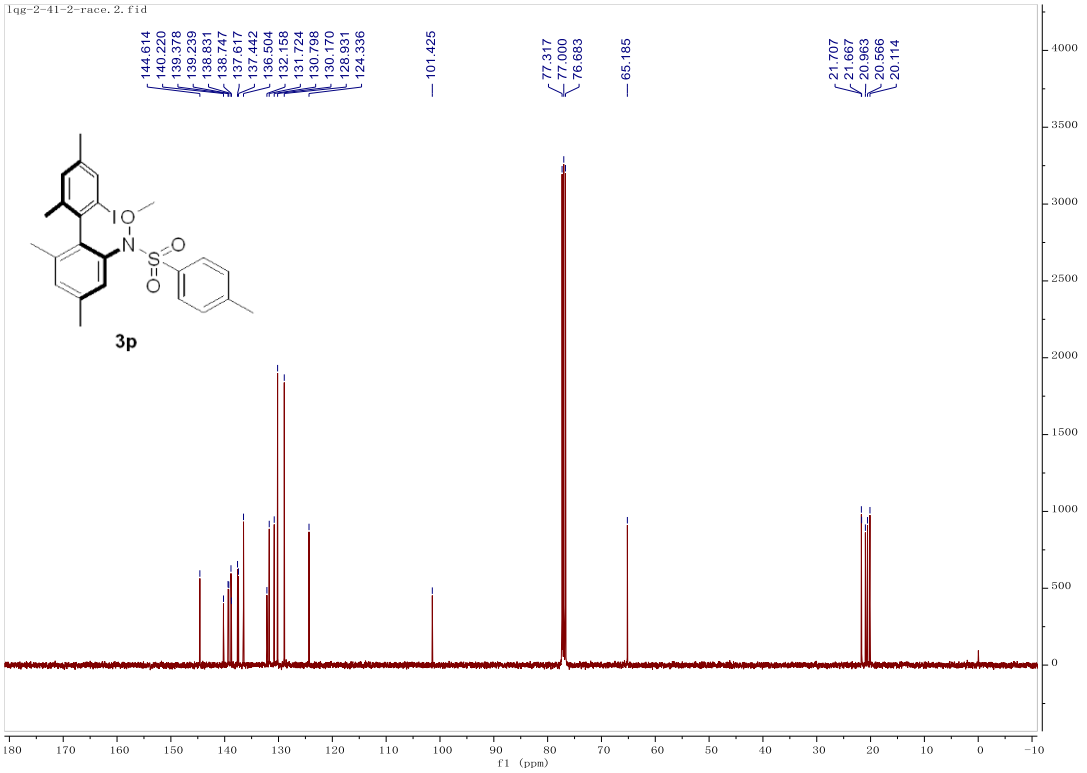
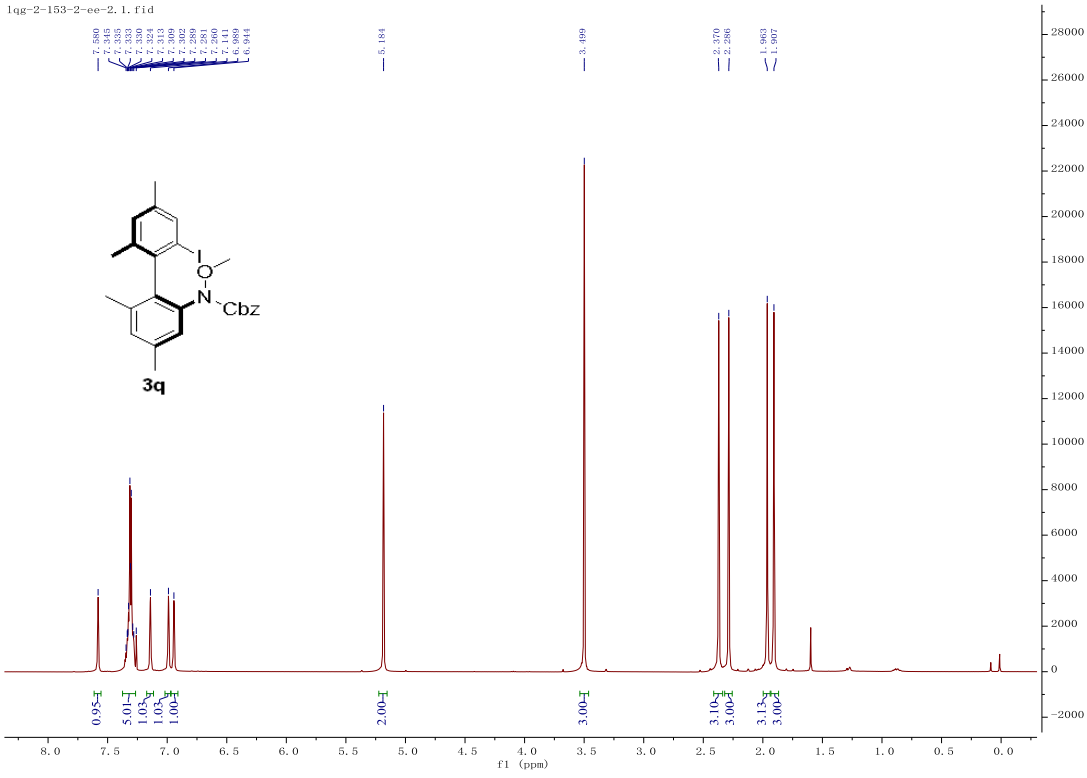
7.618
7.340
7.332
7.319
7.315
7.308
7.300
7.288
7.286
7.280
7.274
7.266
7.260
7.233
7.203
7.057
7.037
6.846
6.826
5.196
5.165
5.156
5.125
3.451
2.351
2.191
1.871
1.815
Cbz
3o
0.97
4.74
0.99
0.98
0.98
2.00
3.00
3.01
3.00
3.03
2.94
f1 (ppm)


lqg-2-153-1-ee-43.13.fid
155.234
142.959
141.657
138.003
136.997
136.630
136.535
136.127
135.686
134.606
130.575
129.238
128.324
127.929
126.230
98.511
77.317
77.000
76.682
67.347
61.510
20.604
20.306
18.028
16.273
Cbz
3o
f1 (ppm)


lqg-2-41-2-race.1.fid
7.614
7.593
7.300
7.280
7.260
7.113
7.091
6.356
3.767
2.462
2.336
2.195
2.157
1.970
3.09
2.08
2.09
1.01
3.00
3.07
3.09
3.06
3.06
3.02
3p
f1 (ppm)


lqg-2-41-2-race.2.fid
144.614
140.220
139.378
139.239
138.831
138.747
137.617
137.442
136.504
132.158
131.724
130.798
130.170
128.931
124.336
101.425
77.317
77.000
76.683
65.185
21.707
21.667
20.963
20.566
20.114
3p
f1 (ppm)


lqg-2-153-2-ee-2.1.fid
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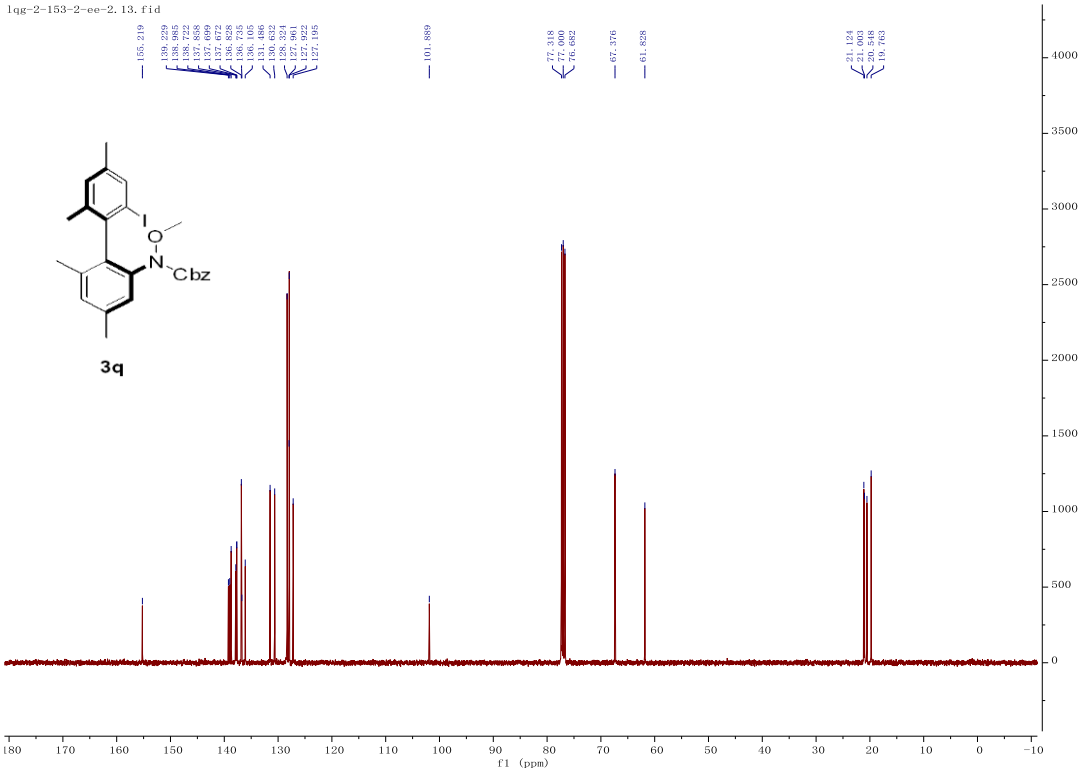
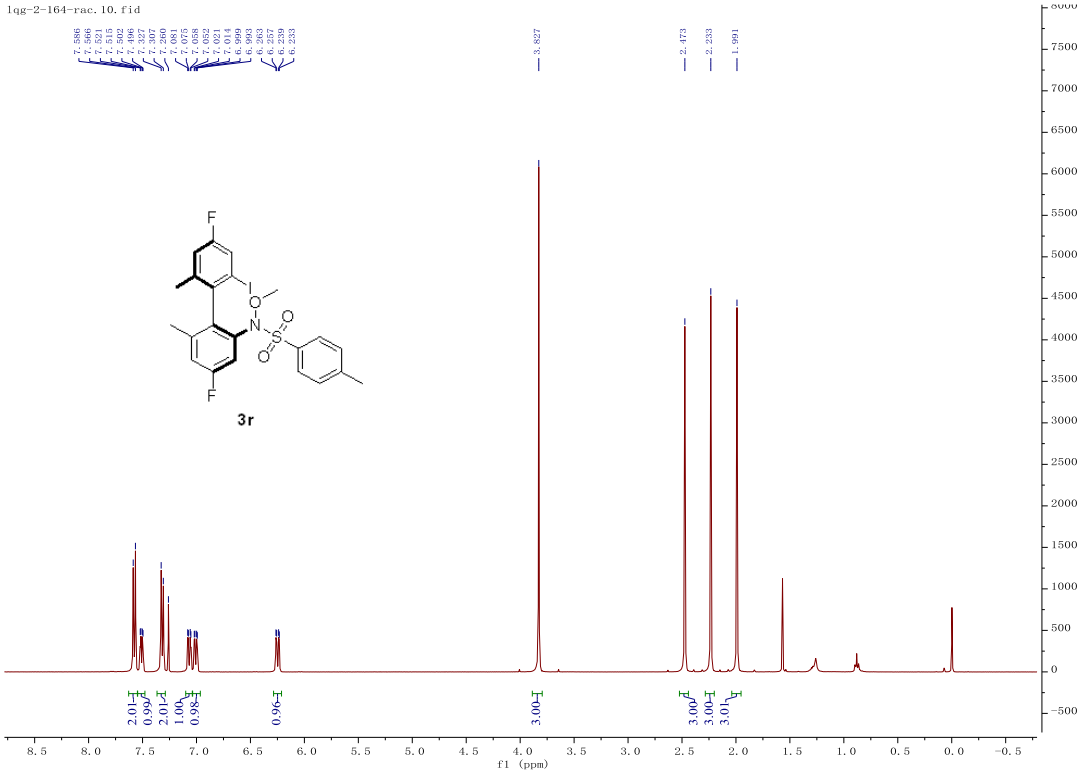
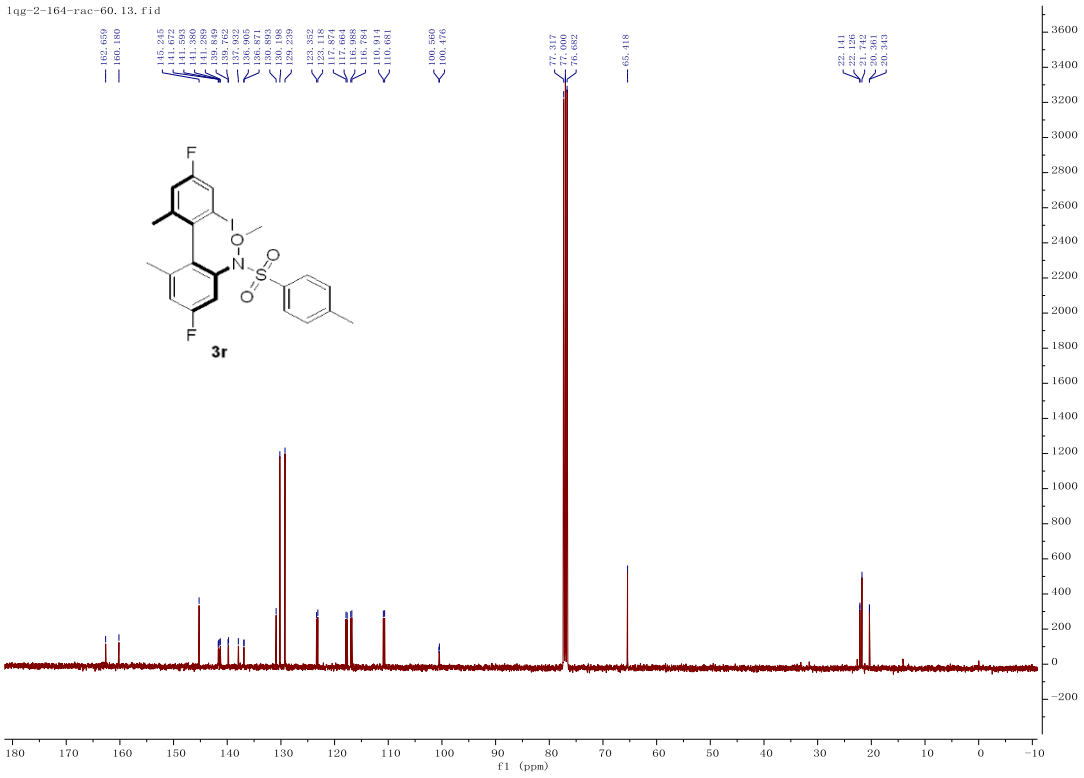
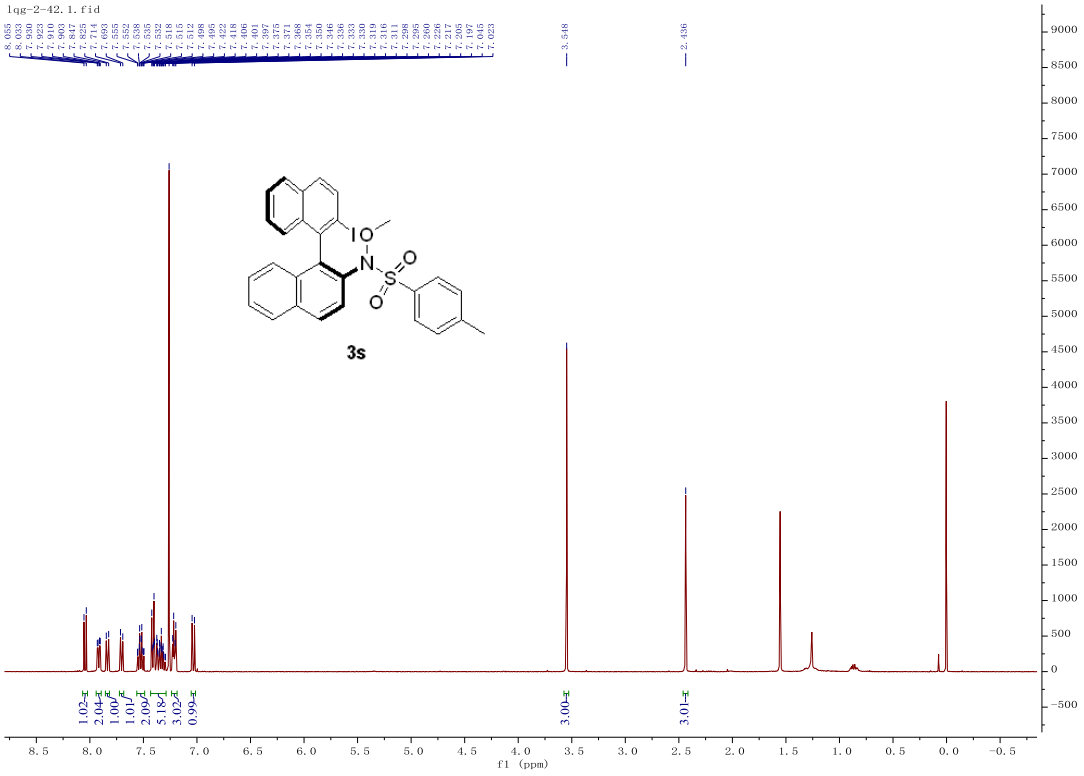
7.345
7.335
7.333
7.330
7.324
7.313
7.309
7.302
7.289
7.281
7.260
7.141
6.989
6.944
5.184
3.499
2.370
2.286
1.963
1.907
0.95
5.01
1.03
1.03
1.00
2.00
3.00
3.10
3.00
3.13
3.00
3q
Cbz
f1 (ppm)

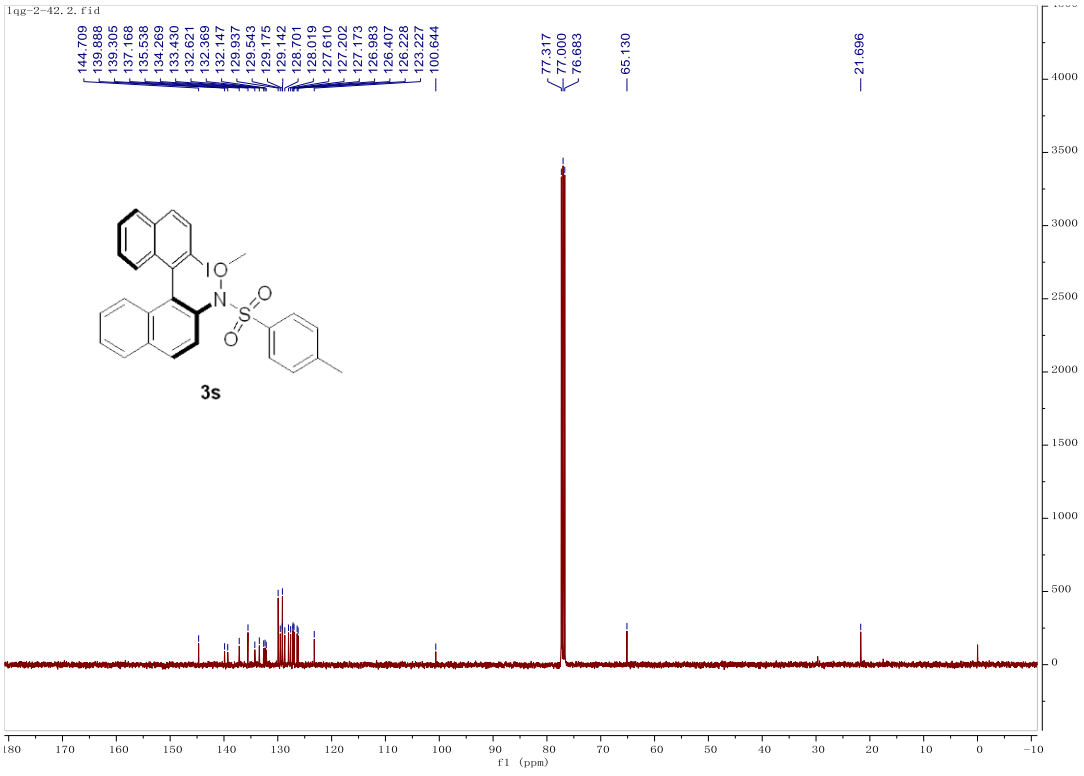

lqg-2-153-2-ee-2.13.fid
155.219
139.229
138.985
138.722
137.868
137.699
137.672
136.828
136.735
136.105
131.486
130.632
129.354
128.041
127.922
127.195
101.889
77.318
77.000
76.682
67.376
61.828
21.124
21.003
20.548
18.783
Cbz
3q
f1 (ppm)

