Supporting Information

Cooperative Catalysis in Highly Enantioselective Mannich-type Three-component Reactions of a Diazoacetophenone with an Alcohol and an Imine

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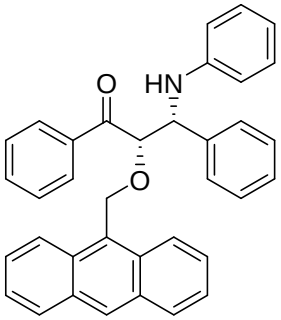
General: HRMS (ESI) Mass spectra were recorded on Bruker micrOTOF-II mass spectrometer. NMR spectra were recorded on a Brucker-400 MHz spectrometer. HPLC analysis was performed on Shimadzu (SPD-20AV UV-VIS Detector and LC-20AT Liquid Chromatograph Pump). Chiralpak OD, AD, AD-H, IA were purchased from Daicel Chemical Industries, LTD. The racemic standards used in HPLC studies were prepared according to the general procedure by using racemic BINOL derivatived phosphoric acid catalysts.

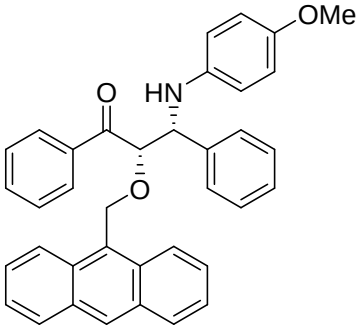
Materials: Dichloromethane was distilled from calcium hydride. Diazo compounds **1** were prepared according to the literature procedure.[1] Imines **3** were prepared by condensation of corresponding aldehydes and amines.[2] Cyclohexanecarboxaldehyde was purchased from ACROS. Chiral phosphoric acid **5** were prepared according to the literature procedure.[3] Solvents for the column chromatography were distilled before using.

General Procedure for the Enantioselective Three-component Reaction of 9-anthryl Alcohol (2a) With Various Diazo Compounds 1 and Imines 3 (Table 2 in the manuscript**):**

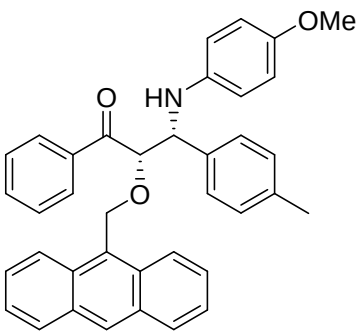
To an flame-dried vial, $Rh_2(OAc)_4$ (0.004 mmol), chiral phosphoric acid **5f** (0.01 mmol), alcohol **2a** (0.20 mmol), imine **3** (0.20 mmol) and 5Å MS (0.1 g) were added and charged with 1.5 mL toluene. Diazo compound **1** (0.24 mmol) in 0.5 mL of toluene was then added over 1 h period of time via a syringe pump at room temperature. After completion of the addition, the reaction mixture was stirred for additional 3 h and followed by addition of saturated aqueous $NaHCO_3$ (0.1 mL) to quench the reaction. Solvents were removed to give the crude products, which were subjected to ^1H NMR spectroscopy analysis for the determination of diastereoselectivity. The crude products were purified by flash chromatography on silica gel (eluent: EtOAc/light petroleum ether = 1:50 ~1:30) to give the pure products.



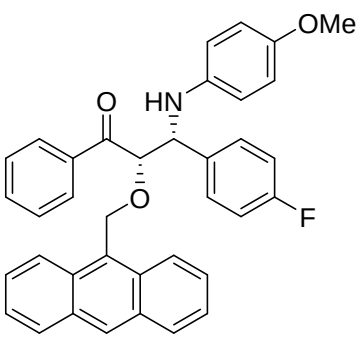
(4a): yield 85%; 90% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 9.2 min, and t_{minor} = 12.4 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 4.80 (br, 1H), 4.90 (m, 1H), 5.14 (m, 1H), 5.32 (d, *J* = 11.5 Hz, 1H), 5.66 (d, *J* = 11.5 Hz, 1H), 6.30-8.07 (m, 23H), 8.49 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ (ppm) 59.36, 64.71, 84.52, 113.58, 117.41, 124.30, 124.97, 126.40, 127.04, 127.15, 127.37, 128.44, 128.74, 128.81, 128.82, 128.86, 131.18, 131.29, 133.34; 135.93, 139.69, 146.62, 198.57; HRMS (ESI) calcd for $C_{36}H_{29}NNaO_2$ $(M+Na)^+$ 530.2091, found 530.2088.



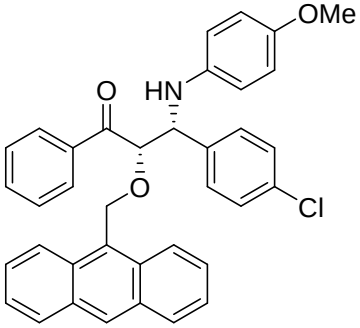
(4b): yield 88%; 91% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 15.5 min, and t_{minor} = 21.3 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.60 (s, 3H), 4.50 (br, 1H), 4.80 (m, 1H), 5.08 (m, 1H), 5.25 (d, *J* = 11.0 Hz, 1H), 5.62 (d, *J* = 11.0 Hz, 1H), 6.22 (d, *J* = 9.0 Hz, 2H), 6.51 (d, *J* = 9.0 Hz, 2H), 7.13-8.03 (m, 18H), 8.46 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.59, 60.31, 64.72, 84.85, 114.47, 114.87, 124.30, 124.97, 126.39, 127.15, 127.22, 127.35, 128.42, 128.45, 128.68, 128.83, 131.16, 131.29, 133.24; 136.06, 139.78, 140.84, 151.96, 198.80; HRMS (ESI) calcd for $C_{37}H_{31}NNaO_3$ $(M+Na)^+$ 560.2196, found 560.2205.



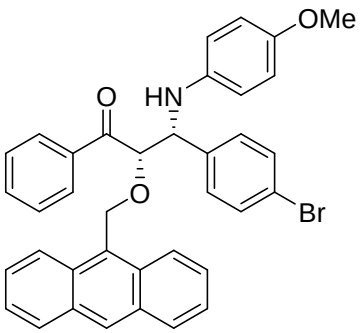
(4c): yield 83%; $[\alpha]_D^{20}$ = -23.0° (c = 1, CH_2Cl_2); 92% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 11.4 min, and t_{minor} = 20.0 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 2.27 (s, 3H), 3.60 (s, 3H), 4.51 (br, 1H), 4.77 (m, 1H), 5.04 (m, 1H), 5.25 (d, *J* = 11.0 Hz, 1H), 5.61 (d, *J* = 11.0 Hz, 1H), 6.22 (d, *J* = 9.0 Hz, 2H), 6.51 (d, *J* = 9.0 Hz, 2H), 6.90 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 7.35-8.03 (m, 13H), 8.46 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 21.10, 55.55, 59.92, 64.62, 84.95, 114.39, 114.79, 124.08, 124.91, 126.28, 126.89, 127.21, 127.55, 128.46, 128.53, 128.66, 128.78, 129.06, 131.12, 131.23; 133.20, 136.57, 136.74, 140.87, 151.82, 198.87; HRMS (ESI) calcd for $C_{38}H_{33}NNaO_3$ $(M+Na)^+$ 574.2353, found 574.2366.