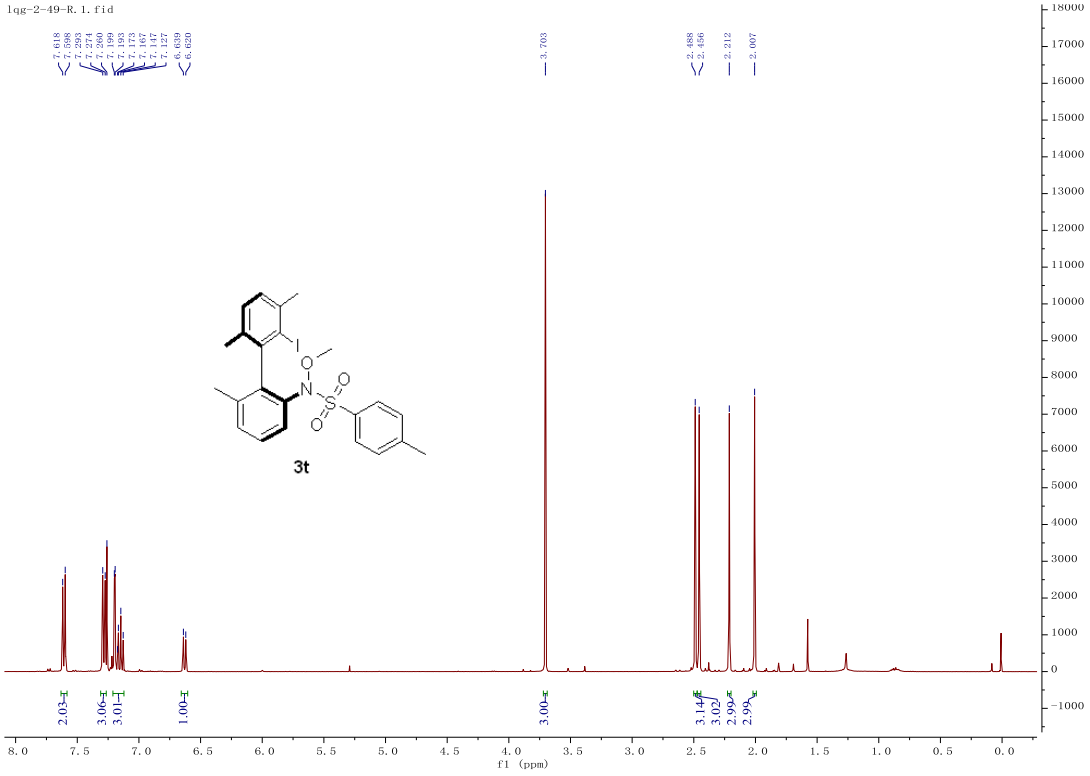

lqg-2-164-rac.10.fid
7.586
7.566
7.521
7.515
7.502
7.496
7.327
7.307
7.260
7.081
7.075
7.058
7.052
7.021
7.014
6.969
6.963
6.257
6.239
6.233
3.827
2.473
2.233
1.991
F
F
3r
2.01
0.99
2.01
1.00
0.98
0.96
3.00
3.00
3.00
3.01
f1 (ppm)

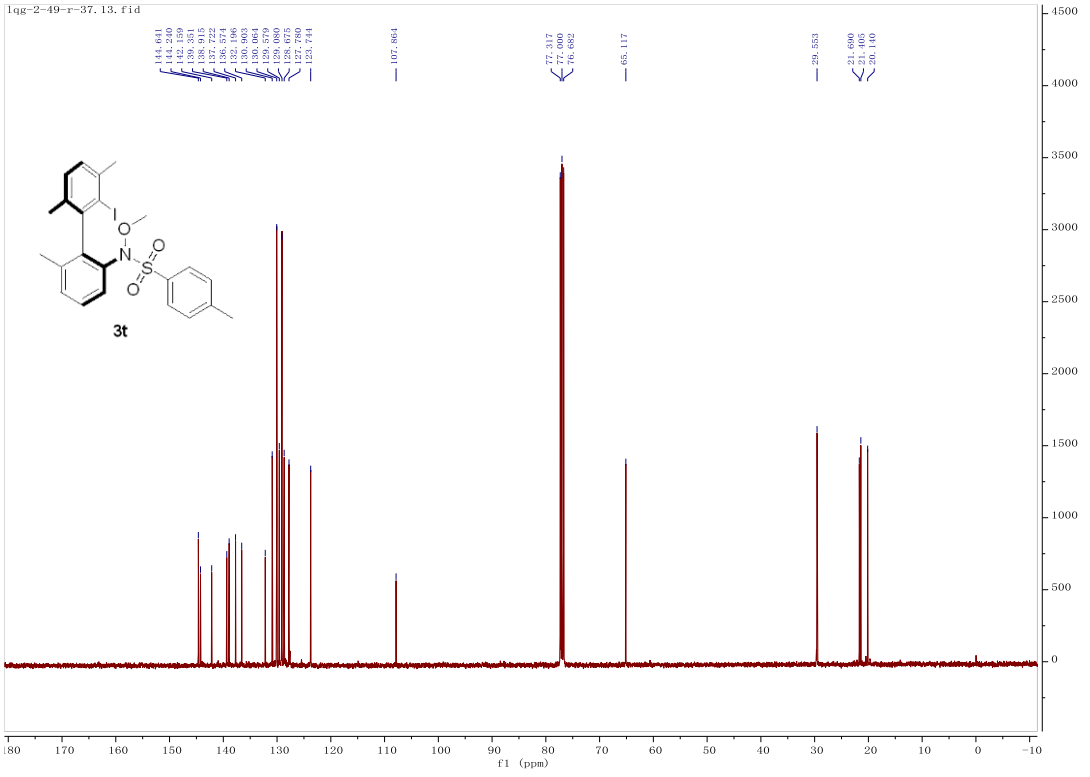

lqg-2-164-rac.11.fid
-112.490
-112.515
-112.539
-114.080
-114.092
-114.104
-114.126
F
I
O
N
S
O
O
F
3r
f1 (ppm)

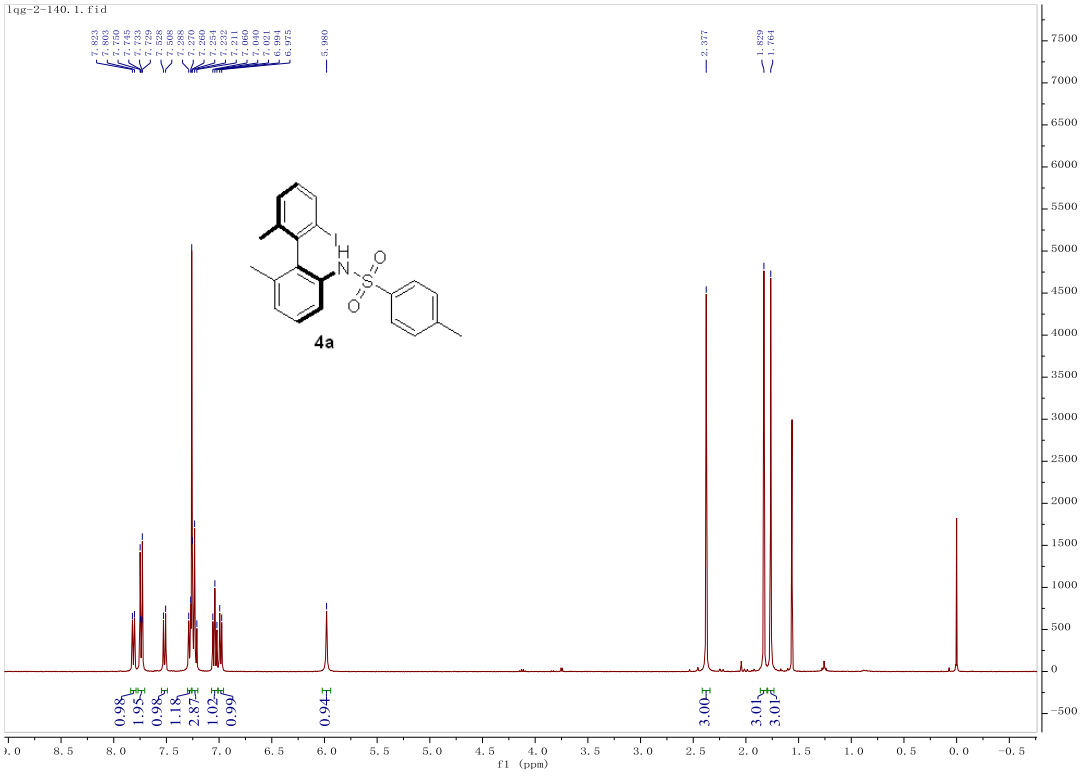

lqg-2-164-rac-60.13.fid
162.659
160.180
145.245
141.672
141.585
141.380
141.289
139.849
139.752
137.932
136.905
136.871
130.804
130.198
129.239
123.352
123.118
117.874
117.664
116.888
116.704
110.914
110.681
100.569
100.476
77.317
77.000
76.682
65.418
22.141
22.126
21.742
20.361
20.343
F
I
O
N
S
O
O
F
3r
f1 (ppm)

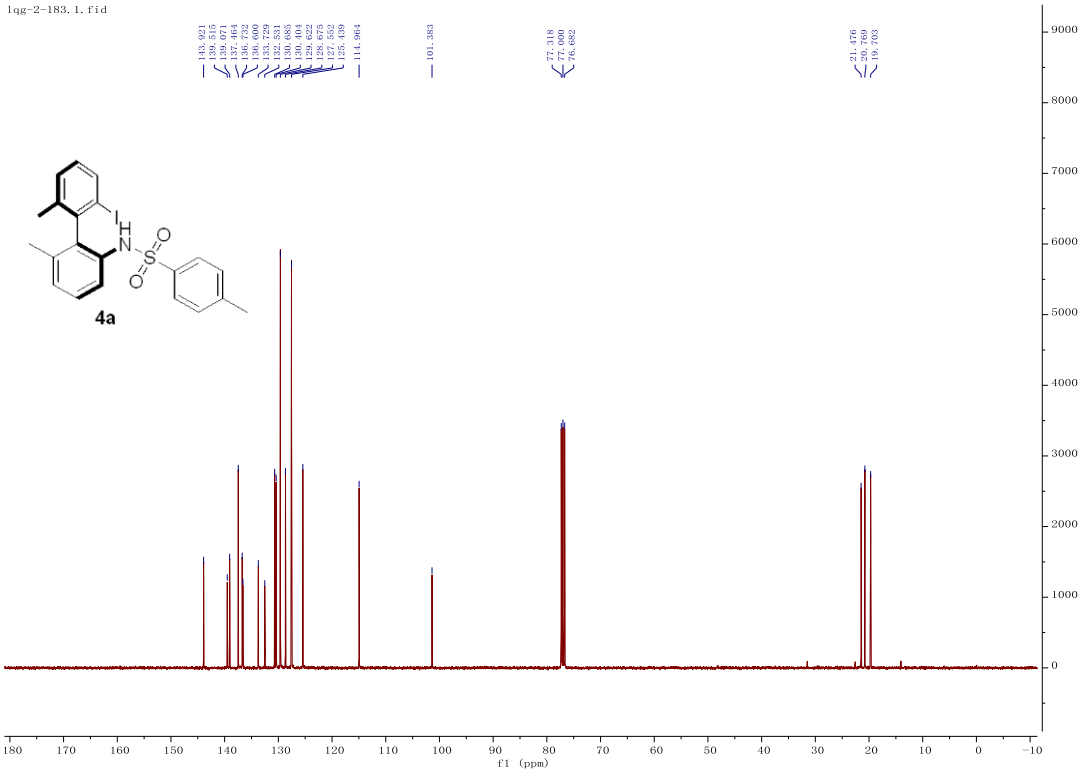

lqg-2-42.1.fid
3s
f1 (ppm)

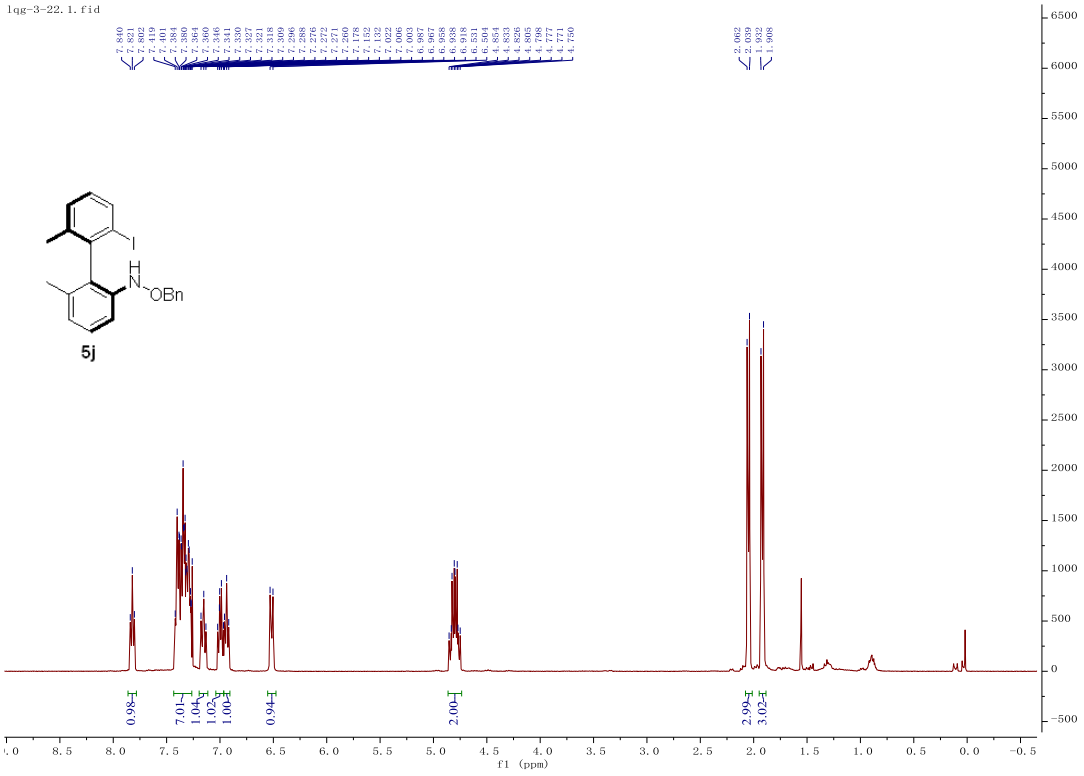

lqg-2-42.2.fid
3s
f1 (ppm)

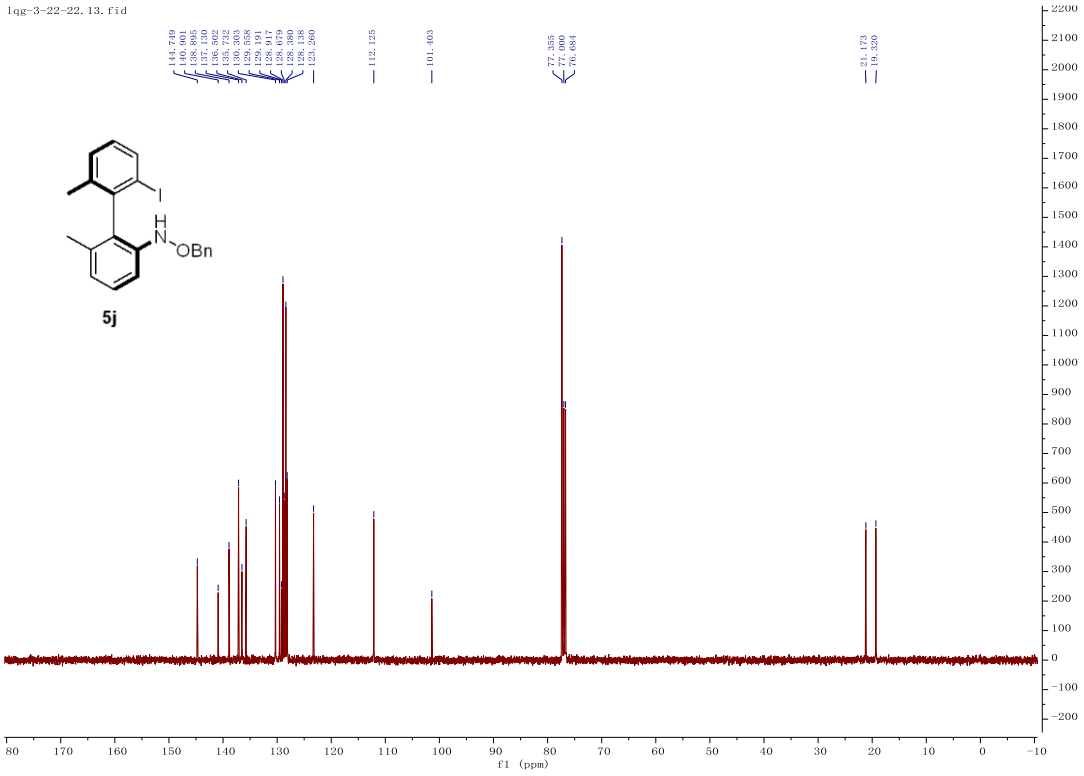

lqg-2-49-R.1.fid
7.618
7.598
7.293
7.274
7.260
7.199
7.193
7.173
7.167
7.147
7.127
6.639
6.620
3.703
2.488
2.466
2.212
2.007
3t
2.03
3.06
3.01
1.00
3.00
3.14
3.03
2.99
2.99
f1 (ppm)

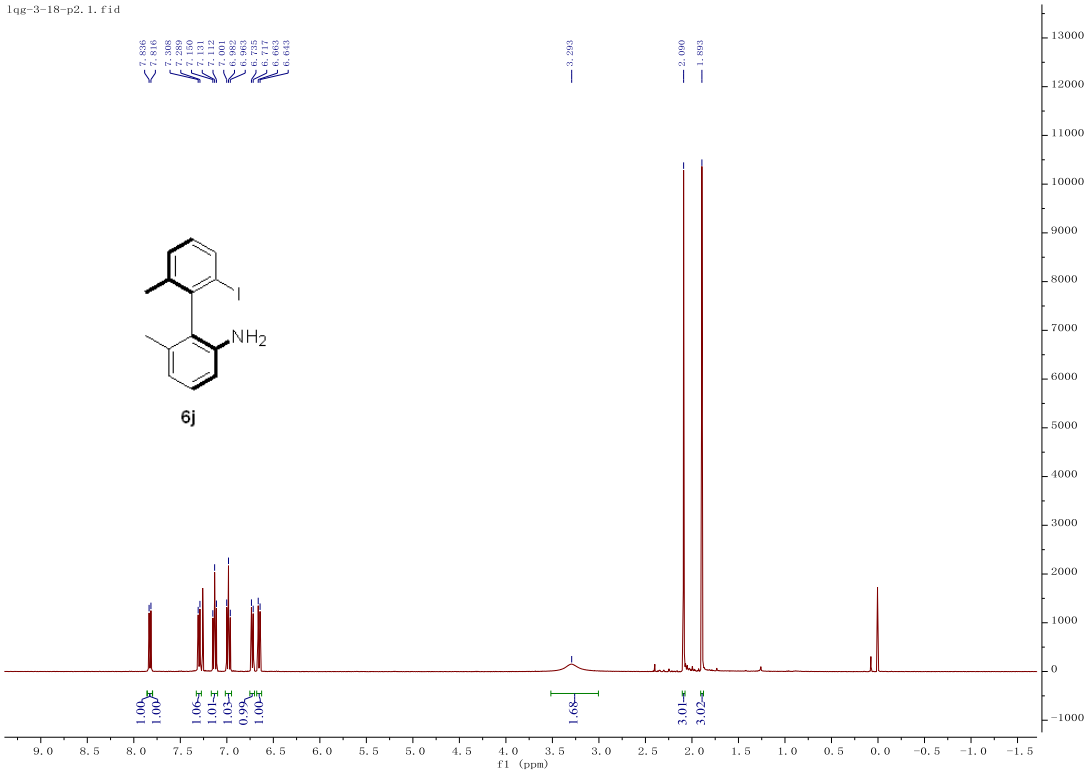

lqg-2-49-r-37.13.fid
144.641
144.240
142.159
139.351
138.915
138.722
136.574
132.196
130.903
130.664
129.579
129.089
128.675
127.789
126.744
107.864
77.317
77.000
76.682
65.117
29.553
21.690
21.405
20.140
3t
f1 (ppm)

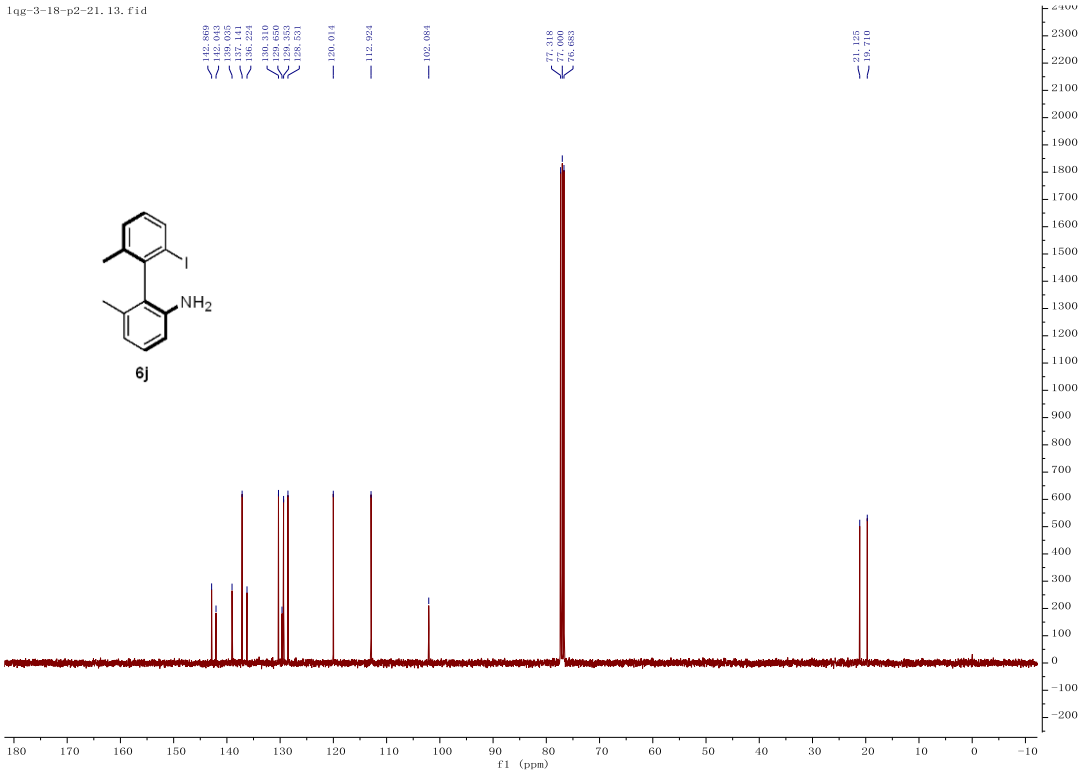

lqg-2-140.1.fid
7.823
7.803
7.790
7.745
7.733
7.729
7.628
7.606
7.288
7.270
7.260
7.254
7.232
7.211
7.060
7.040
7.021
6.994
6.975
5.980
2.377
1.829
1.764
4a
0.98
1.95
0.98
1.18
2.87
1.02
0.99
0.94
3.00
3.01
3.01
f1 (ppm)

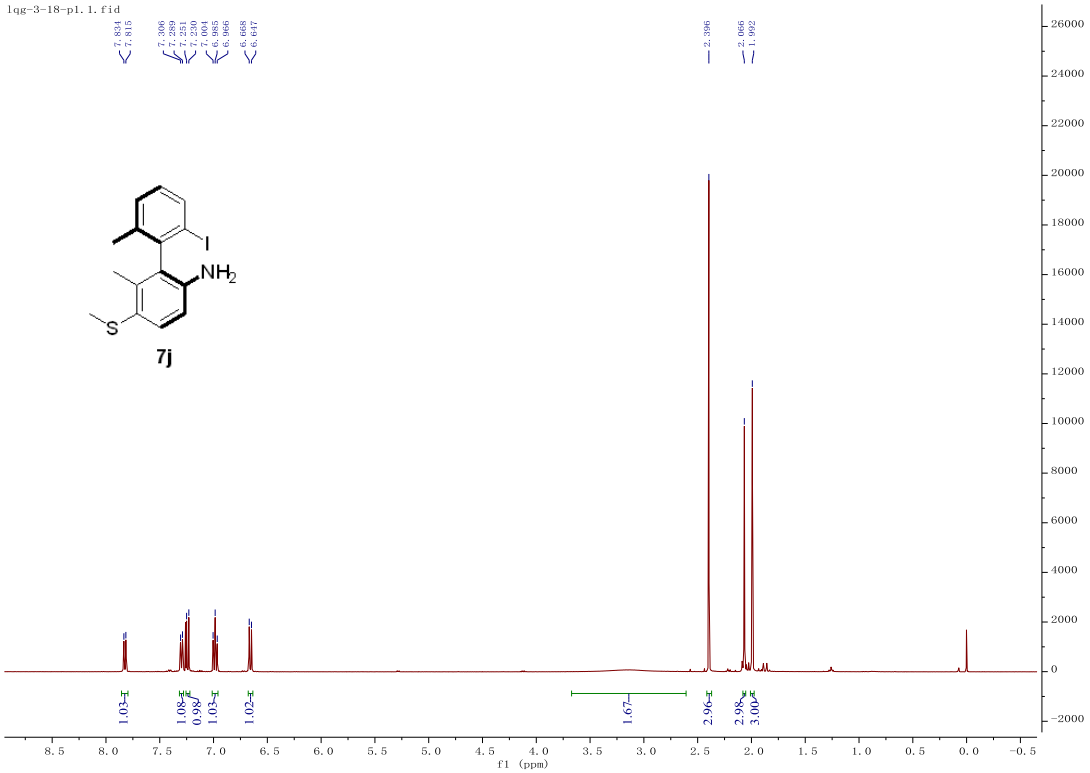

lqg-2-183.1.fid
143.921
139.575
139.071
137.464
136.732
136.600
135.291
132.531
130.685
130.404
129.822
128.675
127.552
125.439
114.964
101.383
77.318
77.000
76.682
21.476
20.769
19.703
4a
f1 (ppm)

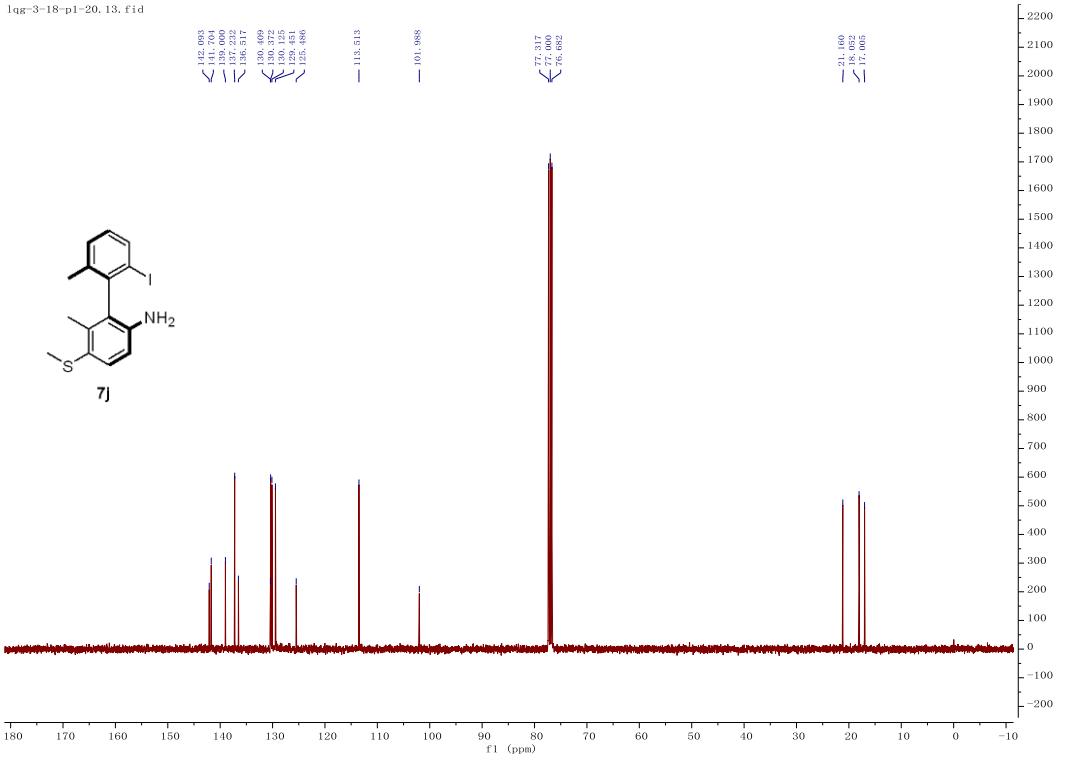

lqg-3-22.1.fid
5j
f1 (ppm)

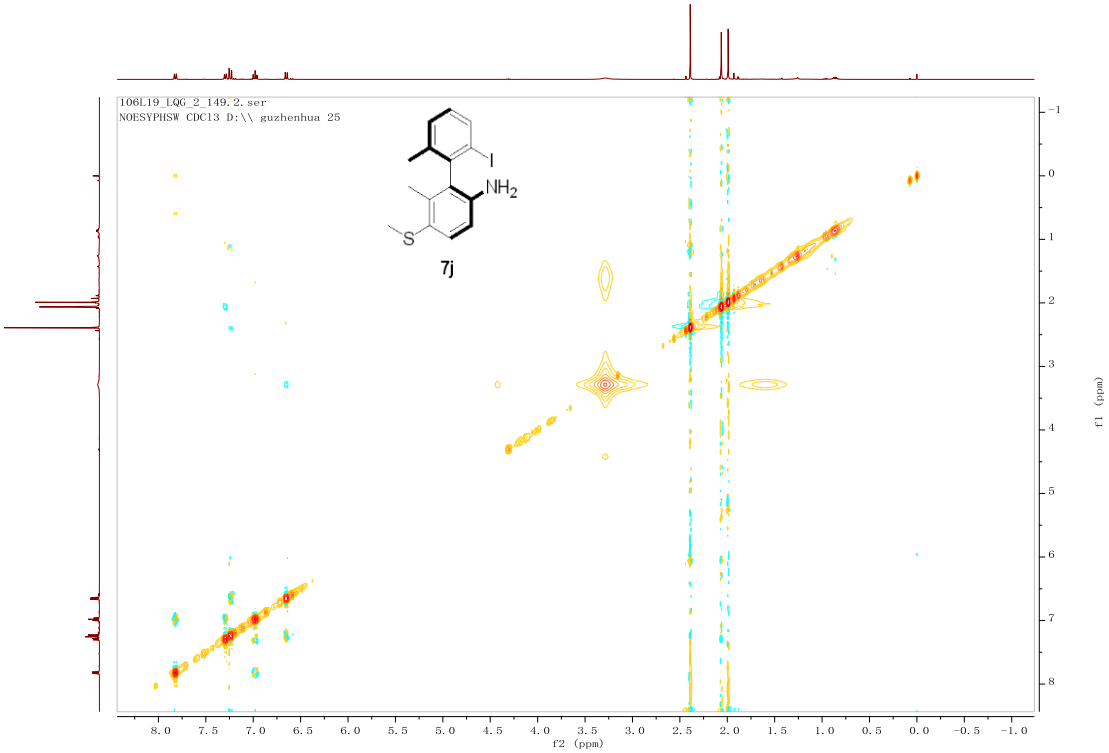

lqg-3-22-22.13.fid
5j
f1 (ppm)


lqg-3-18-p2.1.fid
7.836
7.816
7.308
7.289
7.150
7.131
7.122
7.001
6.982
6.963
6.735
6.717
6.663
6.643
3.293
2.090
1.893
I
NH2
6j
1.00
1.00
1.06
1.01
1.03
0.99
1.00
1.68
3.01
3.02
f1 (ppm)


lqg-3-18-p2-21.13.fid
142.869
142.043
139.655
137.141
136.224
130.310
129.650
129.353
128.831
120.014
112.924
102.084
77.318
77.000
76.683
21.125
19.710
I
NH2
6j
f1 (ppm)


lqg-3-18-p1.1.fid
7.834
7.815
7.306
7.289
7.253
7.230
7.004
6.985
6.966
6.668
6.647
2.396
2.066
1.992
I
NH2
S
7j
1.03
1.08
0.98
1.03
1.02
1.67
2.96
2.98
3.00
f1 (ppm)

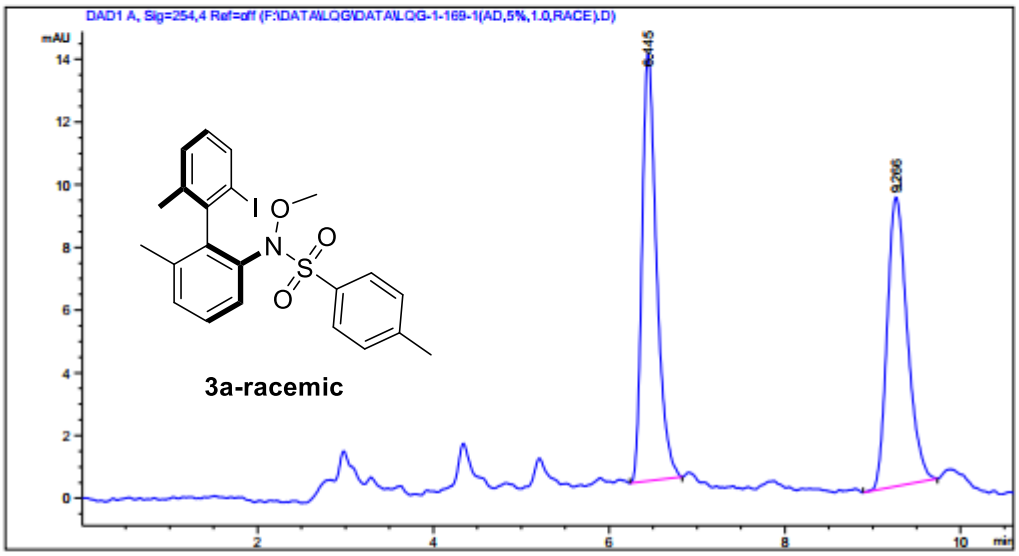

lqg-3-18-p1-20.13.fid
142.093
141.704
139.000
137.232
136.517
130.469
130.372
130.215
129.451
125.486
113.513
101.988
77.317
77.000
76.682
21.160
18.052
17.005
I
NH2
S
7j
f1 (ppm)


106L19_LQG_2_149.2.ser
NOESYPHSW CDC13 D:\\ guzhenhua 25
I
NH2
S
7j
f2 (ppm)
f1 (ppm)

Copies of HPLC Traces

Data File F:\DATA\LQG\DATA\LQG-1-169-1(AD,5%,1.0,RACE).D
Sample Name: LQG-1-169-1(AD,5%,1.0,RACE)

```
==================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 07/12/2018 22:23:32
                                               Inj Volume : No inj
Acq. Method     : F:\METHOD\ZK.M
Last changed    : 07/12/2018 22:16:34 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```

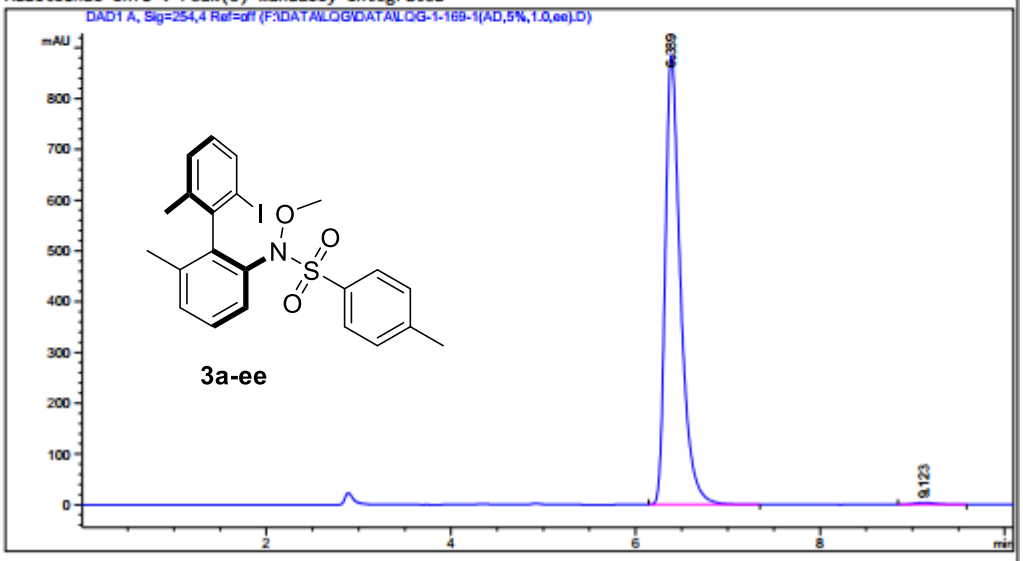



```
==================================================================
                        Area Percent Report
==================================================================

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.445 BB    0.1759  156.41916   13.62501  51.4834
   2   9.266 BB    0.2406  147.40503    9.21721  48.5166