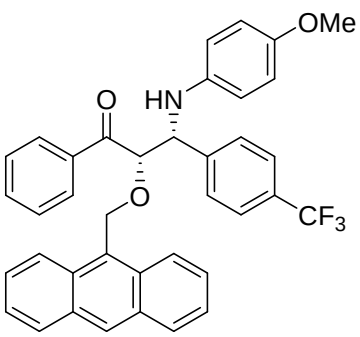
(4d): yield 78%; 92% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 15.6 min, and t_{minor} = 21.0 min.); ^{1}H NMR ($CDCl_3$, 400 MHz,): δ (ppm) 3.60 (s, 3H), 4.50 (br, 1H), 4.69 (m, 1H), 4.97 (m, 1H), 5.30 (d, *J* = 12.0 Hz, 1H), 5.65 (d, *J* = 12.0 Hz, 1H), 6.18 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.34-8.01 (m, 13H), 8.46 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz,): δ (ppm) 55.59, 59.78, 64.46, 84.29, 114.56, 114.88, 121.26, 123.90, 125.06, 126.51, 126.60, 128.58, 128.98, 129.04, 129.80, 131.08, 131.21, 131.42, 134.14, 138.52, 139.83, 140.22, 152.26, 197.72; HRMS (ESI) calcd for $C_{37}H_{30}FNNaO_3$ $(M+Na)^+$ 578.2102, found 578.2127.



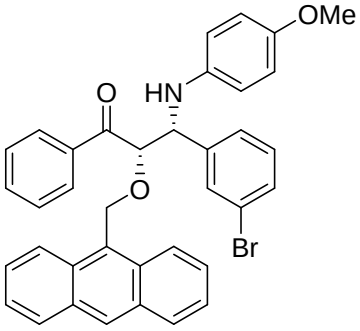
(4e): yield 73%; $[\alpha]_D^{20}$ = -34.0° (c = 1, CH_2Cl_2); 98% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 15.2 min, and t_{minor} = 25.0 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.61 (s, 3H), 4.50 (br, 1H), 4.72 (m, 1H), 4.98 (m, 1H), 5.31 (d, *J* = 11.5 Hz, 1H), 5.65 (d, *J* = 11.5 Hz, 1H), 6.18 (d, *J* = 9.0 Hz, 2H), 6.52 (d, *J* = 9.0 Hz, 2H), 6.79-8.01 (m, 17H), 8.47 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.60, 59.57, 64.40, 84.05, 113.69, 114.53, 114.77, 124.09, 125.02, 126.43, 126.90, 128.27, 128.34, 128.43, 128.79, 128.91, 131.14, 131.24, 133.38; 138.27, 140.39, 152.10, 198.56; HRMS (ESI) calcd for $C_{37}H_{30}ClNNaO_3$ $(M+Na)^+$ 594.1806, found 594.1793.



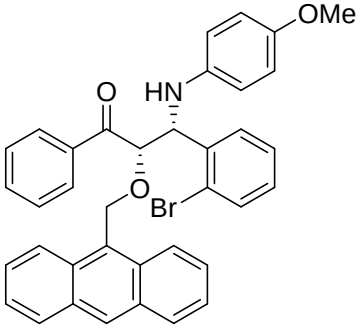
(4f): yield 70%; $[\alpha]_D^{20}$ = -35.0° (c = 1, CH_2Cl_2); 93% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 14.6 min, and t_{minor} = 33.1 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.60 (s, 3H), 4.50 (br, 1H), 4.70 (m, 1H), 4.98 (m, 1H), 5.30 (d, *J* = 11.5 Hz, 1H), 5.65 (d, *J* = 11.5 Hz, 1H), 6.18 (d, *J* = 9.0 Hz, 2H), 6.52 (d, *J* = 9.0 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.35-8.01 (m, 13H), 8.47 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.59, 59.59, 64.39, 83.93, 114.52, 114.73, 121.10, 124.06, 125.03, 126.44, 126.86, 128.32, 128.60, 128.80, 128.90, 129.16, 131.12, 131.22, 131.34; 133.39, 135.90, 138.80, 140.34, 152.08, 198.53; HRMS (ESI) calcd for $C_{37}H_{30}BrNNaO_3$ $(M+K)^+$ 638.1301, found 638.1329.



(4g): yield 75%; 90% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 10.9 min, and t_{minor} = 19.4 min.); ^{1}H NMR ($CDCl_3$, 500 MHz,): δ (ppm) 3.59 (s, 3H), 4.57 (br, 1H), 4.77 (m, 1H), 5.01 (m, 1H), 5.29 (d, *J* = 11.5 Hz, 1H), 5.65 (d, *J* = 11.5 Hz, 1H), 6.17 (d, *J* = 8.5 Hz, 2H), 6.51 (d, *J* = 8.5 Hz, 2H), 7.16-7.98 (m, 17H), 8.43 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz,): δ (ppm) 55.55, 59.73, 64.24, 83.46, 114.53, 114.64, 123.95, 125.02, 125.14, 125.17, 126.39, 126.71, 127.22, 128.24, 128.83, 128.89, 128.93, 131.08, 131.16, 133.45, 135.82, 140.15, 143.99, 152.12, 198.37; HRMS (ESI) calcd for $C_{38}H_{30}F_3NNaO_3$ $(M+Na)^+$ 628.2070, found 628.2093.

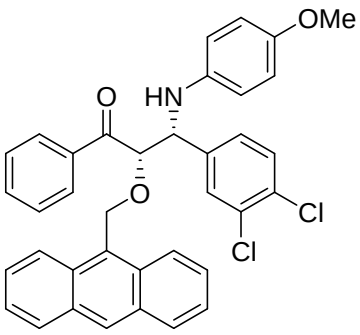


(4h): yield 76%; 92% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 13.1 min, and t_{minor} = 20.5 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.60 (s, 3H), 4.51 (br, 1H), 4.70 (m, 1H), 5.02 (m, 1H), 5.29 (d, *J* = 11.5 Hz, 1H), 5.63 (d, *J* = 11.5 Hz, 1H), 6.18 (d, *J* = 9.0 Hz, 2H), 6.52 (d, *J* = 9.0 Hz, 2H), 6.89-8.01 (m, 17H), 8.46 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.55, 59.70, 64.37, 83.80, 114.46, 114.69, 122.55, 123.98, 124.98, 125.69, 126.51, 126.81, 128.28, 128.77, 128.88, 128.97, 129.84, 129.86, 130.42, 131.10, 131.18, 133.41, 135.82, 140.27, 142.32, 152.04, 198.42; HRMS (ESI) calcd for $C_{37}H_{30}BrNNaO_3$ $(M+Na)^+$ 638.1301, found 638.1328.