Totals :                   303.82419   22.84222
```

LC1260 27/06/2019 03:42:59 SYSTEM Page 1 of 2

Data File F:\DATA\LQG\DATA\LQG-1-169-1(AD,5%,1.0,ee).D
Sample Name: LQG-1-169-1(AD,5%,1.0,ee)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 07/12/2018 22:37:35
Acq. Method : ZK.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



3a-ee

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.389	BB	0.1715	1.01269e4	885.00714	99.4163
2	9.123	BB	0.2299	59.45290	3.81313	0.5837
Totals :				1.01863e4	888.82027	

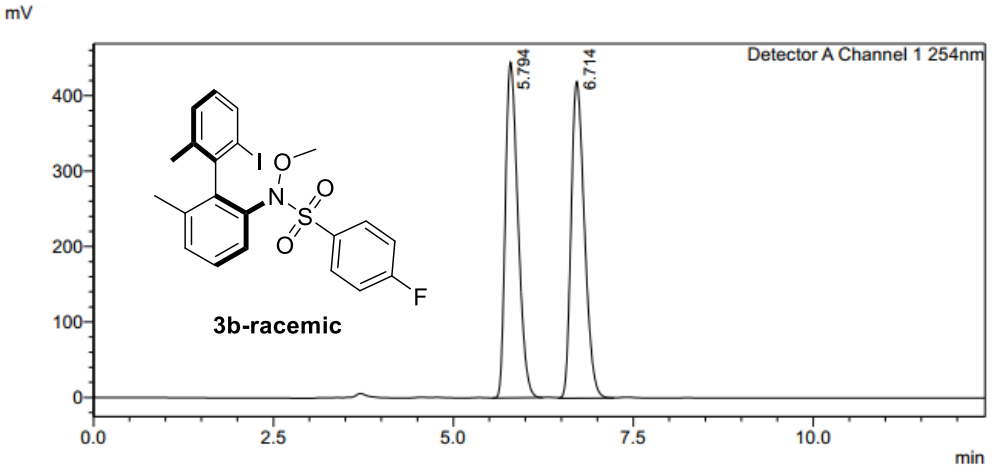
*** End of Report ***

LC1260 27/06/2019 03:41:19 SYSTEM Page 1 of 1

<Sample Information>

Sample Name	: lqg-2-104-1(AD,5%,1.0,race)		
Sample ID	:		
Data Filename	: lqg-2-104-1(AD,5%,1.0,race).lcd		
Method Filename	: LCY single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/2/2019 4:16:48 AM	Acquired by	: System Administrator
Date Processed	: 5/2/2019 4:29:12 AM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

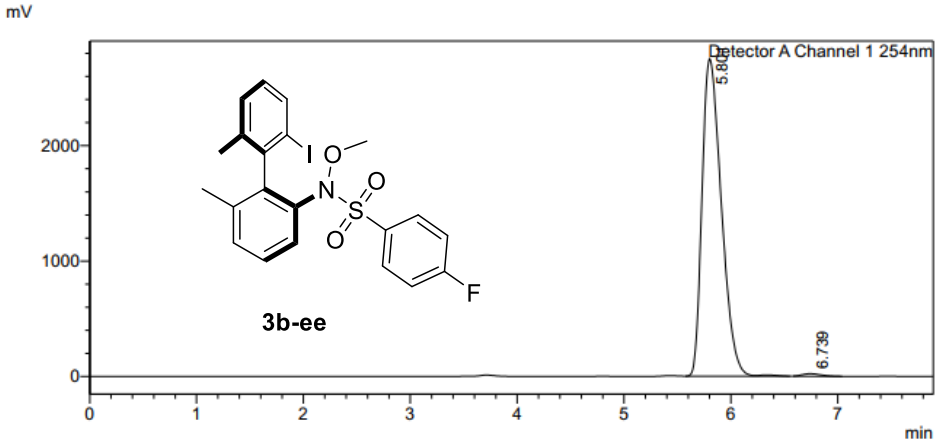
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.794	5392930	444498	49.883		M	
2	6.714	5418155	419538	50.117			
Total		10811085	864036				

<Sample Information>

Sample Name	: lqg-2-104-1(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: lqg-2-104-1(AD,5%,1.0,ee).lcd		
Method Filename	: LCY single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/2/2019 4:31:45 AM	Acquired by	: System Administrator
Date Processed	: 5/2/2019 4:39:39 AM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

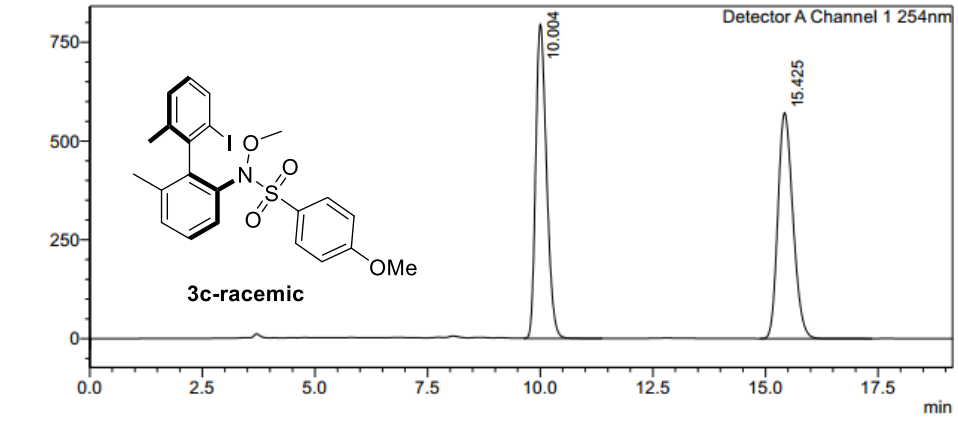
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.801	34578484	2749713	99.252		M	
2	6.739	260493	21855	0.748		M	
Total		34838977	2771569				

<Sample Information>

Sample Name	: lqg-2-110(AD,5%,1.0,race)		
Sample ID	:		
Data Filename	: lqg-2-110(AD,5%,1.0,race).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/3/2019 12:34:05 AM	Acquired by	: System Administrator
Date Processed	: 5/3/2019 12:53:14 AM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

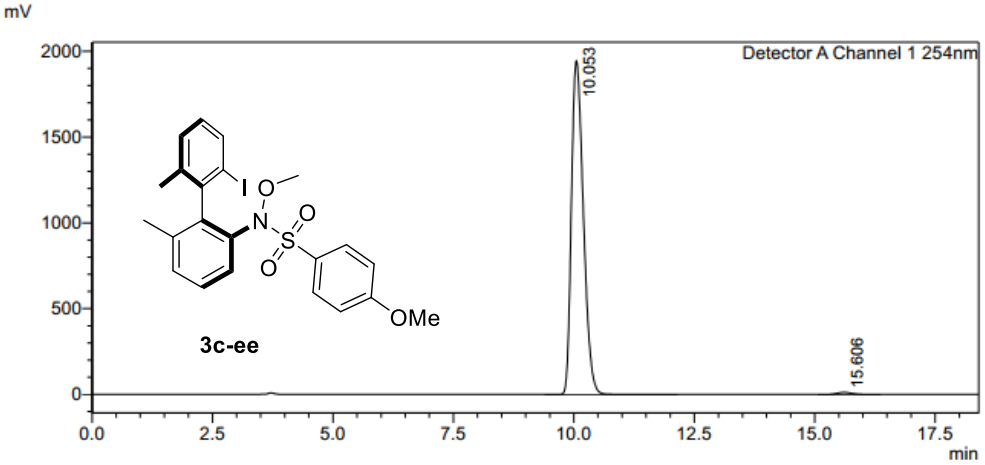
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.004	13295279	794421	49.983		S	
2	15.425	13304399	571315	50.017			
Total		26599678	1365735				

<Sample Information>

Sample Name	: lqg-2-110(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: lqg-2-110(AD,5%,1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/3/2019 12:59:48 AM	Acquired by	: System Administrator
Date Processed	: 5/3/2019 1:18:12 AM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

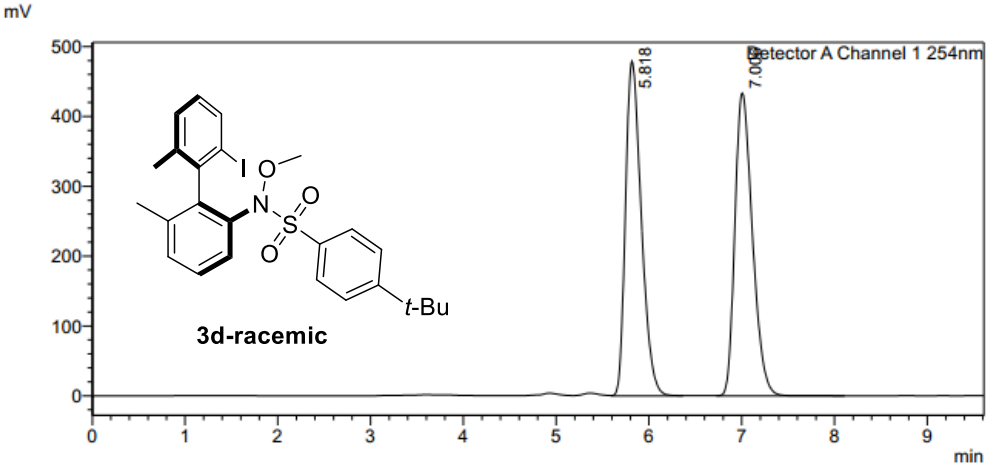
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.053	33550443	1944005	99.227		S M	
2	15.606	261243	11496	0.773			
Total		33811685	1955501				

<Sample Information>

Sample Name	: LQG-2-51-2(AD,5%,1.0,race)		
Sample ID	:		
Data Filename	: LQG-2-51-2(AD,5%,1.0,race).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/16/2019 10:34:45 PM	Acquired by	: System Administrator
Date Processed	: 4/16/2019 10:44:23 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

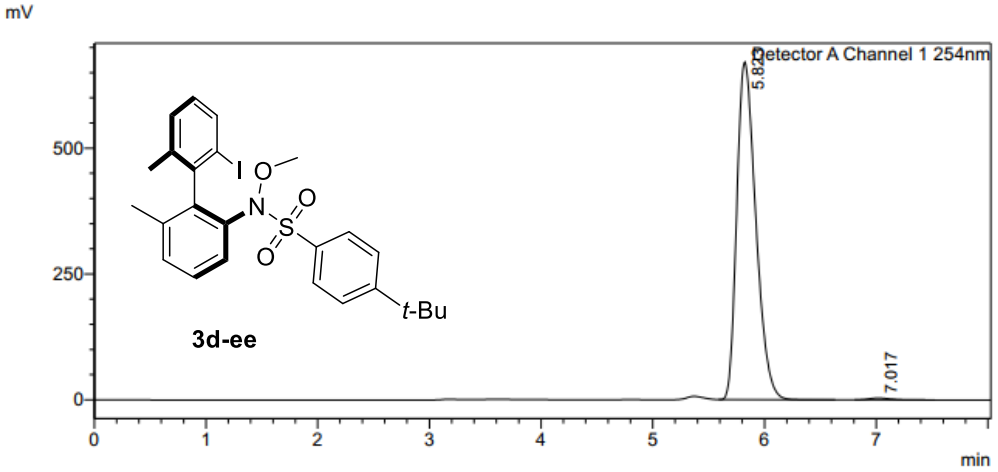
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.818	5684225	479095	49.940			
2	7.006	5697828	433539	50.060			
Total		11382053	912634				

<Sample Information>

Sample Name	: LQG-2-51-2 (2)(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: LQG-2-51-2 (2)(AD,5%,1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/16/2019 10:49:41 PM	Acquired by	: System Administrator
Date Processed	: 4/16/2019 10:57:44 PM	Processed by	: System Administrator

<Chromatogram>



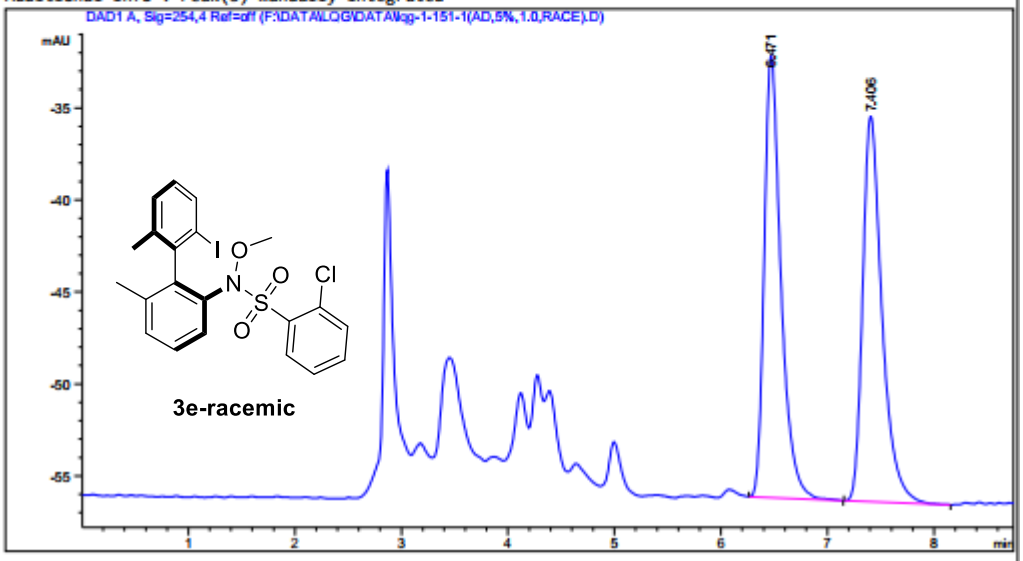
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.823	7953873	670757	99.422		M	
2	7.017	46271	3632	0.578		M	
Total		8000144	674389				

Data File F:\DATA\LQG\DATA\lqg-1-151-1(AD,5%,1.0,RACE).D
Sample Name: lqg-1-151-1(AD,5%,1.0,RACE)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 09/10/2018 21:23:36
Acq. Method : rxdeng.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

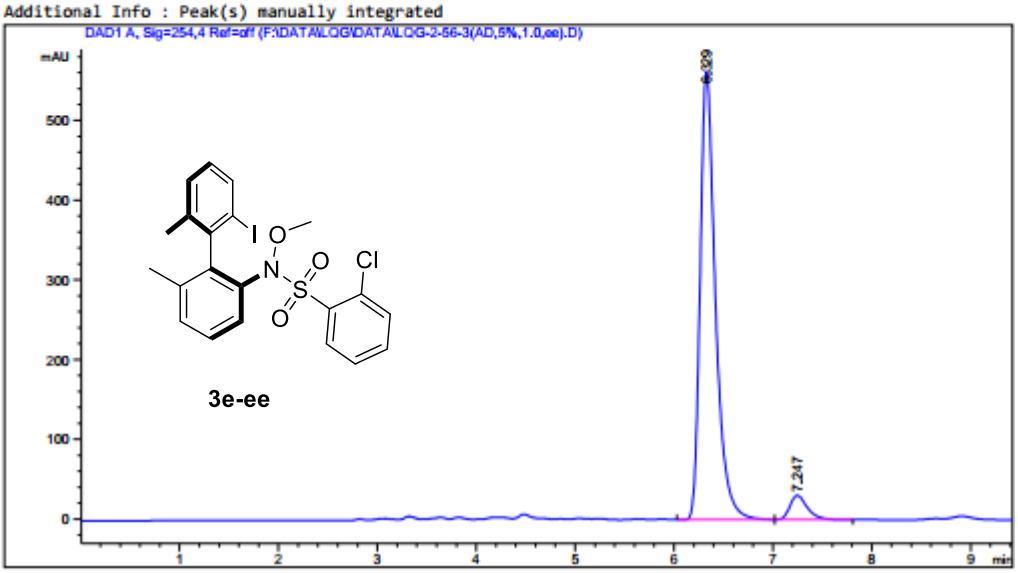
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.471	BB	0.1635	262.93973	24.05271	49.8812
2	7.406	BB	0.1891	264.19220	20.95713	50.1188
Totals :				527.13193	45.00984	

*** End of Report ***

LC1260 27/06/2019 05:24:03 SYSTEM Page 1 of 1

Data File F:\DATA\LQG\DATA\LQG-2-56-3(AD,5%,1.0,ee).D
Sample Name: LQG-2-56-3(AD,5%,1.0,ee)

```
=====================================================================
Acq. Operator   : SYSTEM
                                                  Location :   1
Injection Date  : 11/04/2019 19:58:24
Acq. Method     : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```



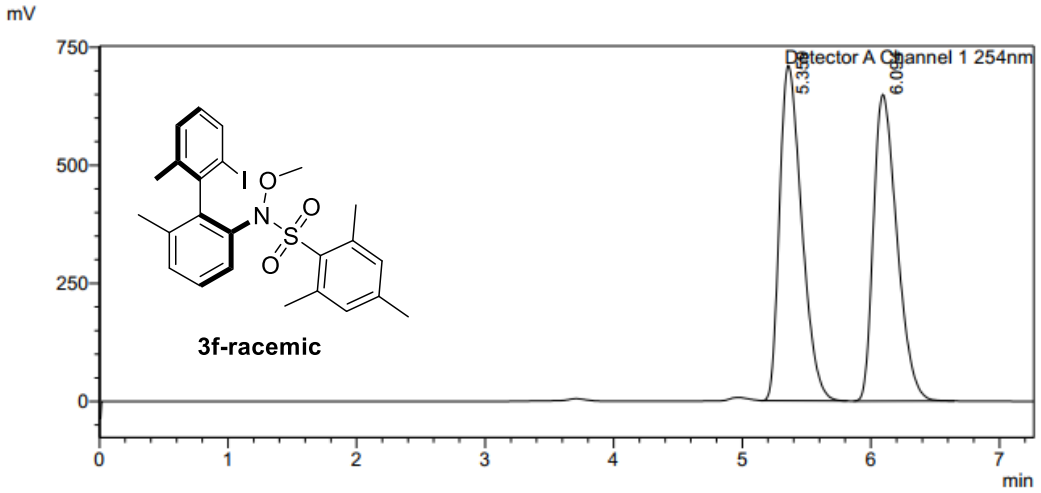
3e-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.329 BV    0.1615 6054.99707  562.74353  94.1444
   2   7.247 VB    0.1859  376.60693   30.53773   5.8556

Totals :                  6431.60400  593.28126

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

<Sample Information>

Sample Name	: LQG-2-99(AD-5%, 1.0,race)		
Sample ID	:		
Data Filename	: LQG-2-99(AD-5%, 1.0,race).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/28/2019 3:58:10 PM	Acquired by	: System Administrator
Date Processed	: 4/28/2019 4:57:55 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

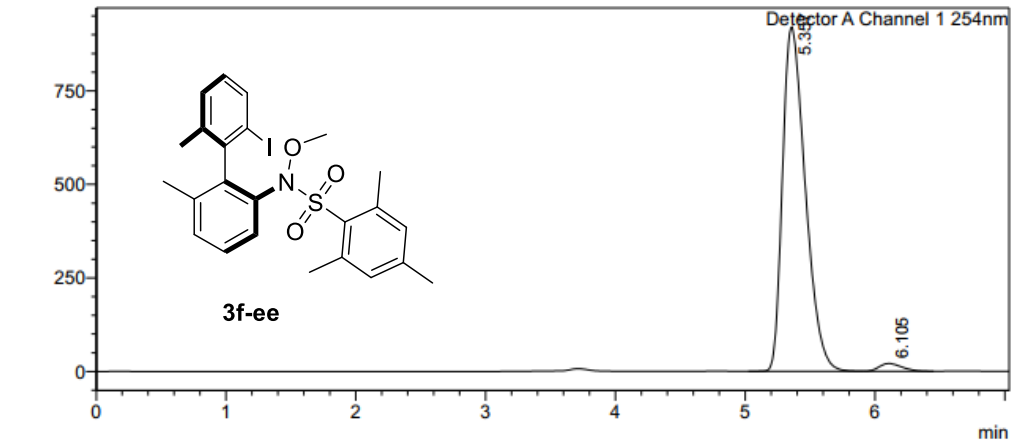
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.359	8514583	709437	50.763		M	
2	6.094	8258544	649237	49.237		M	
Total		16773127	1358675				

<Sample Information>

Sample Name	: LQG-2-99(AD-5%, 1.0,ee)		
Sample ID	:		
Data Filename	: LQG-2-99(AD-5%, 1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/28/2019 4:07:36 PM	Acquired by	: System Administrator
Date Processed	: 4/28/2019 4:57:51 PM	Processed by	: System Administrator

<Chromatogram>



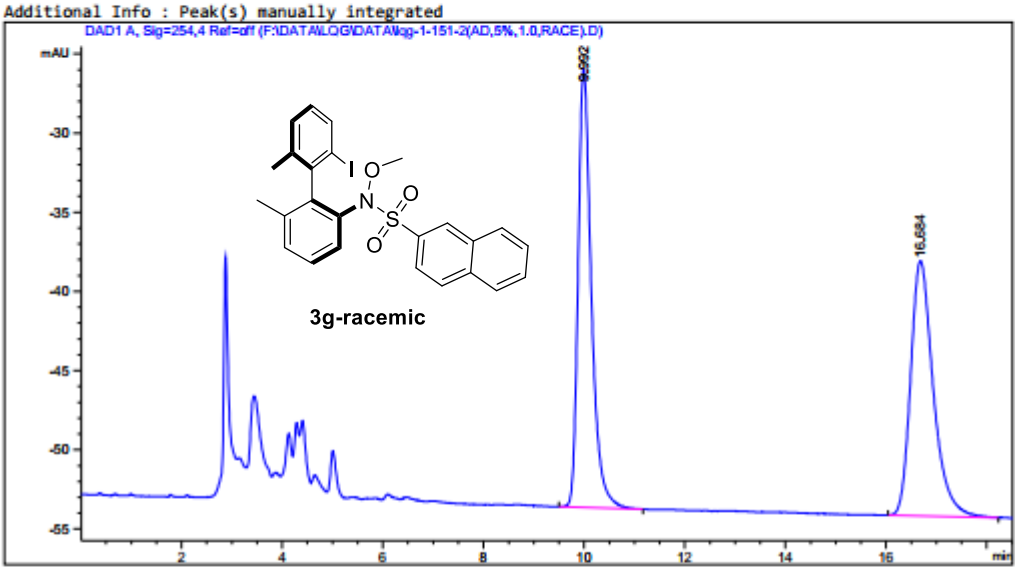
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.357	11247920	920082	97.723			
2	6.105	262117	20958	2.277		V	
Total		11510037	941040				

Data File F:\DATA\LQG\DATA\lqg-1-151-2(AD,5%,1.0,RACE).D
Sample Name: lqg-1-151-2(AD,5%,1.0,RACE)