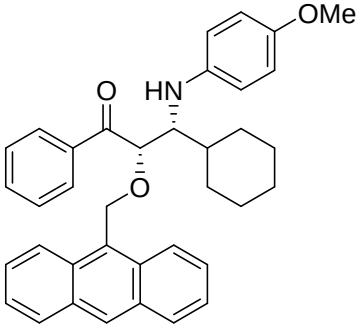


(4i): yield 82%; *syn*: 90% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol / TFA = 90: 10 :0.1, 254nm, Retention time: t_{major} = 9.7 min, and t_{minor} = 21.8 min.), *anti*: 63% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol / TFA = 90: 10: 0.1, 254nm, Retention time: t_{major} = 17.2 min, and t_{minor} = 19.3 min.); ^{1}H NMR ($CDCl_3$, 500 MHz, *syn*): δ (ppm) 3.64 (s, 3H), 4.58 (br, 1H), 5.10 (m, 1H), 5.23 (m, 1H), 5.51 (d, *J* = 12.0 Hz, 1H), 5.69 (d, *J* = 12.0 Hz, 1H), 6.24 (d, *J* = 9.0 Hz, 2H), 6.59 (d, *J* = 9.0 Hz, 2H), 6.86-8.24 (m, 17H), 8.47 (s, 1H), ^{1}H NMR ($CDCl_3$, 500 MHz, *anti*): δ (ppm) 3.57 (s, 3H), 5.01 (br, 1H), 5.21 (m, 1H), 5.24 (m, 1H), 5.34 (m, 1H), 5.62 (d, *J* = 11.2 Hz, 1H), 6.14 (d, *J* = 9.0 Hz, 2H), 6.49 (d, *J* = 9.0 Hz, 2H), 6.90-8.23 (m, 17H), 8.42 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz, *syn* & *anti*): δ (ppm) 55.49, 55.59, 58.67, 59.64, 63.93, 64.73, 80.11, 80.77, 114.28, 114.55, 114.84, 124.21, 124.27,

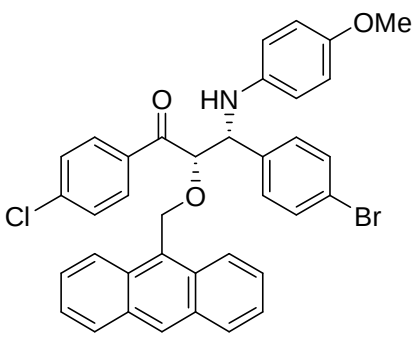
124.84, 125.00, 126.31, 126.41, 127.40, 128.22, 128.32, 128.59, 128.72, 128.88, 128.95, 131.21, 131.30, 132.52, 133.20, 136.17, 140.28, 152.17, 198.80; HRMS (ESI) calcd for $C_{37}H_{30}BrNNaO_3$ $(M+Na)^+$ 638.1301, found 638.1329.



(4k): yield 71%; $[\alpha]_D^{20}$ = -17.4° (c = 1, CH_2Cl_2); 93% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/TFA = 90: 10: 0.1, 254nm, Retention time: t_{major} = 11.5 min, and t_{minor} = 27.3 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.60 (s, 3H), 4.03 (br, 1H), 4.57 (m, 1H), 4.91 (m, 1H), 5.39 (d, *J* = 12.5 Hz, 1H), 5.68 (d, *J* = 12.5 Hz, 1H), 5.97 (d, *J* = 9.0 Hz, 2H), 6.49 (d, *J* = 9.0 Hz, 2H), 6.87-8.13 (m, 16H), 8.51 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.57, 59.56, 64.12, 83.59, 114.57, 114.84, 123.78, 125.16, 126.71, 126.95, 127.01, 128.54, 128.84, 129.14, 129.21, 129.68, 130.10, 131.13, 131.33, 131.57, 132.22, 133.73, 135.66, 138.97, 139.31, 152.31, 198.83; HRMS (ESI) calcd for $C_{37}H_{29}Cl_2NNaO_3$ $(M+Na)^+$ 628.1417, found 628.1450.

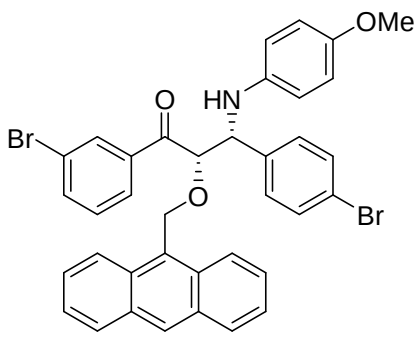


(4l): yield 68%; $[\alpha]_D^{20}$ = -11.0° (c = 1, CH_2Cl_2); 93% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane/isopropanol/EtOH= 450: 25: 25, 254nm, Retention time: t_{major} = 6.8 min, and t_{minor} = 20.8 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 0.55-1.76 (m, 11H), 3.26 (br, 1H), 3.65 (s, 3H), 3.79 (m, 1H), 4.99 (m, 1H), 5.55 (d, *J* = 12.0 Hz, 1H), 5.82 (d, *J* = 12.0 Hz, 1H), 6.09 (d, *J* = 9.0 Hz, 2H), 6.47 (d, *J* = 9.0 Hz, 2H), 7.30-8.41 (m, 13H), 8.52 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 25.50, 26.10, 26.14, 30.10, 30.45, 40.97, 55.72, 61.89, 63.29, 79.16, 114.17, 114.43, 124.43, 125.10, 126.45, 127.95, 128.48, 128.72, 129.01, 131.35, 131.51, 132.67, 136.58, 142.67, 151.32, 200.72; HRMS (ESI) calcd for $C_{37}H_{37}NNaO_3$ $(M+Na)^+$ 566.2666, found 566.2693.

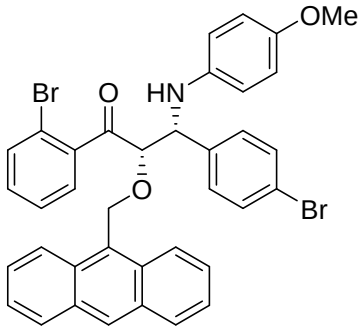


(4m): yield 67%; 94% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/TFA = 90: 10: 0.1, 254nm, Retention time: t_{major} = 12.9 min, and t_{minor} = 25.6 min.); ^{1}H NMR ($CDCl_3$, 500 MHz,): δ (ppm) 3.61 (s, 3H), 4.48 (br, 1H), 4.65 (m, 1H), 4.85 (m, 1H), 5.32 (d, *J* = 11.5 Hz, 1H), 5.62 (d, *J* = 11.5 Hz, 1H), 6.19 (d, *J* = 9.0 Hz, 2H), 6.53 (d, *J* = 9.0 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 7.35-8.01 (m, 12H), 8.47 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz,): δ (ppm) 55.59, 59.78, 64.46, 84.29, 114.56, 114.88, 121.26, 123.90, 125.06, 126.51, 126.60, 128.58, 128.98, 129.04, 129.80, 131.08, 131.21, 131.42, 134.14, 138.52, 139.83, 140.22, 152.26,

197.72; HRMS (ESI) calcd for $C_{37}H_{29}BrClNNaO_3$ $(M+K)^+$ 672.0912, found 672.0895.

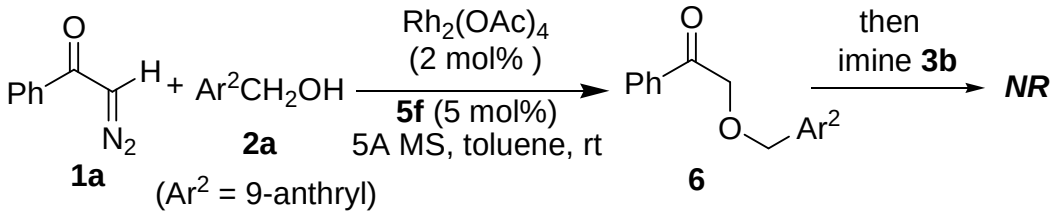


(4n): yield 77%; 92% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane/isopropanol/EtOH/TFA = 500: 20: 20: 1, 254nm, Retention time: t_{major} = 18.3 min, and t_{minor} = 40.0 min.); ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm) 3.61 (s, 3H), 4.46 (br, 1H), 4.65 (m, 1H), 4.85 (m, 1H), 5.32 (d, *J* = 12.0 Hz, 1H), 5.63 (d, *J* = 12.0 Hz, 1H), 6.20 (d, *J* = 8.8 Hz, 2H), 6.53 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.24-8.01 (m, 12H), 8.47 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 55.60, 59.75, 64.54, 84.24, 114.57, 114.98, 121.31, 123.07, 123.87, 125.08, 126.59, 126.73, 128.62, 128.99, 129.08, 130.18, 131.09, 131.21, 131.42, 131.45, 136.12, 137.58, 138.45, 140.18, 152.32, 197.63; HRMS (ESI) calcd for $C_{37}H_{29}Br_2NNaO_3$ $(M+Na)^+$ 716.0406, found 716.0351.