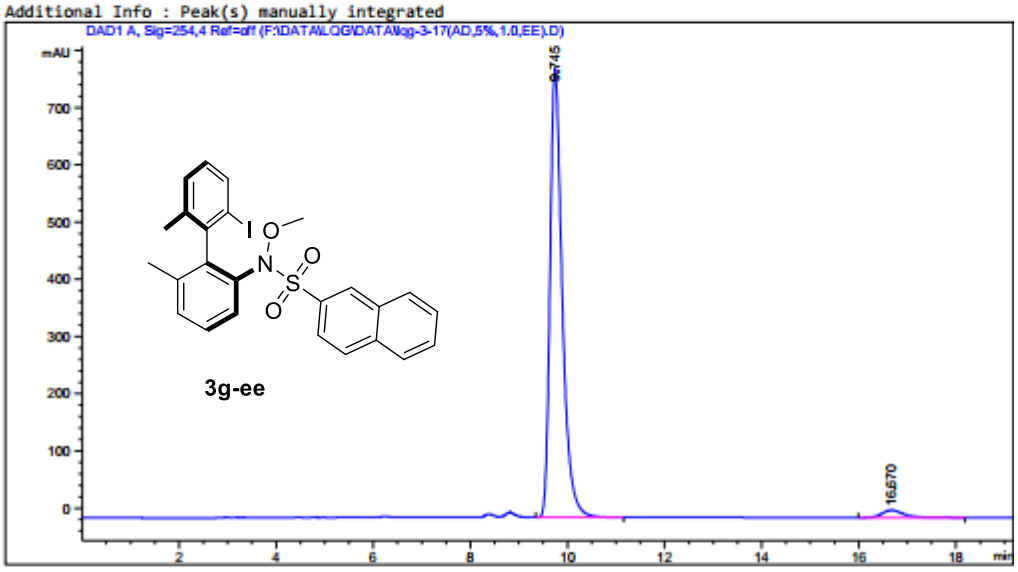
```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 09/10/2018 20:40:19
                                                Inj Volume : Manually
Acq. Method     : F:\METHOD\rxdeng.M
Last changed    : 09/10/2018 20:19:51 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.992 BB    0.2773  506.69492   27.71560  50.2195
   2  16.684 BB    0.4739  502.26578   16.10133  49.7805

Totals :                  1008.96069   43.81694
```

Data File F:\DATA\LQG\DATA\lqg-3-17(AD,5%,1.0,EE).D
Sample Name: lqg-3-17(AD,5%,1.0,EE)

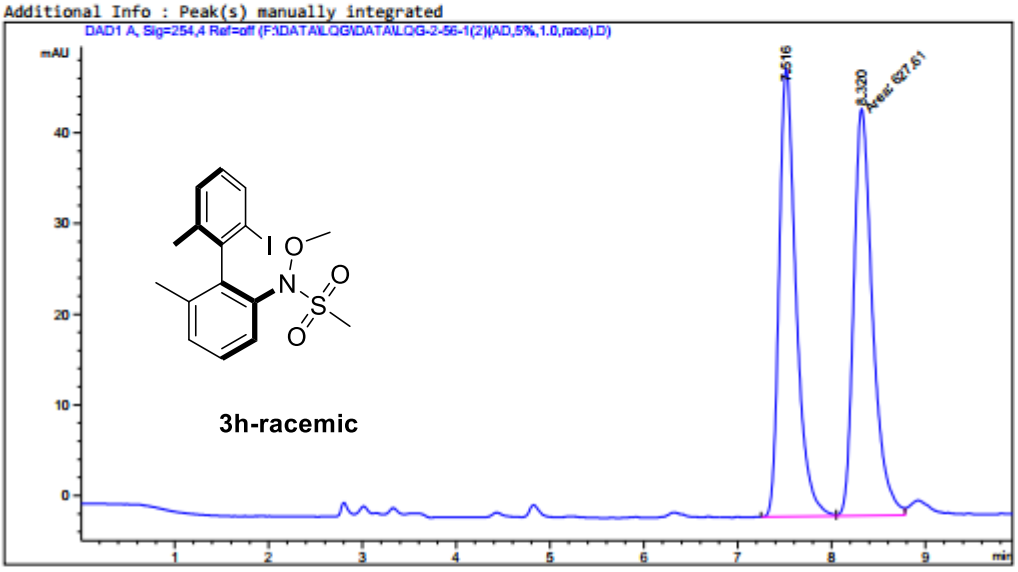
```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 16/06/2019 09:40:37
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 16/06/2019 09:37:20 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.745 BB    0.2632 1.36613e4   784.35779  97.2126
   2  16.670 BB    0.4599  391.71805   12.76794   2.7874

Totals :                  1.40530e4   797.12573
```

Data File F:\DATA\LQG\DATA\LQG-2-56-1(2)(AD,5%,1.0,race).D
Sample Name: LQG-2-56-1(2)(AD,10%,1.0,race)

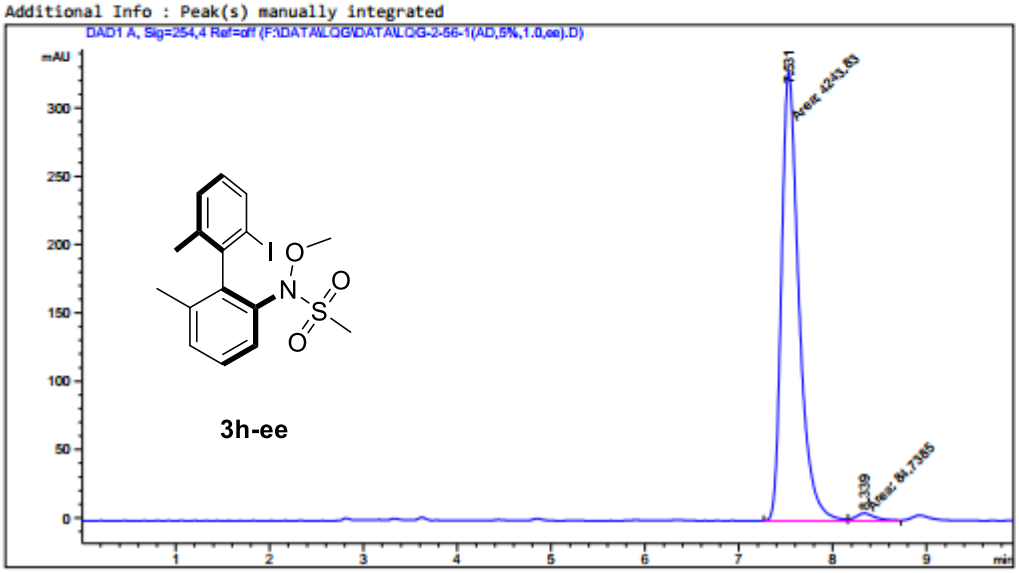
```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 11/04/2019 20:47:11
                                               Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 11/04/2019 20:44:24 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.516 BV    0.1879  617.53455   49.39601  49.5954
   2   8.320 MF    0.2332  627.60956   44.86383  50.4046

Totals :                  1245.14410   94.25984
```

Data File F:\DATA\LQG\DATA\LQG-2-56-1(AD,5%,1.0,ee).D
Sample Name: LQG-2-56-1(AD,5%,1.0,ee)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 11/04/2019 20:59:46
Acq. Method : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



3h-ee

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

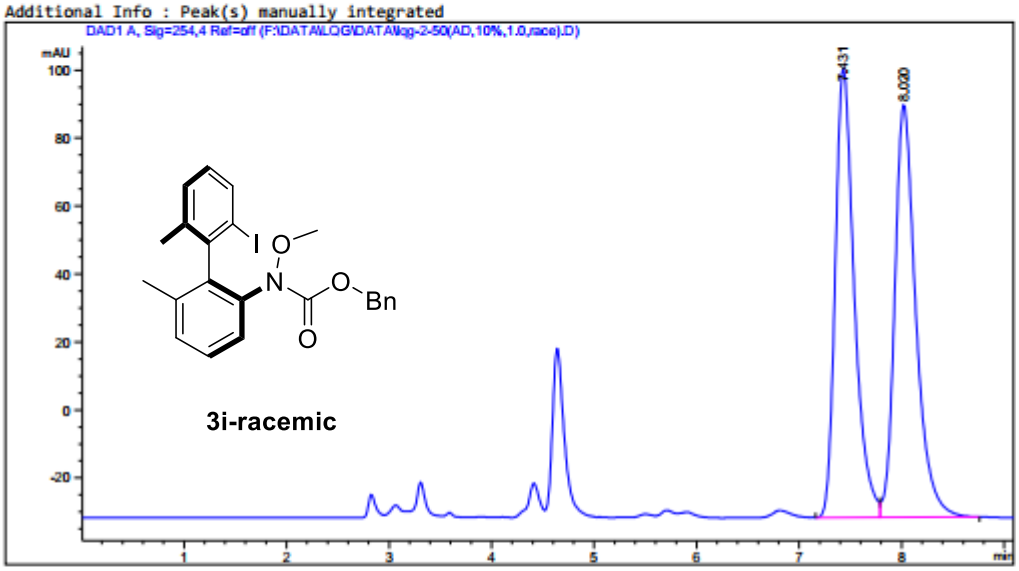
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.531	MF	0.2147	4243.83008	329.37405	98.0423
2	8.339	FM	0.2536	84.73846	5.56881	1.9577
Totals :				4328.56854	334.94286	

*** End of Report ***

LC1260 27/06/2019 04:23:17 SYSTEM Page 1 of 1

Data File F:\DATA\LQG\DATA\lqg-2-50(AD,10%,1.0,race).D
Sample Name: lqg-2-50(AD,10%,1.0,race)

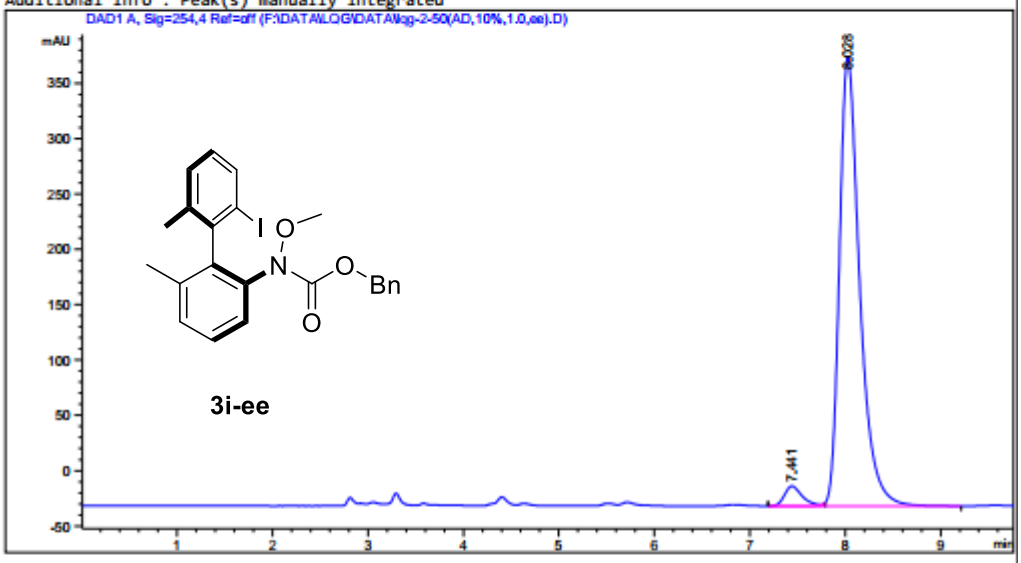
```
    ======================================================================
    Acq. Operator   : SYSTEM
    Sample Operator : SYSTEM
    Acq. Instrument : LC1260                        Location :   1
    Injection Date  : 07/04/2019 19:25:37
                                                    Inj Volume : No inj
    Acq. Method     : F:\METHOD\LQG.M\LQG.M
    Last changed    : 07/04/2019 19:17:58 by SYSTEM
                      (modified after loading)
    Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
    Last changed    : 13/02/2014 23:27:44 by SYSTEM
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.431 BV    0.1967 1705.54810  132.07222  49.5472
       2   8.020 VB    0.2172 1736.72070  121.21343  50.4528

    Totals :                   3442.26880  253.28565
```

Data File F:\DATA\LQG\DATA\lqg-2-50(AD,10%,1.0,ee).D
Sample Name: lqg-2-50(AD,10%,1.0,ee)

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 07/04/2019 19:37:35
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 07/04/2019 19:17:58 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```

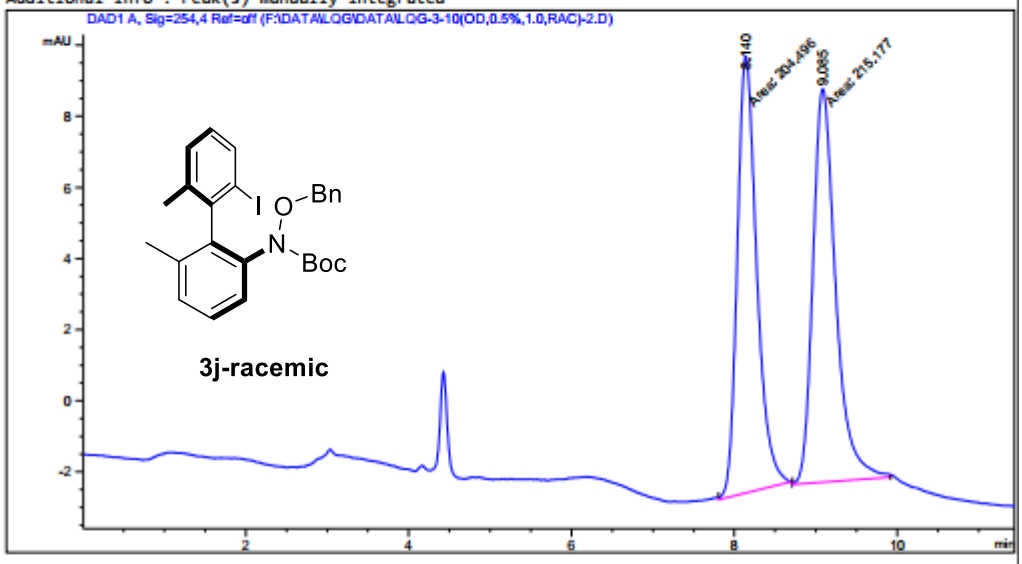


3i-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.441 BV E  0.1919  229.95912   17.89172   3.6998
   2   8.028 VB R  0.2239 5985.53516  406.23102  96.3002

Totals :                  6215.49428  424.12273
```

Data File F:\DATA\LQG\DATA\LQG-3-10(OD,0.5%,1.0,RAC)-2.D
Sample Name: LQG-3-10(OD,0.5%,1.0,RAC)-2

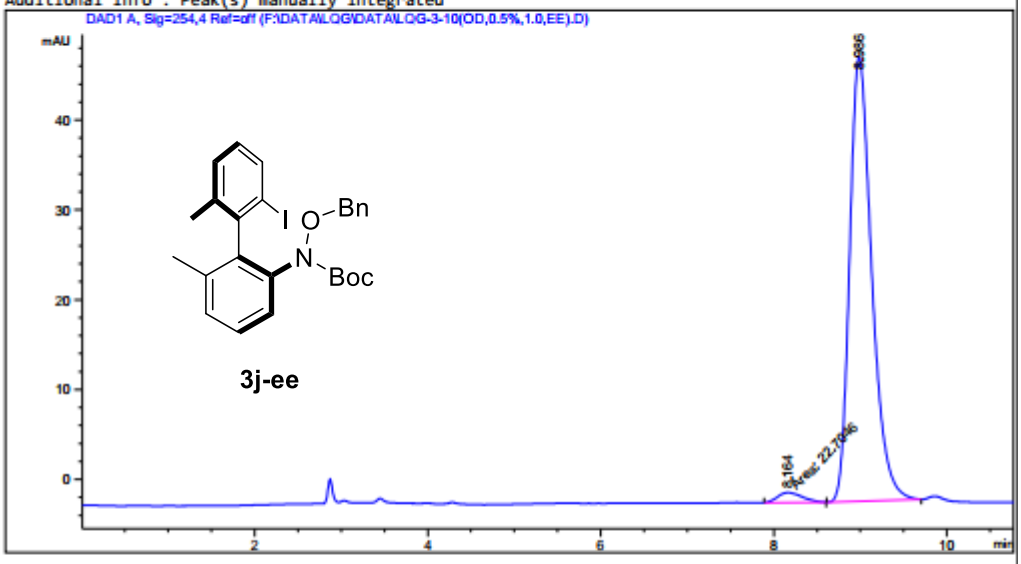
```
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 13/06/2019 21:05:48
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 13/06/2019 20:40:33 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.140 MM    0.2776  204.49597   12.27596  48.7274
   2   9.085 MM    0.3241  215.17735   11.06642  51.2726

Totals :                   419.67332   23.34238
```

Data File F:\DATA\LQG\DATA\LQG-3-10(OD,0.5%,1.0,EE).D
Sample Name: LQG-3-10(OD,0.5%,1.0,EE)

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 13/06/2019 21:19:37
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 13/06/2019 20:40:33 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```



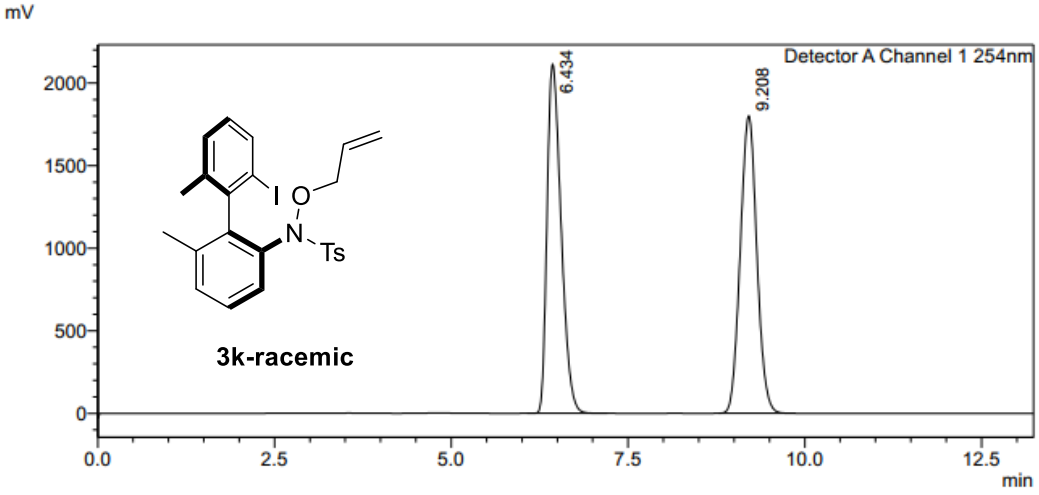
3j-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.164 MM    0.3359   22.70460    1.12672   2.5209
   2   8.986 BB    0.2731  877.96289   49.46983  97.4791

Totals :                   900.66749   50.59655
```

<Sample Information>

Sample Name	: lqg-2-139-1(AD,5%,1.0,rac)		
Sample ID	:		
Data Filename	: lqg-2-139-1(AD,5%,1.0,rac).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/13/2019 5:51:39 PM	Acquired by	: System Administrator
Date Processed	: 5/13/2019 6:04:53 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

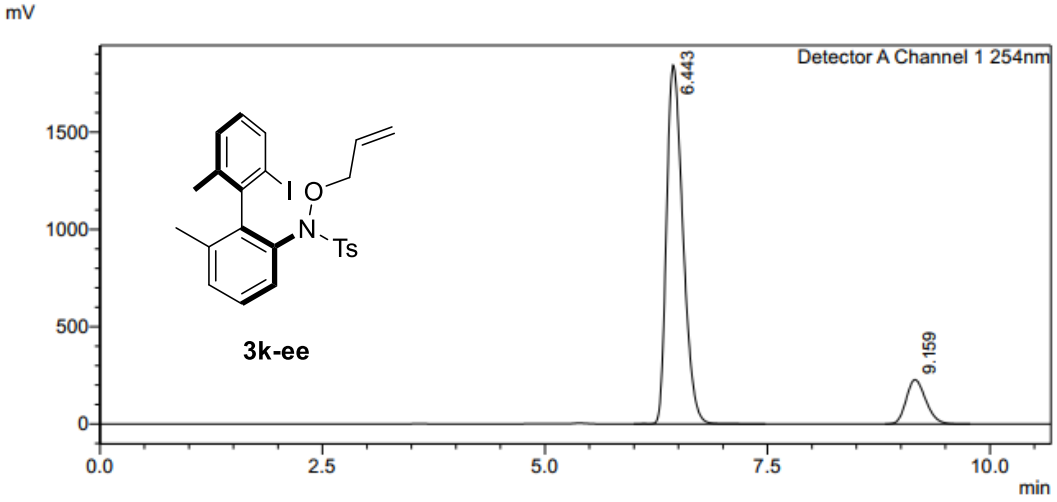
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.434	29153697	2111293	49.815		M	
2	9.208	29369842	1800452	50.185		M	
Total		58523539	3911745				

<Sample Information>

Sample Name	: lqg-2-139-1(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: lqg-2-139-1(AD,5%,1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/13/2019 6:05:44 PM	Acquired by	: System Administrator
Date Processed	: 5/13/2019 6:16:26 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

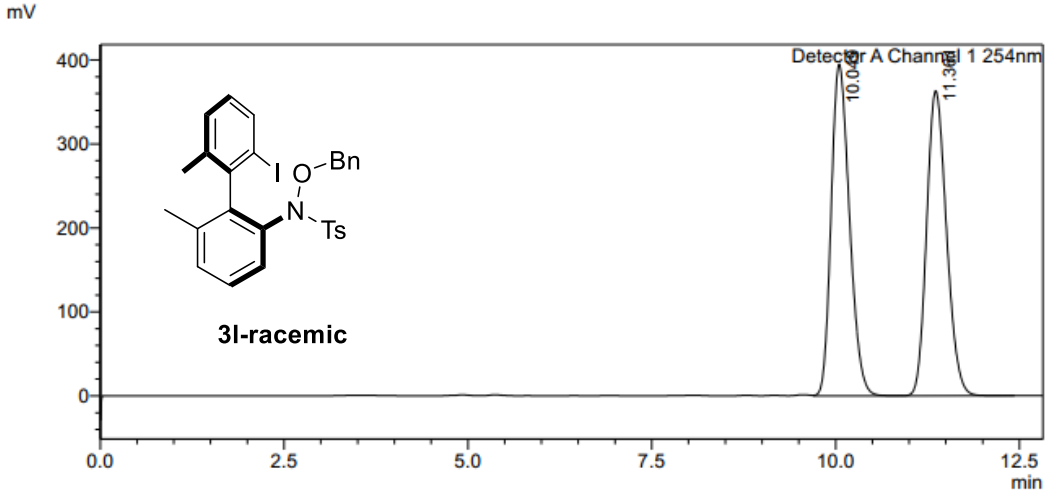
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.443	23576801	1841594	87.145		M	
2	9.159	3477787	227148	12.855		M	
Total		27054588	2068742				

<Sample Information>

Sample Name	: LQG-2-61(AD,5%,1.0,race)		
Sample ID	:		
Data Filename	: LQG-2-61(AD,5%,1.0,race).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/13/2019 6:48:46 PM	Acquired by	: System Administrator
Date Processed	: 4/13/2019 7:01:36 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

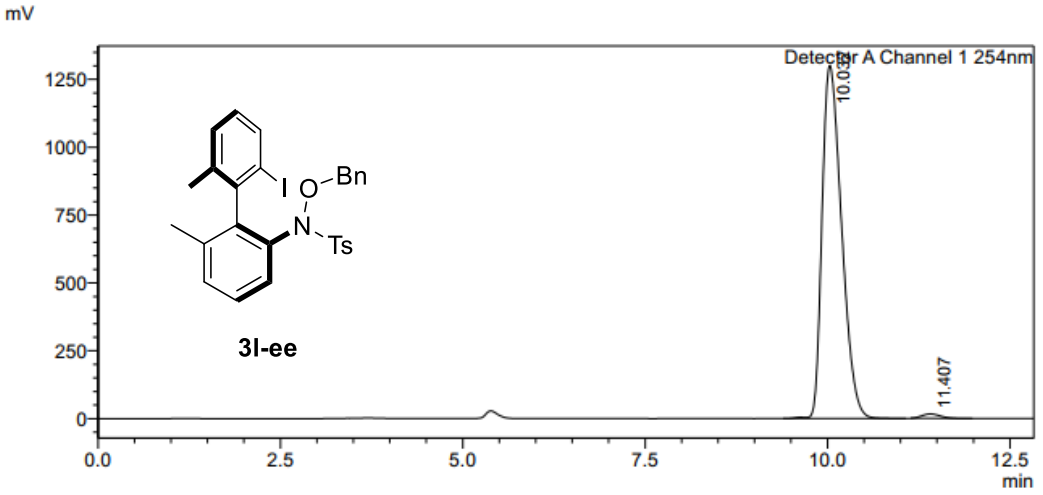
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.049	6833796	394522	50.048			
2	11.361	6820697	362831	49.952		V	
Total		13654494	757353				

<Sample Information>

Sample Name	: LQG-2-61(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: LQG-2-61(AD,5%,1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 4/13/2019 7:06:26 PM	Acquired by	: System Administrator
Date Processed	: 4/13/2019 7:19:16 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

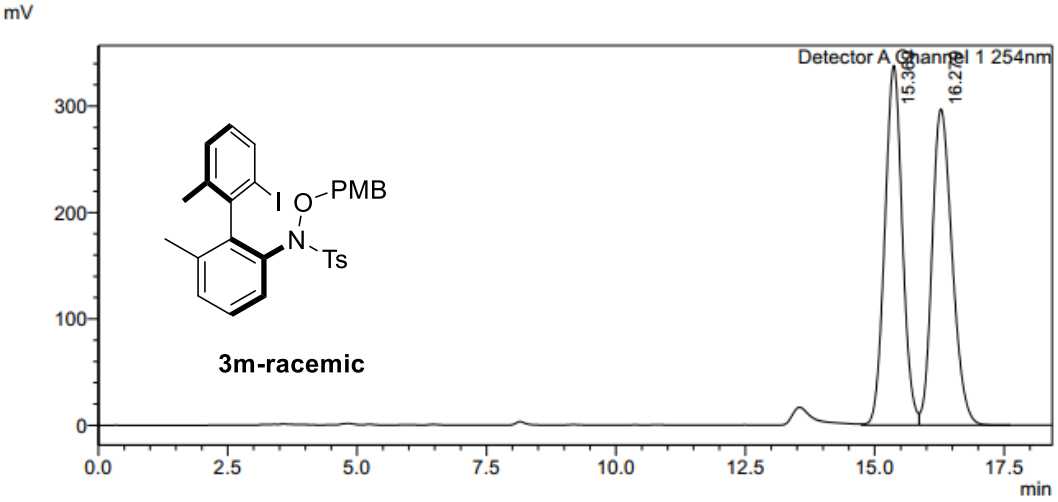
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.032	24222313	1300280	98.829		M	
2	11.407	286886	16385	1.171		M	
Total		24509199	1316664				

<Sample Information>

Sample Name	: lqg-2-139-2(AD,5%,1.0,rac)		
Sample ID	:		
Data Filename	: lqg-2-139-2(AD,5%,1.0,rac).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/13/2019 6:20:26 PM	Acquired by	: System Administrator
Date Processed	: 5/13/2019 8:02:33 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

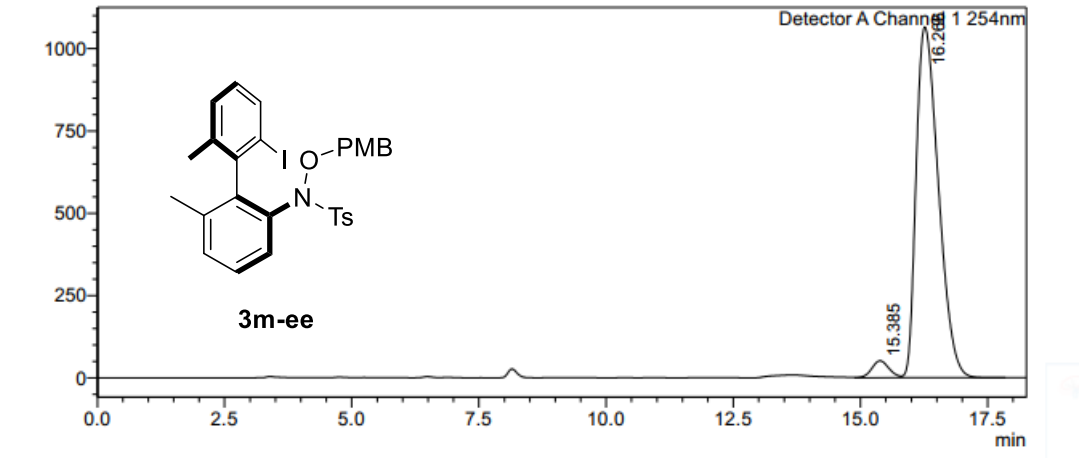
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.369	7962296	337984	49.907			
2	16.279	7992077	297179	50.093		SV	
Total		15954373	635163				

<Sample Information>

Sample Name	: lqg-2-139-2(AD,5%,1.0,ee)		
Sample ID	:		
Data Filename	: lqg-2-139-2(AD,5%,1.0,ee).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/13/2019 6:47:05 PM	Acquired by	: System Administrator
Date Processed	: 5/13/2019 8:01:14 PM	Processed by	: System Administrator

<Chromatogram>



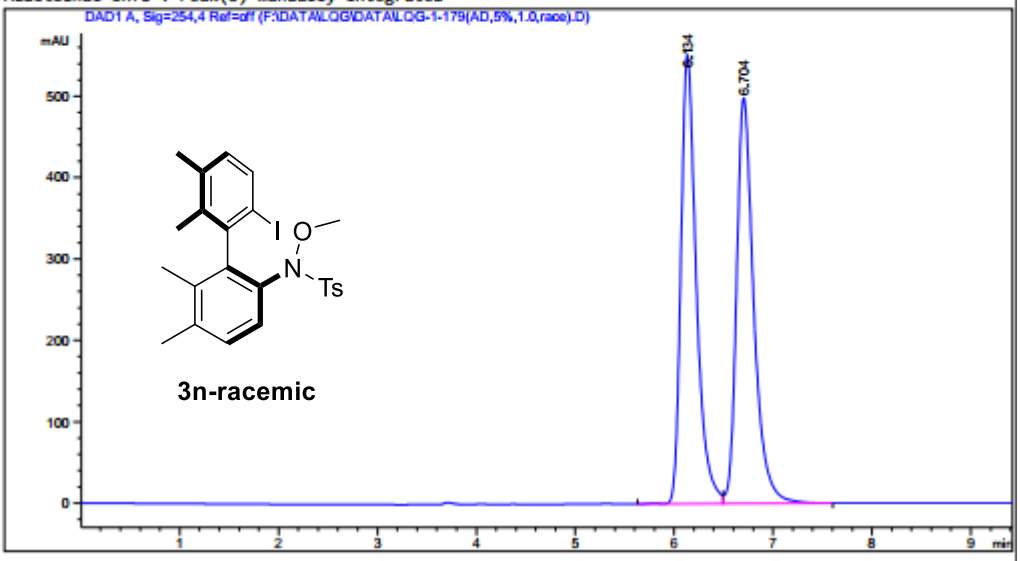
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.385	1220020	51682	3.621			
2	16.266	32468845	1065603	96.379		V	
Total		33688864	1117286				

Data File F:\DATA\LQG\DATA\LQG-1-179(AD,5%,1.0,race).D
Sample Name: LQG-1-179(AD,5%,1.0,race)

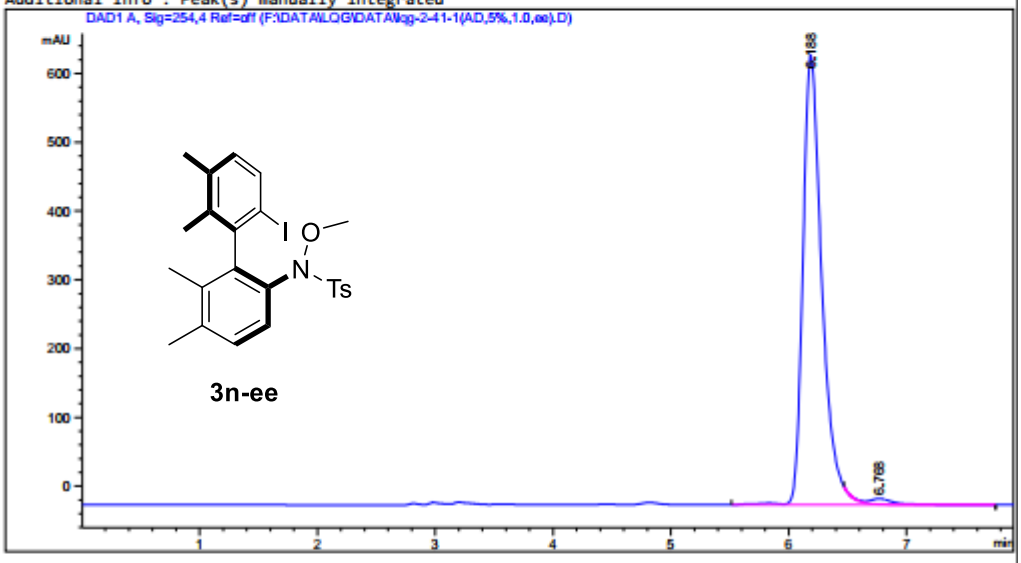
```
==========================================================================
Acq. Operator   : SYSTEM
                                                    Location :   1
Injection Date  : 19/12/2018 17:47:13
Acq. Method     : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.134 VV R  0.1622 5960.21729  551.11469  49.7273
   2   6.704 VB    0.1831 6025.59229  498.21259  50.2727

Totals :                  1.19858e4  1049.32727


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


LC1260 27/06/2019 03:30:28 SYSTEM                          Page   1 of 1
```

Data File F:\DATA\LQG\DATA\lqg-2-41-1(AD,5%,1.0,ee).D
Sample Name: lqg-2-41-1(AD,5%,1.0,ee)

```
==================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 04/04/2019 15:42:54
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 04/04/2019 15:38:24 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```



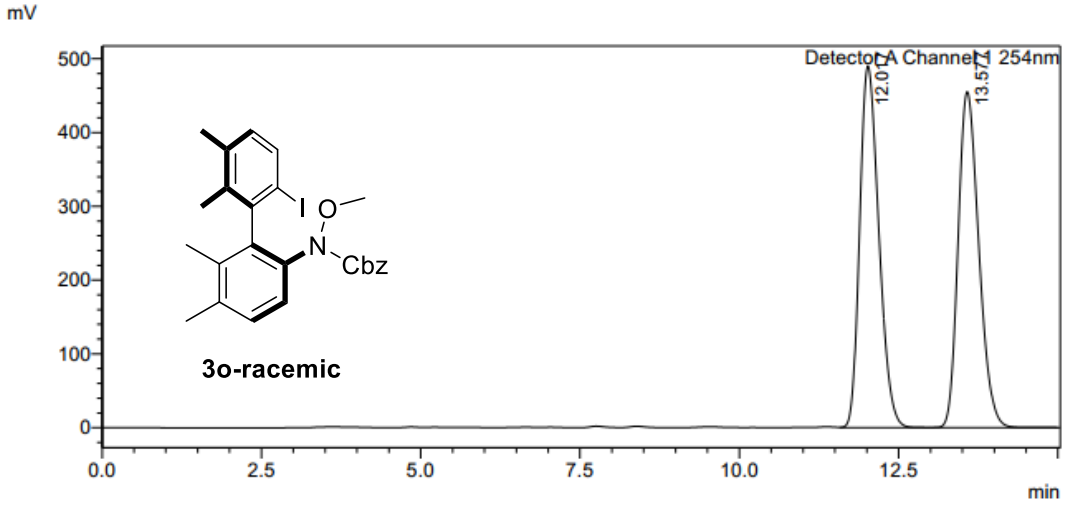
3n-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.188 VV R  0.1687 7347.11621  652.88885  98.3552
   2   6.768 VB E  0.2118  122.86988    8.24781   1.6448

Totals :                  7469.98609  661.13667
```

<Sample Information>

Sample Name	: lqg-2-153-1(AD,5%,1.0,rac)		
Sample ID	:		
Data Filename	: lqg-2-153-1(AD,5%,1.0,rac).lcd		
Method Filename	: GWJ single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/17/2019 10:43:47 AM	Acquired by	: System Administrator
Date Processed	: 5/17/2019 10:58:50 AM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

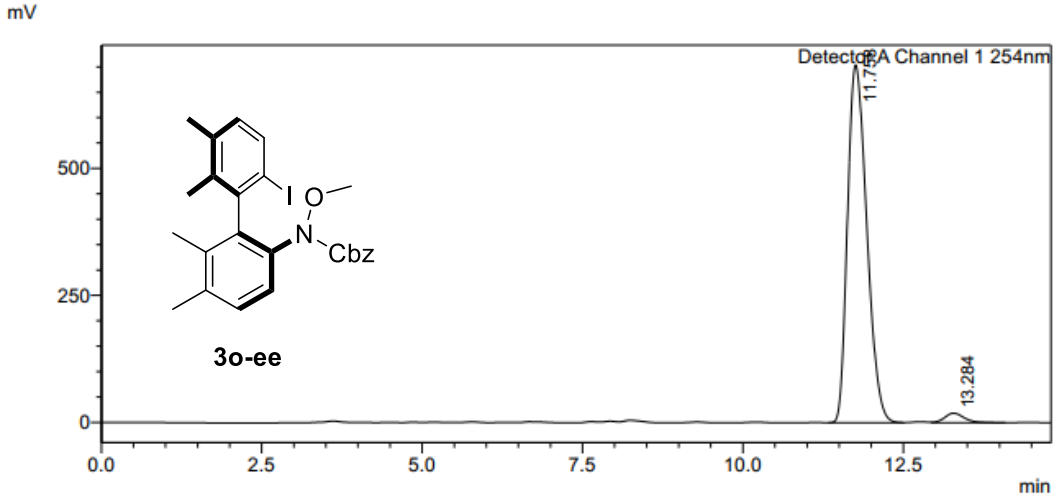
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.017	10246056	489727	49.902			
2	13.577	10286221	454864	50.098		V	
Total		20532276	944590				

<Sample Information>

Sample Name	: LQG-2-153-1(AD-5%, 1.0,EE)		
Sample ID	:		
Data Filename	: LQG-2-153-1(AD-5%, 1.0,EE).lcd		
Method Filename	: LCY single.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/17/2019 1:36:09 PM	Acquired by	: System Administrator
Date Processed	: 5/17/2019 1:50:59 PM	Processed by	: System Administrator

<Chromatogram>



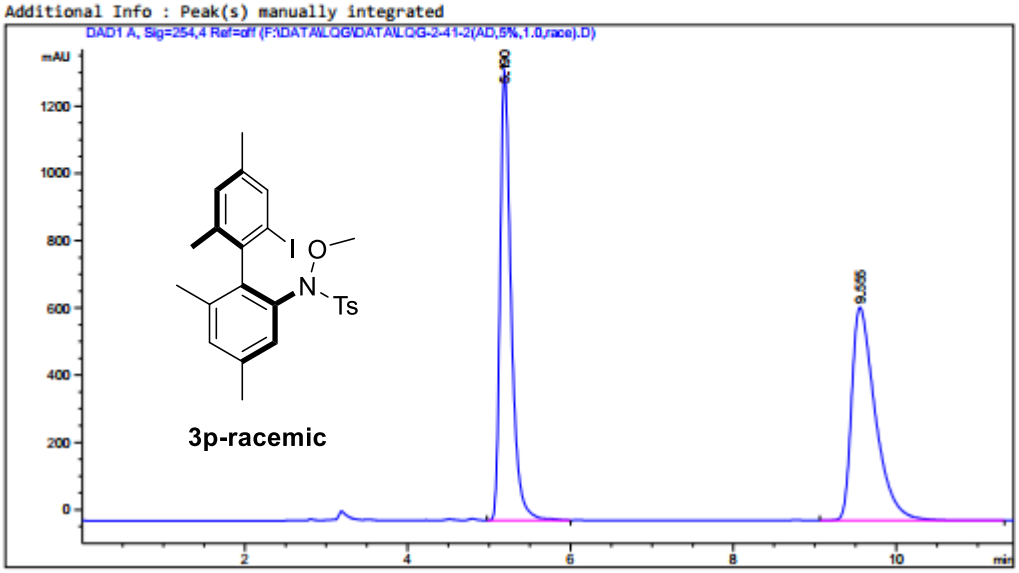
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.758	14715431	704132	97.330			
2	13.284	403736	18833	2.670			
Total		15119168	722966				

Data File F:\DATA\LQG\DATA\LQG-2-41-2(AD,5%,1.0,race).D
Sample Name: LQG-2-41-2(AD,5%,1.0,race)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 07/04/2019 18:17:05
Acq. Method : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.190	BB	0.1420	1.25895e4	1335.55835	49.5909
2	9.555	BB	0.3036	1.27972e4	633.78894	50.4091