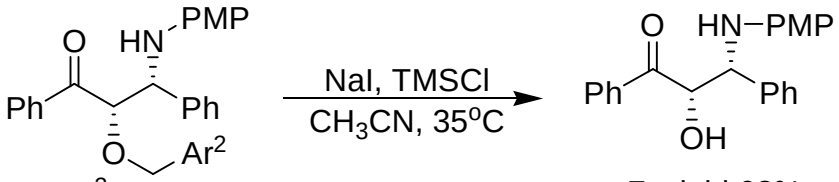


(4o): yield 74%; 87% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane/isopropanol/EtOH = 450: 25: 25, 254nm, Retention time: t_{major} = 15.8 min, and t_{minor} = 37.6 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.68 (s, 3H), 4.03 (br, 1H), 4.70 (m, 1H), 5.06 (m, 1H), 5.46 (d, *J* = 12.0 Hz, 1H), 5.67 (d, *J* = 12.0 Hz, 1H), 6.11 (d, *J* = 8.5 Hz, 2H), 6.58 (d, *J* = 8.5 Hz, 2H), 6.80-8.19 (m, 16H), 8.53 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 57.01, 60.66, 66.46, 86.67, 116.02, 116.48, 120.07, 121.06, 123.09, 125.42, 126.53, 128.09, 128.43, 128.64, 130.51, 130.60, 130.68, 131.35, 132.57, 132.78, 133.42, 135.47, 138.72, 140.10, 140.89, 153.78, 203.55; HRMS (ESI) calcd for $C_{37}H_{29}Br_2NNaO_3$ $(M+Na)^+$ 716.0406, found 716.0422.

Control experiment of 6 with 3b was carried out in the strand conditions.



Deprotection of the product 4b: [4]

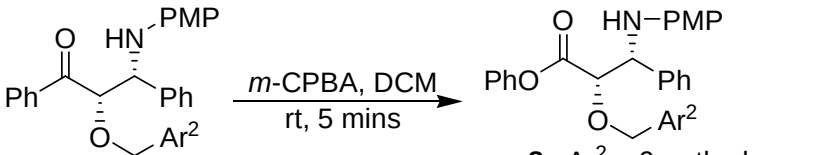


4b, Ar^2 = 9-anthryl
91% *ee*

7, yield 68%
$[\alpha]_D^{25}$ = 8.7° (c = 1.0, $CHCl_3$)

To a solution of compound **4b (**0.1 mmol) and NaI (30mg, 2eq) in 1.0 mL of CH_3CN was added 30 μL TMSCl (2 ~ 2.5eq) via a syringe pump at room temperature under an argon atmosphere. The reaction temperature was warmed to 30 °C and stirred over night. The reaction mixture was poured into water and stirred for 10 min. The aqueous phase was extracted with EtOAc. The organic phase was separated, washed with saturated aqueous $Na_2S_2O_3$, and dried over anhydrous $MgSO_4$. After evaporating the solvents, the crude product was purified by flash chromatography on silica gel (eluent: EtOAc/light petroleum ether = 1:50~1:30) to give compound **7** in 68% yield. After recrystallization from CH_2Cl_2, EtOAc and light petroleum ether, give the optical pure product in 47% yield. $[\alpha]_D^{25}$ = 8.7° (c =1.0, $CHCl_3$,); 99% ee, determined by HPLC (Daicel Chirapak OD, flow rate 0.9 mL/min, hexane/isopropanol = 90:10, 254nm, Retention time: t_{minor} = 17.8 min, and t_{major} = 20.5 min.); ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.68 (s, 3H), 4.86 (d, *J* = 2.7 Hz, 1H), 5.59 (d, *J* = 2.7 Hz, 1H), 6.55 (d, *J* = 9.0 Hz, 2H), 6.69 (d, *J* = 9.0 Hz, 2H), 6.92-7.18 (m, 5H), 7.53-7.90 (m, 5H); HRMS (ESI) calcd for $C_{22}H_{21}NNaO_3$ $(M+Na)^+$ 370.1419, found 370.1408. Reference Data: [4] $[\alpha]_D^{25}$ = -9.58° (c = 0.748, $CHCl_3$, 99% ee); syn diastereomer: t_{major} = 18.1 min, and t_{minor} = 20.9 min, (Chiralcel OD, 254 nm, heptane/*i*-PrOH = 90:10, 0.9 mL/min); ^{1}H NMR ($CDCl_3$, 300 MHz): δ (ppm) 3.66 (s, 3 H), 4.82 (dd, *J* = 1.9, 6.8 Hz, 1 H), 5.58 (br s, 1 H), 6.54 (d, *J* = 8.8 Hz, 2 H), 6.66 (d, *J* = 8.8 Hz, 2 H), 7.14 (m, 2 H), 6.90 (m, 2 H), 7.52 (t, *J* = 7.8 Hz, 3 H), 7.88 (m, 3 H).

Oxidation of the product 4b: [5]



4b, Ar^2 = 9-anthryl
91% *ee*

8, Ar^2 = 9-anthryl
yield 53% (35% **4b** recovered)

To a solution of compound **4b** (0.19 mmol) in 2.0 mL of CH_2Cl_2 was added $NaH_2PO_4 \cdot 2H_2O$ (106 mg, 0.76 mmol), and *m*-CPBA (98 mg, 0.57 mmol) was added in portions at room temperature. About 5 min later, the reaction mixture was quenched by aqueous $Na_2S_2O_3$, and extracted with ether, washed with cold aqueous $NaHCO_3$ and aqueous NaCl in sequence. Then the organic phase was dried over

anhydrous $MgSO_4$. After evaporating the solvents, the crude product was purified by flash chromatography on silica gel (eluent: EtOAc/light petroleum ether = 1:80~1:50) to give compound **8** in 53% yield (with 35% of the material recovered). ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 3.63 (s, 3H), 4.85 (d, *J* = 3.2 Hz, 1H), 5.10 (d, *J* = 3.2 Hz, 1H), 5.12 (d, *J* = 10.8 Hz, 2H), 5.46 (d, *J* = 10.8 Hz, 2H), 6.19 -6.97 (m, 4H), 7.19 -8.03 (m, 18H), 8.39 (s, 1H); HRMS (ESI) calcd for $C_{37}H_{31}NaO_4$ $(M+Na)^+$ 576.2151, found 576.2137.

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1 a) M. P. Doyle, M. A. McKervey, T. Ye, in *Modern Catalytic Methods for Organic Synthesis with Diazo Compounds*, John Wiley & Sins, New York, **1998**, pp. 1-46.

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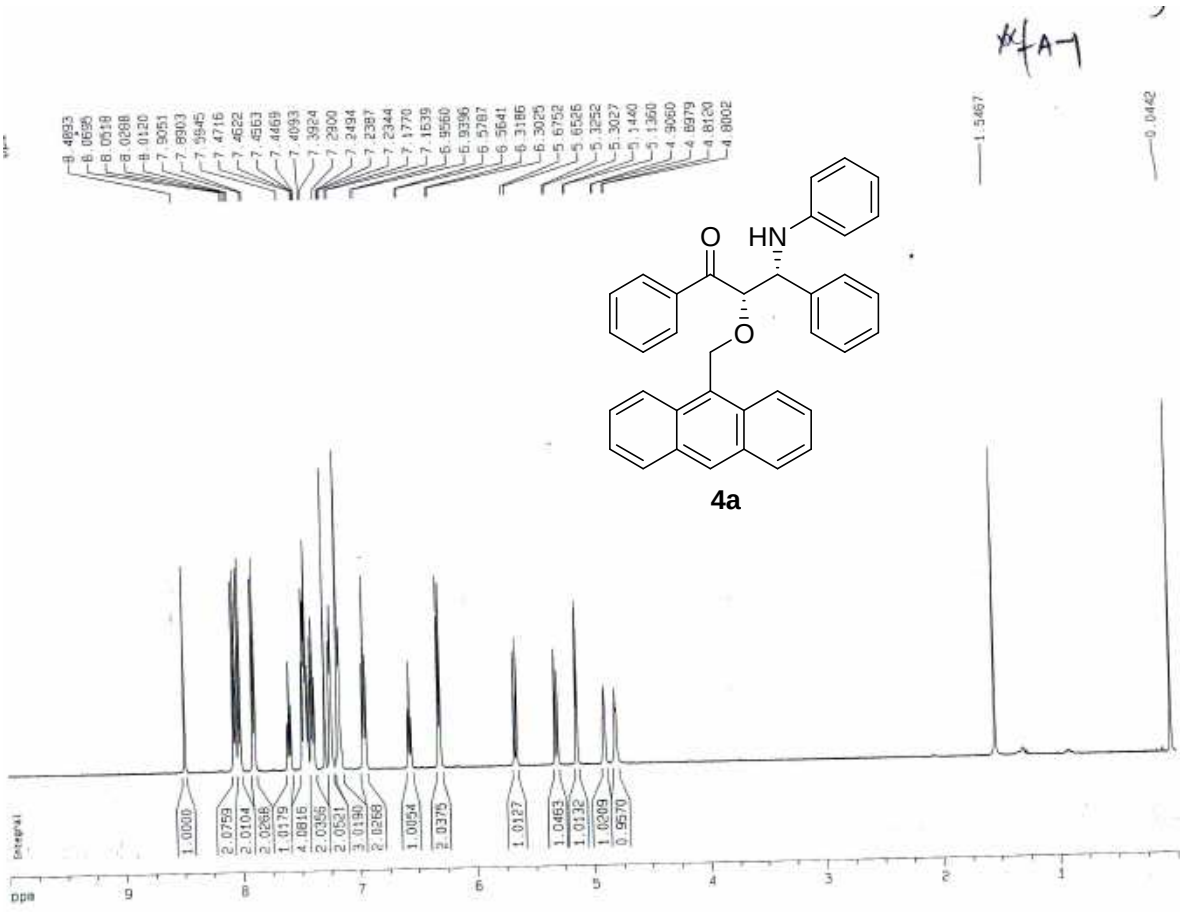
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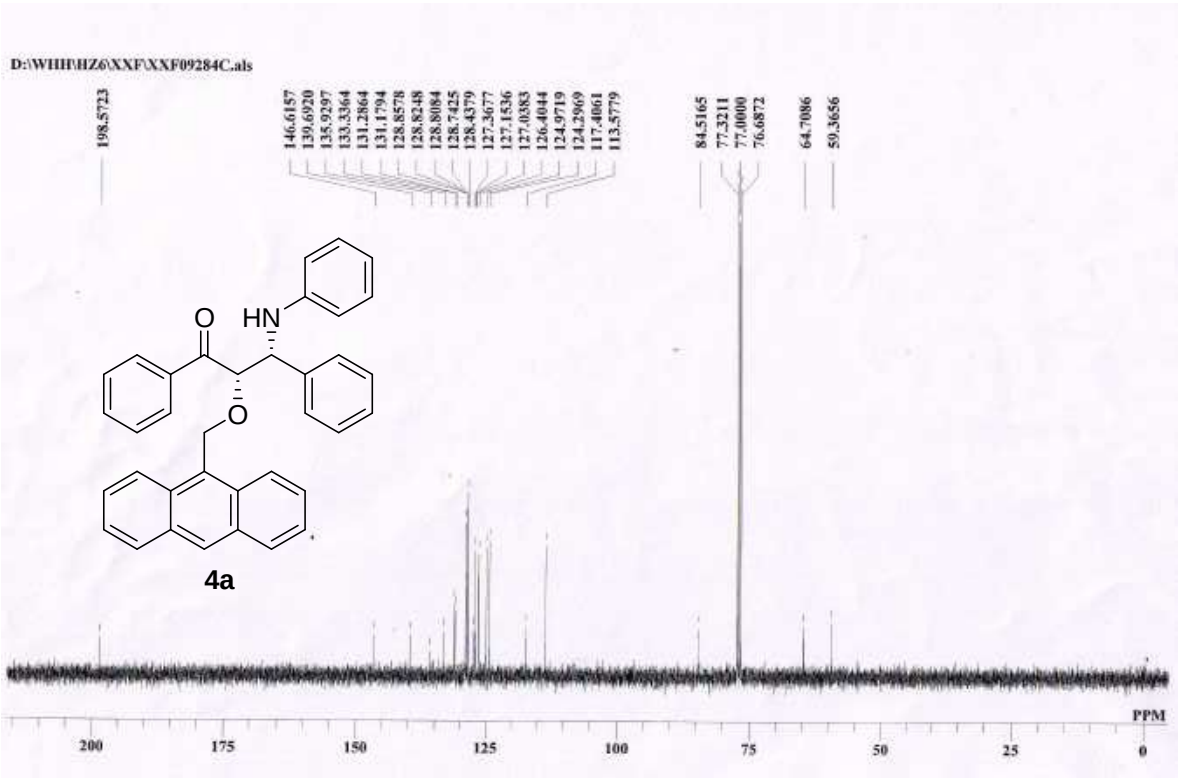
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