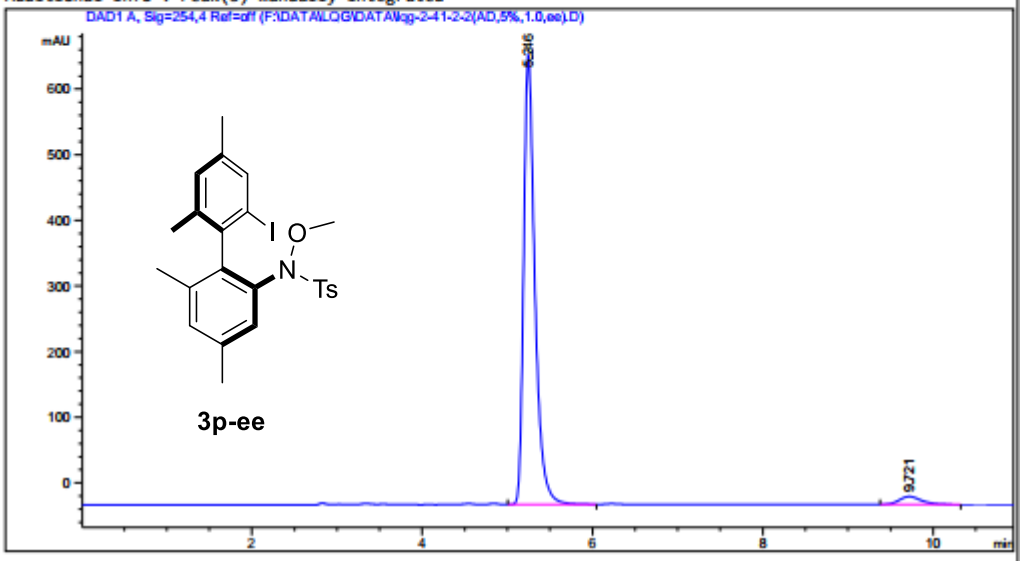
Totals : 2.53866e4 1969.34729

*** End of Report ***

LC1260 23/06/2019 10:54:32 SYSTEM Page 1 of 1

Data File F:\DATA\LQG\DATA\lqg-2-41-2-2(AD,5%,1.0,ee).D
Sample Name: lqg-2-41-2-2(AD,5%,1.0,ee)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 07/04/2019 19:54:48
Acq. Method : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.246	BB	0.1370	6274.51465	684.17462	96.7616
2	9.721	BB	0.2693	209.99706	11.93598	3.2384
Totals :				6484.51170	696.11061	

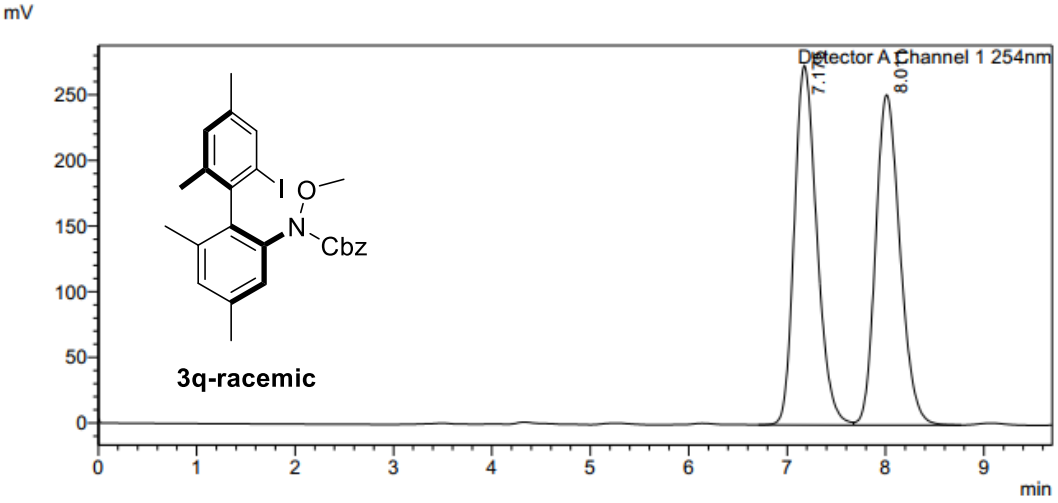
*** End of Report ***

LC1260 27/06/2019 03:21:18 SYSTEM Page 1 of 1

<Sample Information>

Sample Name	: LQG-2-153-2(OD,2%,1.0,rac)		
Sample ID	:		
Data Filename	: LQG-2-153-2(OD,2%,1.0,rac).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/30/2019 11:28:50 PM	Acquired by	: System Administrator
Date Processed	: 5/30/2019 11:38:32 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

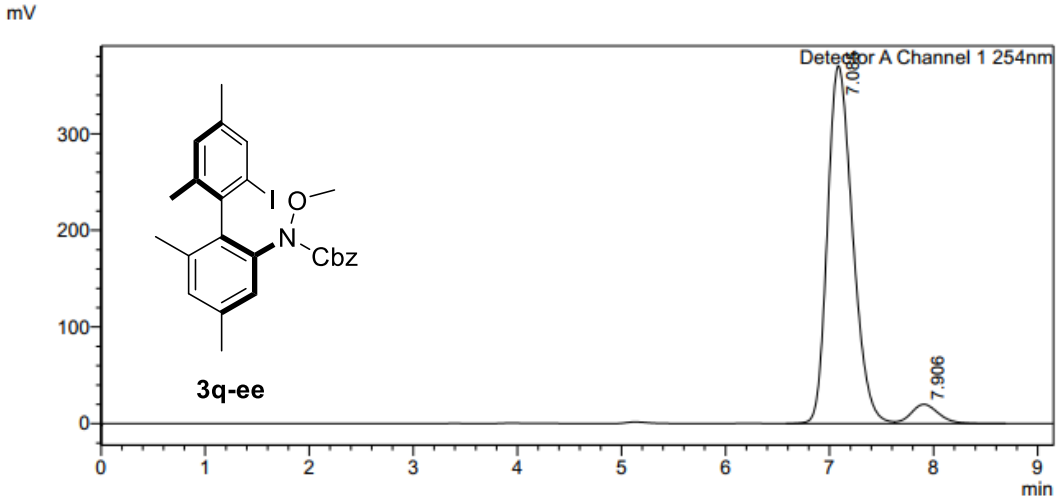
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.175	4366517	273718	50.225			
2	8.011	4327365	251313	49.775		V	
Total		8693881	525031				

<Sample Information>

Sample Name	: LQG-2-153-2(OD,2%,1.0,EE)		
Sample ID	:		
Data Filename	: LQG-2-153-2(OD,2%,1.0,EE).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/30/2019 11:43:45 PM	Acquired by	: System Administrator
Date Processed	: 5/30/2019 11:52:54 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

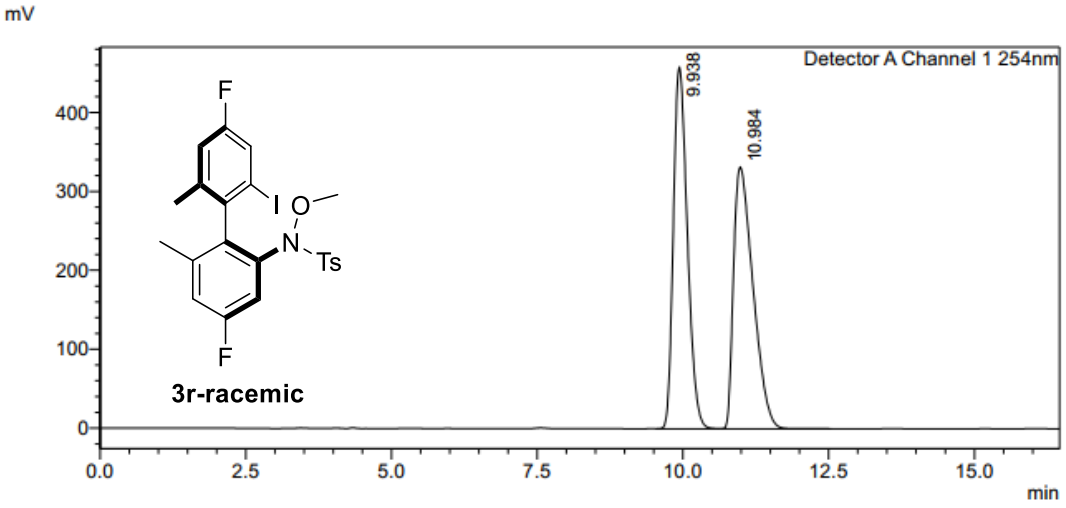
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.085	6222239	370115	94.360			
2	7.906	371895	19955	5.640		V	
Total		6594133	390070				

<Sample Information>

Sample Name	: LQG-2-164(AD,2%,1.0,rac)		
Sample ID	:		
Data Filename	: LQG-2-164(AD,2%,1.0,rac).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/22/2019 8:04:08 PM	Acquired by	: System Administrator
Date Processed	: 5/22/2019 8:20:37 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

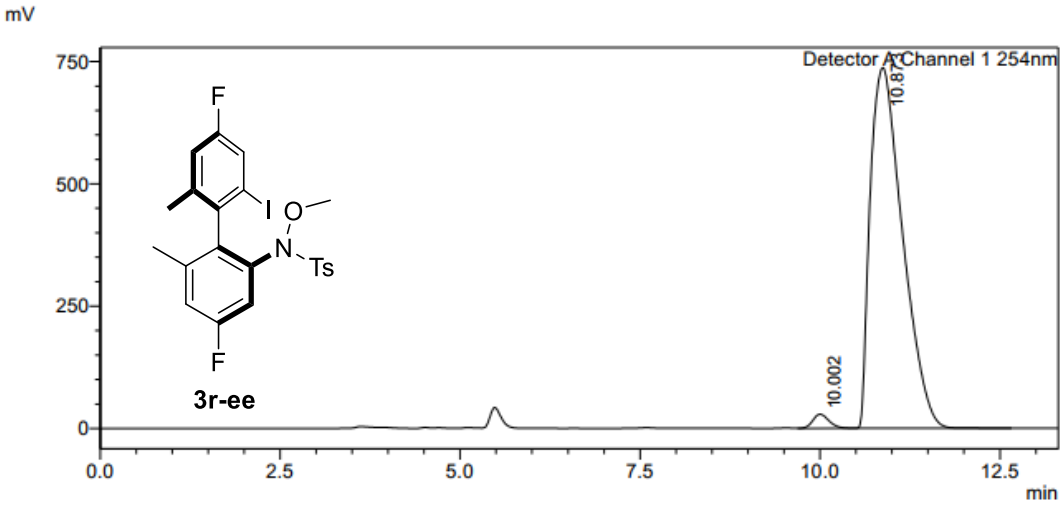
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.938	7647447	458004	49.819		V	
2	10.984	7703083	331421	50.181		SV	
Total		15350530	789425				

<Sample Information>

Sample Name	: LQG-2-164(AD,2%,1.0,EE)		
Sample ID	:		
Data Filename	: LQG-2-164(AD,2%,1.0,EE).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/22/2019 8:25:34 PM	Acquired by	: System Administrator
Date Processed	: 5/22/2019 8:38:53 PM	Processed by	: System Administrator

<Chromatogram>



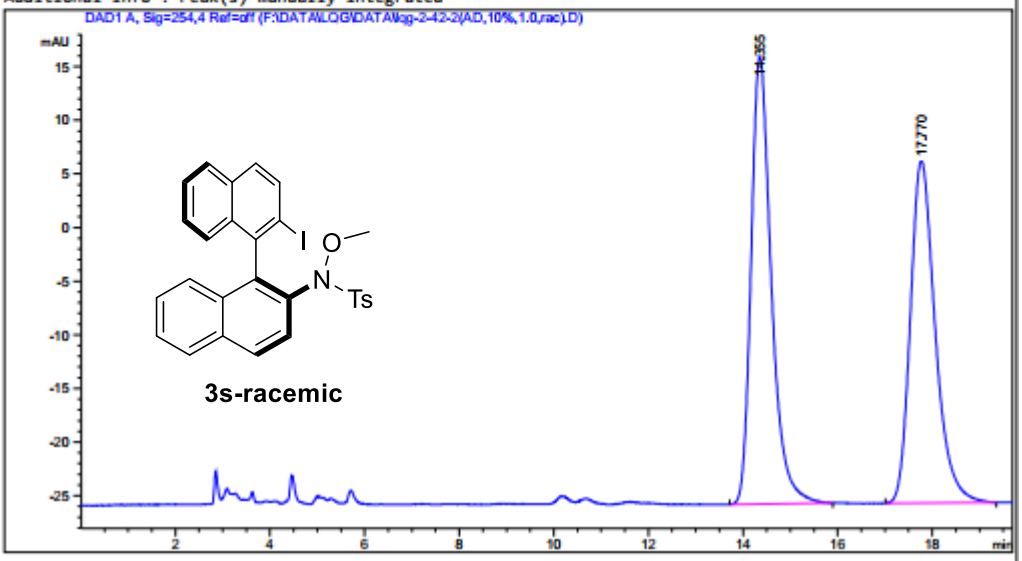
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.002	454248	28782	1.936			
2	10.873	23012579	737071	98.064		V	
Total		23466827	765852				

Data File F:\DATA\LQG\DATA\lqg-2-42-2(AD,10%,1.0,rac).D
Sample Name: lqg-2-42-2(AD,10%,1.0,rac)

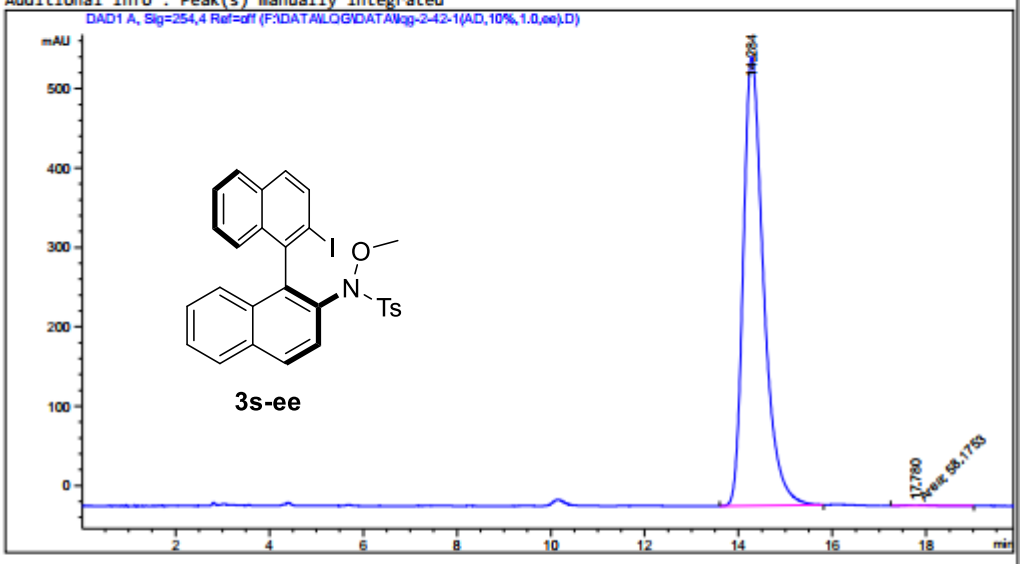
```
==================================================================
Acq. Operator   : SYSTEM
                                                  Location :   1
Injection Date  : 04/04/2019 16:36:47
Acq. Method     : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.355 BB    0.4499 1237.13672   41.72853  51.5447
   2  17.770 BB    0.5510 1162.98914   31.92636  48.4553

Totals :                  2400.12585   73.65489


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


LC1260 27/06/2019 03:44:53 SYSTEM                    Page   1 of 1
```

Data File F:\DATA\LQG\DATA\lqg-2-42-1(AD,10%,1.0,ee).D
Sample Name: lqg-2-42-1(AD,10%,1.0,ee)

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 04/04/2019 16:58:22
                                               Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 04/04/2019 16:10:35 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```

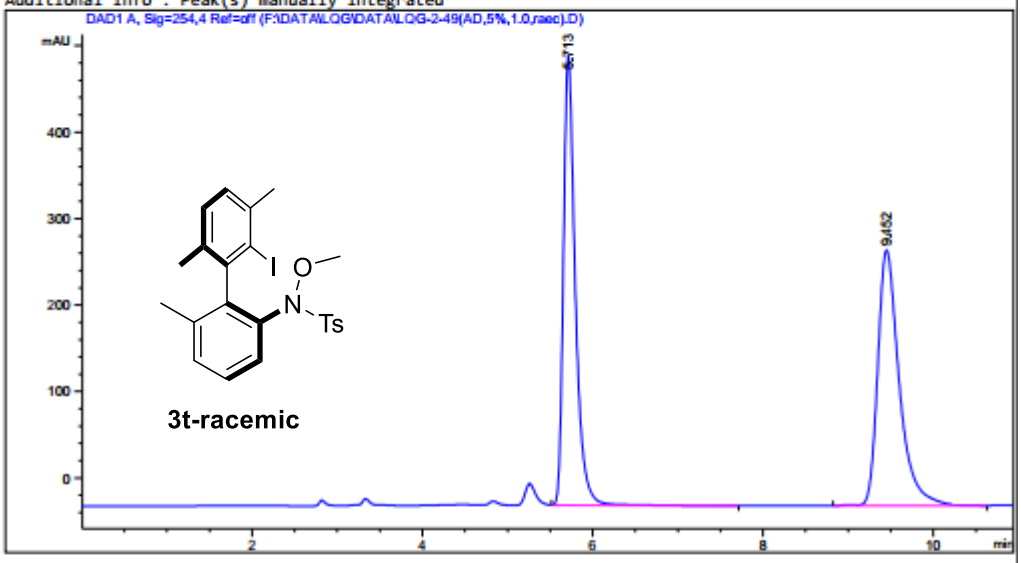


3s-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.284 BB    0.4577 1.69493e4   565.48126  99.6579
   2  17.780 MM    0.6182   58.17533    1.56849   0.3421

Totals :                  1.70075e4   567.04976
```

Data File F:\DATA\LQG\DATA\LQG-2-49(AD,5%,1.0,raec).D
Sample Name: LQG-2-49(AD,5%,1.0,raec)

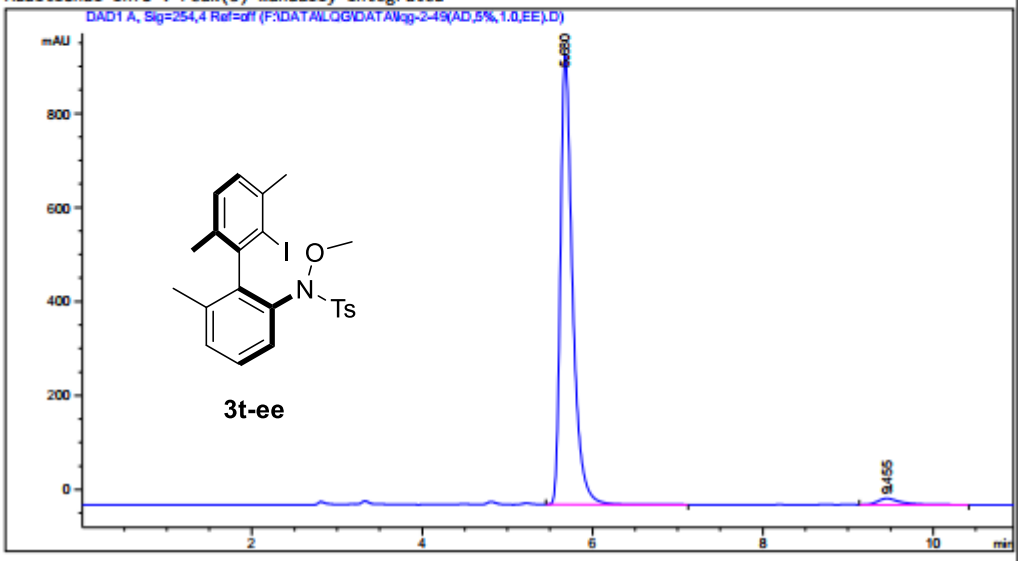
```
==================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 07/04/2019 18:44:01
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 07/04/2019 18:14:18 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.713 VB    0.1456 5062.84375  519.84351  49.3886
   2   9.452 VB R  0.2624 5188.19824  295.46429  50.6114

Totals :                  1.02510e4   815.30780
```

Data File F:\DATA\LQG\DATA\lqg-2-49(AD,5%,1.0,EE).D
Sample Name: lqg-2-49(AD,5%,1.0,EE)

```
==========================================================================
Acq. Operator   : SYSTEM
                                                    Location :   1
Injection Date  : 07/04/2019 19:05:56
Acq. Method     : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```