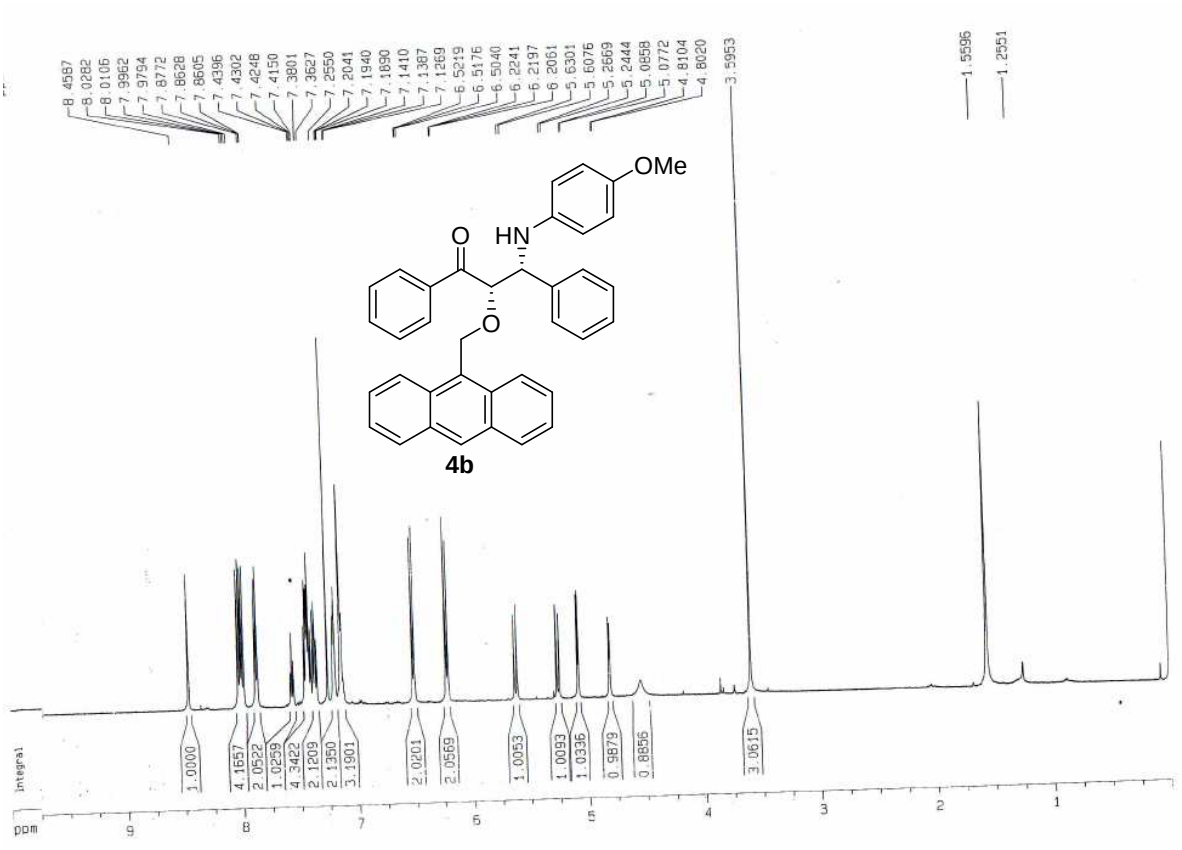
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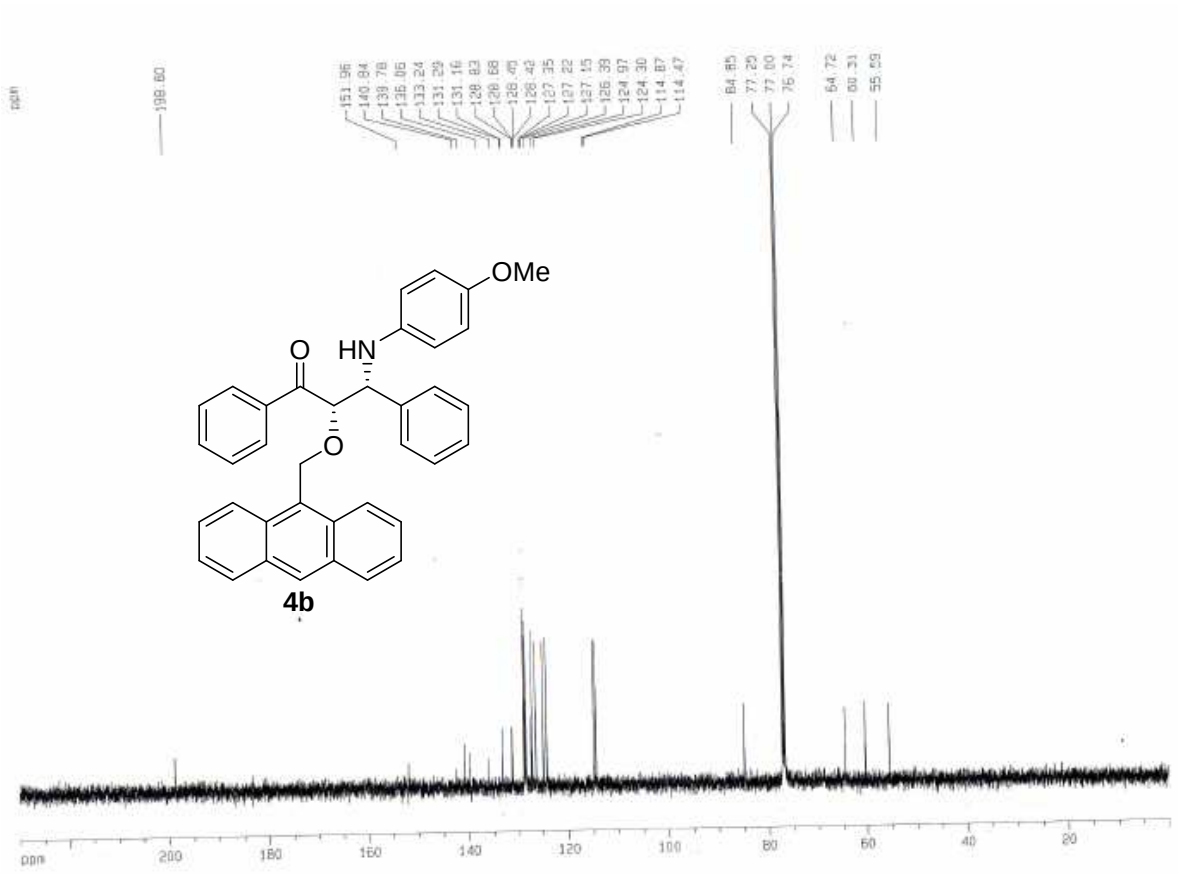
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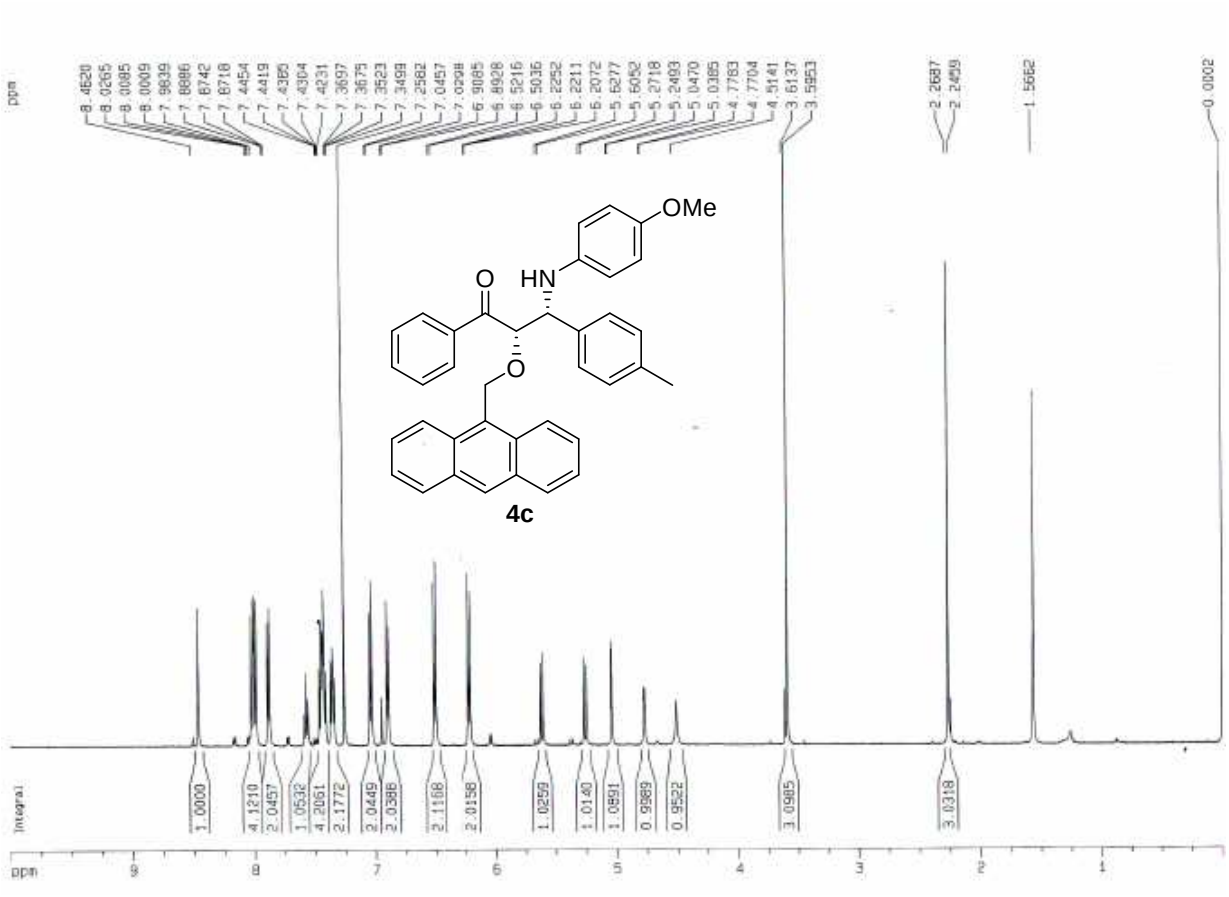
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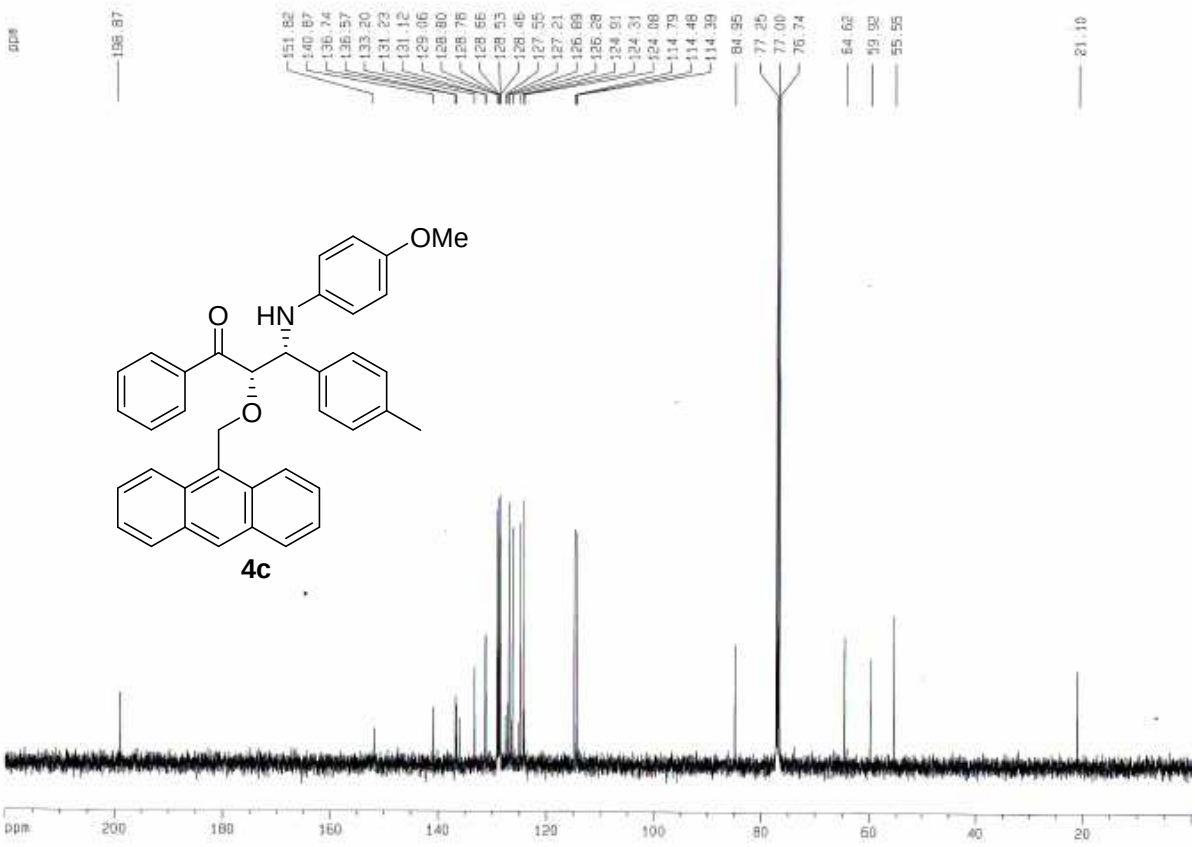
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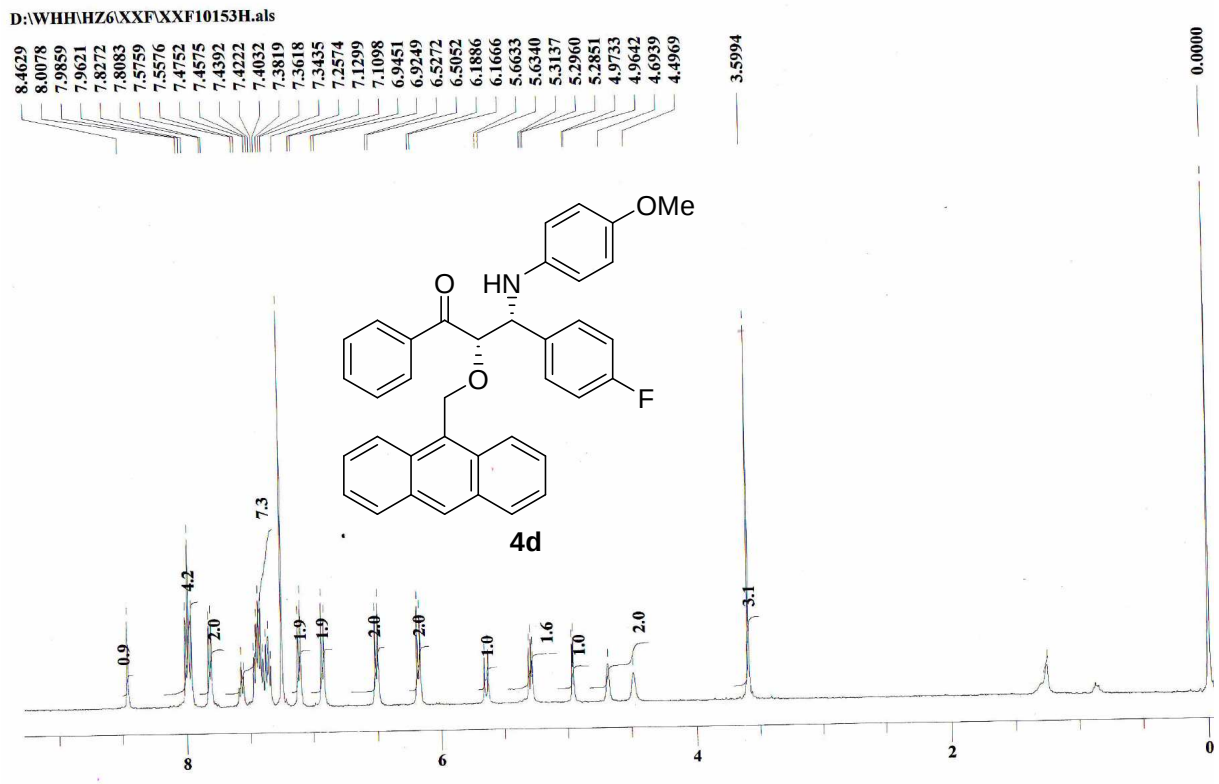