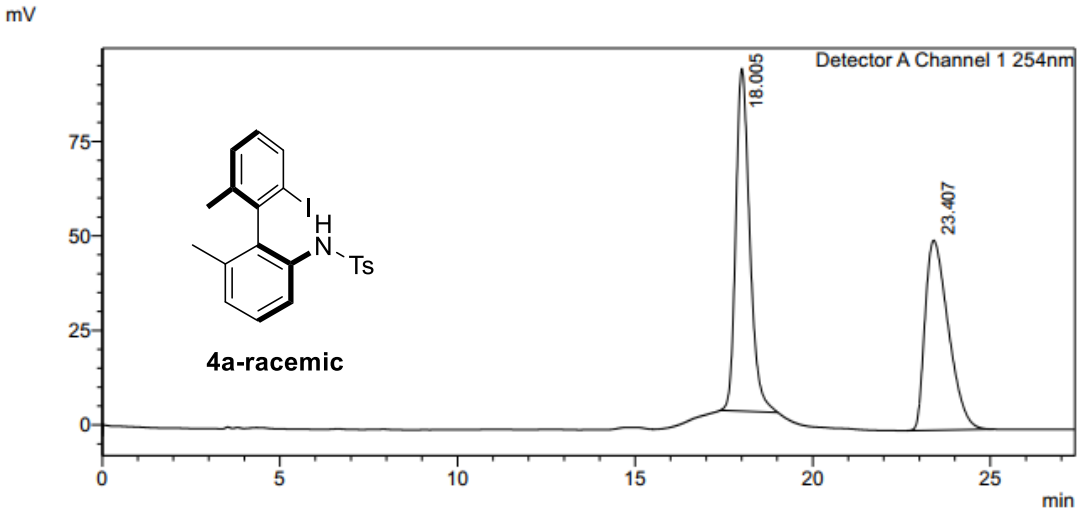
3t-ee

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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.680 VB    0.1497 9482.16504  955.73468  97.4887
   2   9.455 BB    0.2706  244.25656   13.27935   2.5113

Totals :                  9726.42160  969.01403

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


LC1260 27/06/2019 03:50:42 SYSTEM                          Page   1 of 1
```

<Sample Information>

Sample Name	: LQG-2-140(ID,5%,1.0,RAC)		
Sample ID	:		
Data Filename	: LQG-2-140(ID,5%,1.0,RAC).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/30/2019 7:45:39 PM	Acquired by	: System Administrator
Date Processed	: 5/30/2019 8:13:02 PM	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

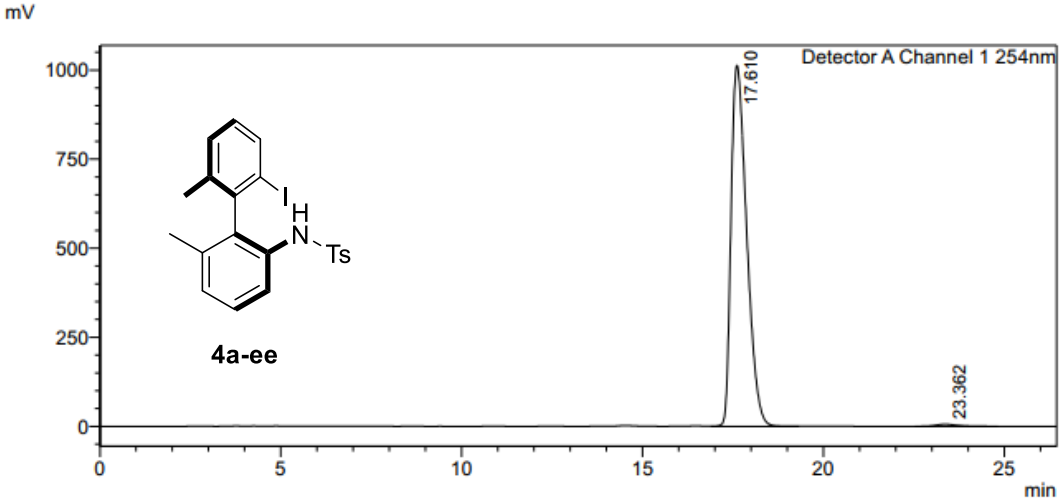
Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.005	2477397	90555	51.904		M	
2	23.407	2295613	50168	48.096			
Total		4773009	140723				

<Sample Information>

Sample Name	: LQG-2-183(ID,5%,1.0,ee)		
Sample ID	:		
Data Filename	: LQG-2-183(ID,5%,1.0,ee).lcd		
Method Filename	: LQG.lcm		
Batch Filename	:		
Vial #	: 1-1	Sample Type	: Unknown
Injection Volume	: 15 uL		
Date Acquired	: 5/30/2019 8:19:43 PM	Acquired by	: System Administrator
Date Processed	: 5/30/2019 8:46:10 PM	Processed by	: System Administrator

<Chromatogram>



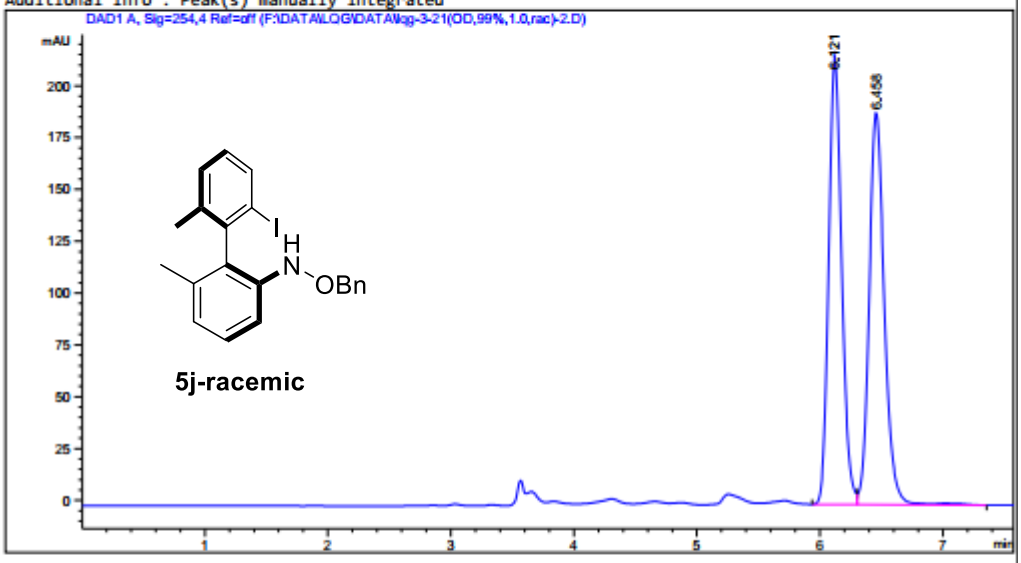
<Peak Table>

Detector A Channel 1 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.610	30483390	1012893	99.267			
2	23.362	225103	5555	0.733		S	
Total		30708493	1018448				

Data File F:\DATA\LQG\DATA\lqg-3-21(OD,99%,1.0,rac)-2.D
Sample Name: lqg-3-21(OD,99%,1.0,rac)-2

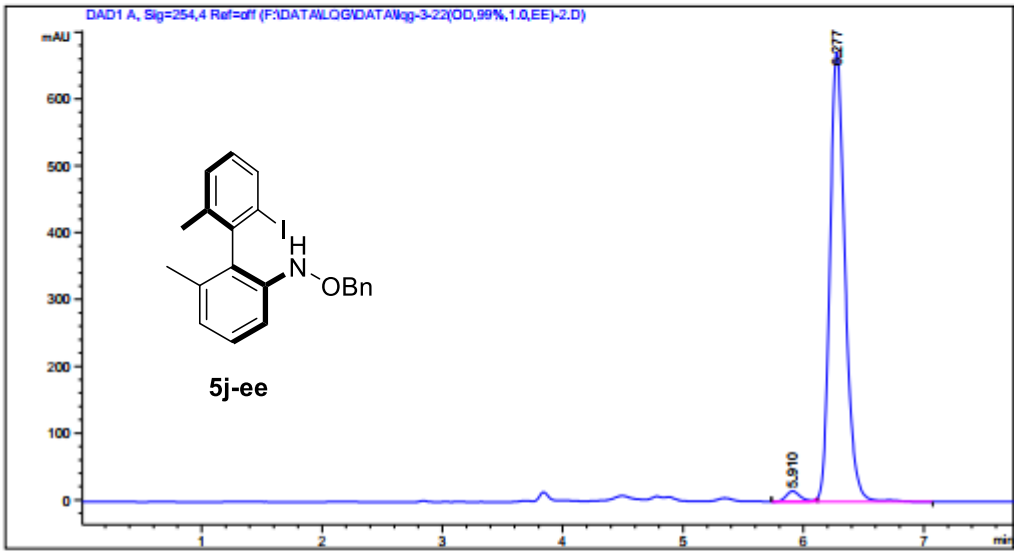
```
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 27/06/2019 03:07:54
                                                Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 27/06/2019 02:45:02 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




```
                        Area Percent Report

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.121 BV    0.1135 1583.65442  216.01329  49.2245
   2   6.458 VV R  0.1312 1633.55298  189.10907  50.7755

Totals :                  3217.20740  405.12236
```

Data File F:\DATA\LQG\DATA\lqg-3-22(OD,99%,1.0,EE)-2.D
Sample Name: lqg-3-22(OD,99%,1.0,EE)-2

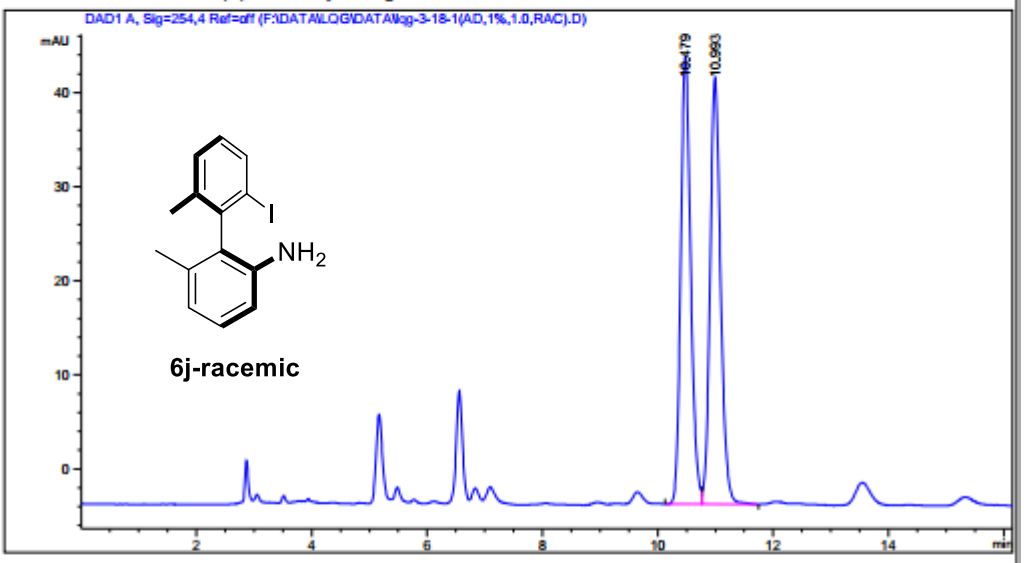
```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : LC1260                        Location :   1
Injection Date  : 27/06/2019 03:26:02
                                               Inj Volume : No inj
Acq. Method     : F:\METHOD\LQG.M\LQG.M
Last changed    : 27/06/2019 02:45:02 by SYSTEM
                  (modified after loading)
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed    : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.910 BV E  0.1231  127.86519   15.68876   2.1217
   2   6.277 VV R  0.1338 5898.67578  672.73169  97.8783

Totals :                  6026.54097  688.42045
```

Data File F:\DATA\LQG\DATA\lqg-3-18-1(AD,1%,1.0,RAC).D
Sample Name: lqg-3-18-1(AD,1%,1.0,RAC)

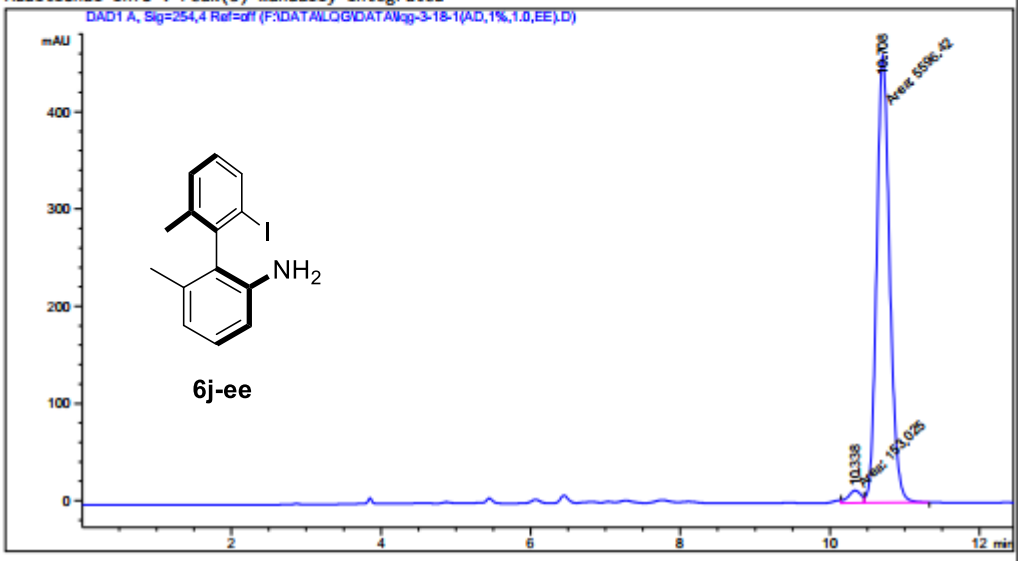
```
    ======================================================================
    Acq. Operator   : SYSTEM
    Sample Operator : SYSTEM
    Acq. Instrument : LC1260                        Location :   1
    Injection Date  : 27/06/2019 04:12:45
                                                   Inj Volume : No inj
    Acq. Method     : F:\METHOD\LQG.M\LQG.M
    Last changed    : 27/06/2019 04:02:06 by SYSTEM
                      (modified after loading)
    Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
    Last changed    : 13/02/2014 23:27:44 by SYSTEM
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  10.479 BV    0.1840  570.94690   47.57078  49.8269
       2  10.993 VB    0.1942  574.91272   45.25718  50.1731

    Totals :                  1145.85962   92.82796


LC1260 27/06/2019 04:31:37 SYSTEM                          Page   1 of 2
```

Data File F:\DATA\LQG\DATA\lqg-3-18-1(AD,1%,1.0,EE).D
Sample Name: lqg-3-18-1(AD,1%,1.0,EE)

Acq. Operator : SYSTEM
Location : 1
Injection Date : 27/06/2019 04:41:46
Acq. Method : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



6j-ee

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

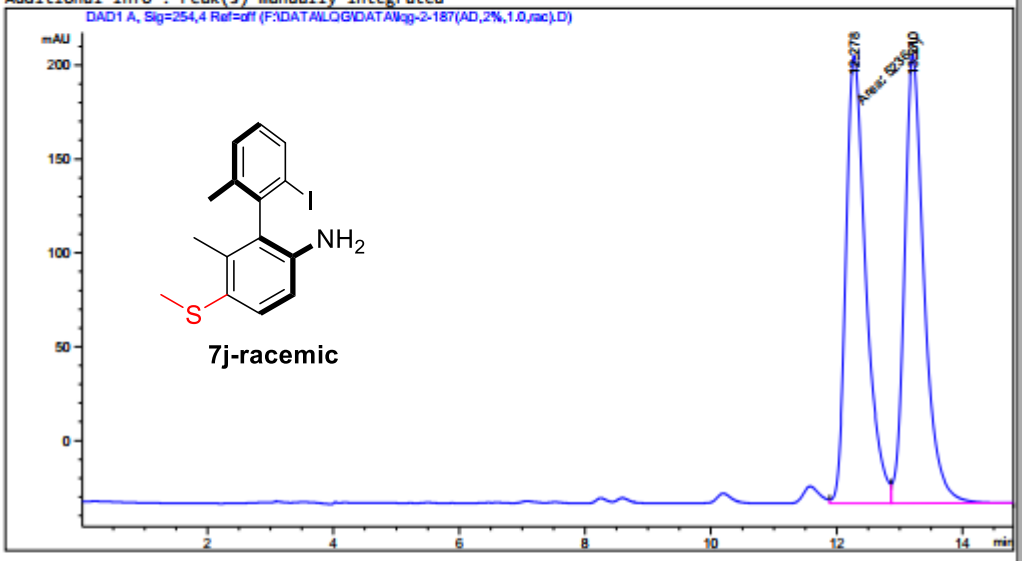
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.338	FM	0.1993	153.02519	12.80002	2.6616
2	10.708	FM	0.2026	5596.42139	460.40860	97.3384
Totals :				5749.44658	473.20862	

*** End of Report ***

LC1260 27/06/2019 04:57:59 SYSTEM Page 1 of 1

Data File F:\DATA\LQG\DATA\lqg-2-187(AD,2%,1.0,rac).D
Sample Name: lqg-2-187(AD,2%,1.0,rac)

```
    ================================================================
    Acq. Operator    : SYSTEM
    Sample Operator  : SYSTEM
    Acq. Instrument  : LC1260                        Location :   1
    Injection Date   : 01/06/2019 17:17:23
                                                  Inj Volume : No inj
    Acq. Method      : F:\METHOD\LQG.M\LQG.M
    Last changed     : 01/06/2019 17:14:08 by SYSTEM
                       (modified after loading)
    Analysis Method  : C:\Chem32\1\Methods\DEF_LC.M
    Last changed     : 13/02/2014 23:27:44 by SYSTEM
    Additional Info  : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 A, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.278 FM    0.3658 5236.31006  238.59991  50.1401
       2  13.210 VBA   0.3263 5207.04443  238.92494  49.8599

    Totals :                  1.04434e4   477.52486


LC1260 27/06/2019 04:03:33 SYSTEM                          Page   1 of 2
```

Data File F:\DATA\LQG\DATA\lqg-3-18-2(AD,2%,1.0,EE)-2.D
Sample Name: lqg-3-18-2(AD,2%,1.0,EE)-2

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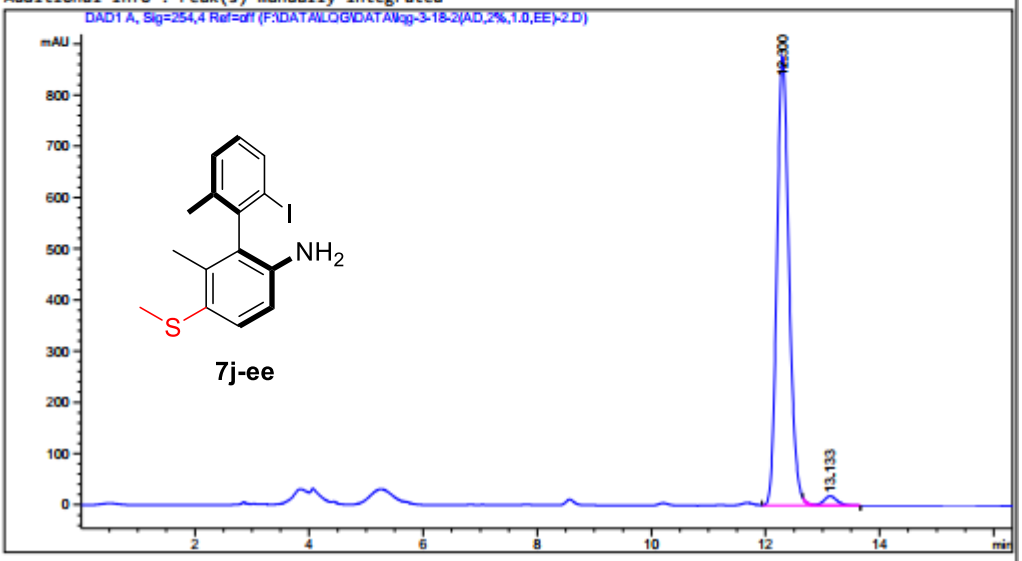
Acq. Operator : SYSTEM

Location : 1

Injection Date : 27/06/2019 05:16:30
Acq. Method : LQG.M
Analysis Method : C:\Chem32\1\Methods\DEF_LC.M
Last changed : 13/02/2014 23:27:44 by SYSTEM
Additional Info : Peak(s) manually integrated



7j-ee

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Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.300	VV R	0.2261	1.29332e4	876.59192	97.7341
2	13.133	VB E	0.2483	299.85281	18.38339	2.2659
Totals :				1.32330e4	894.97531	

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*** End of Report ***

LC1260 27/06/2019 05:34:36 SYSTEM

Page 1 of 1