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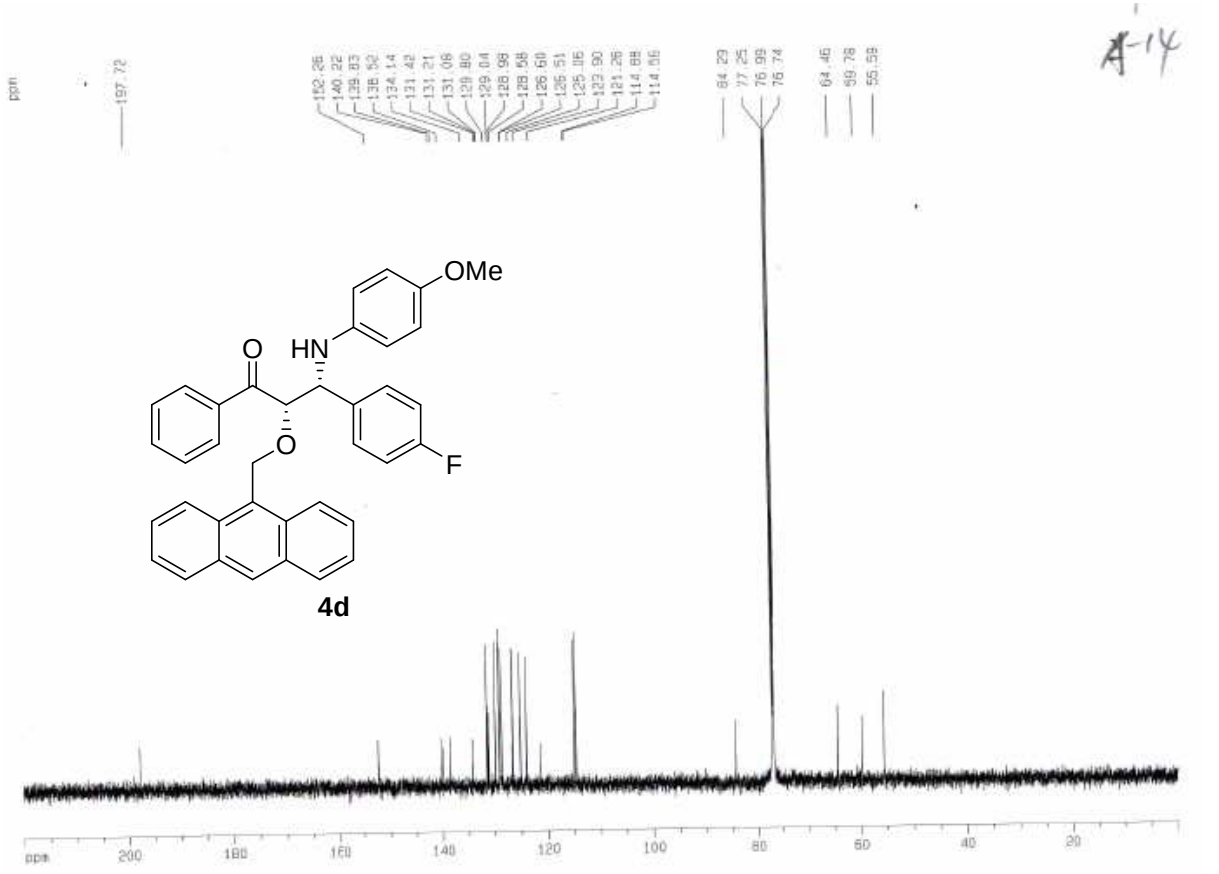
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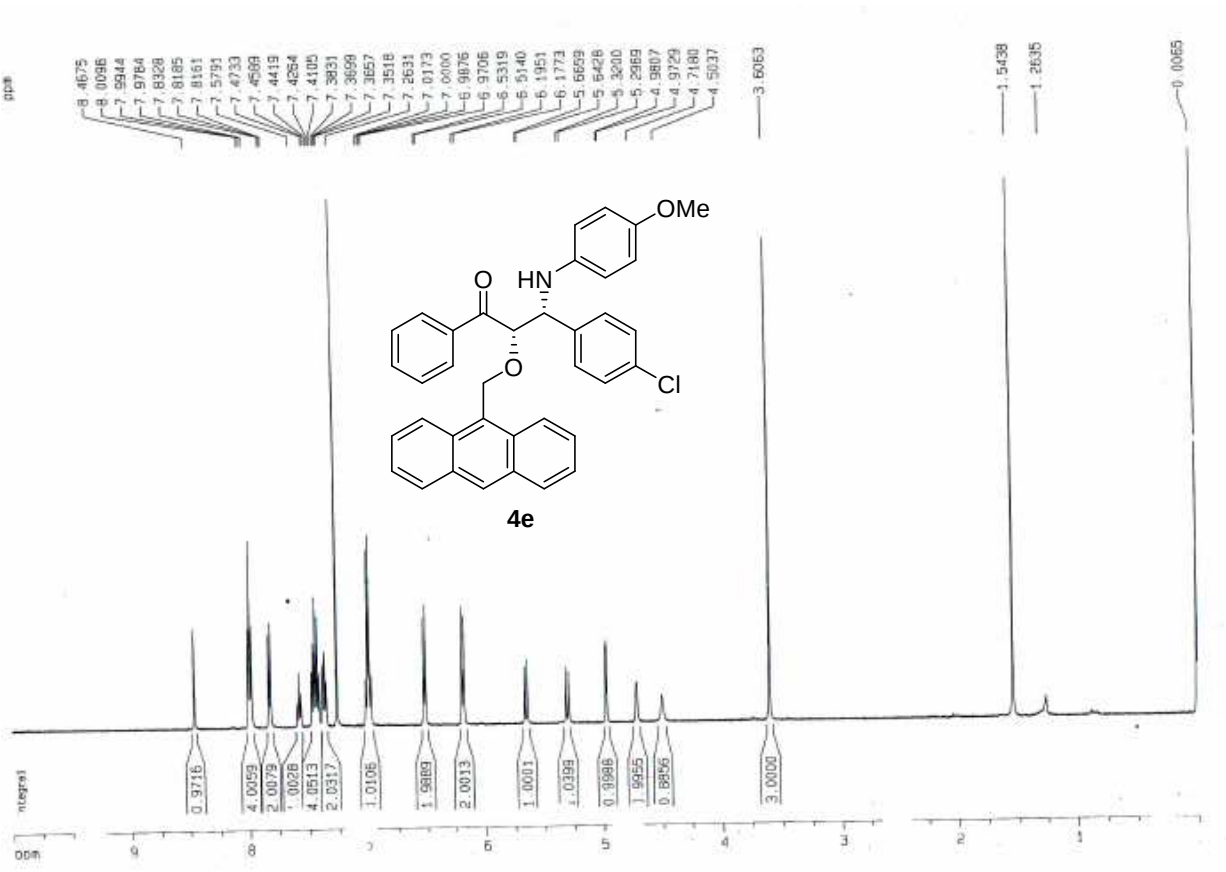

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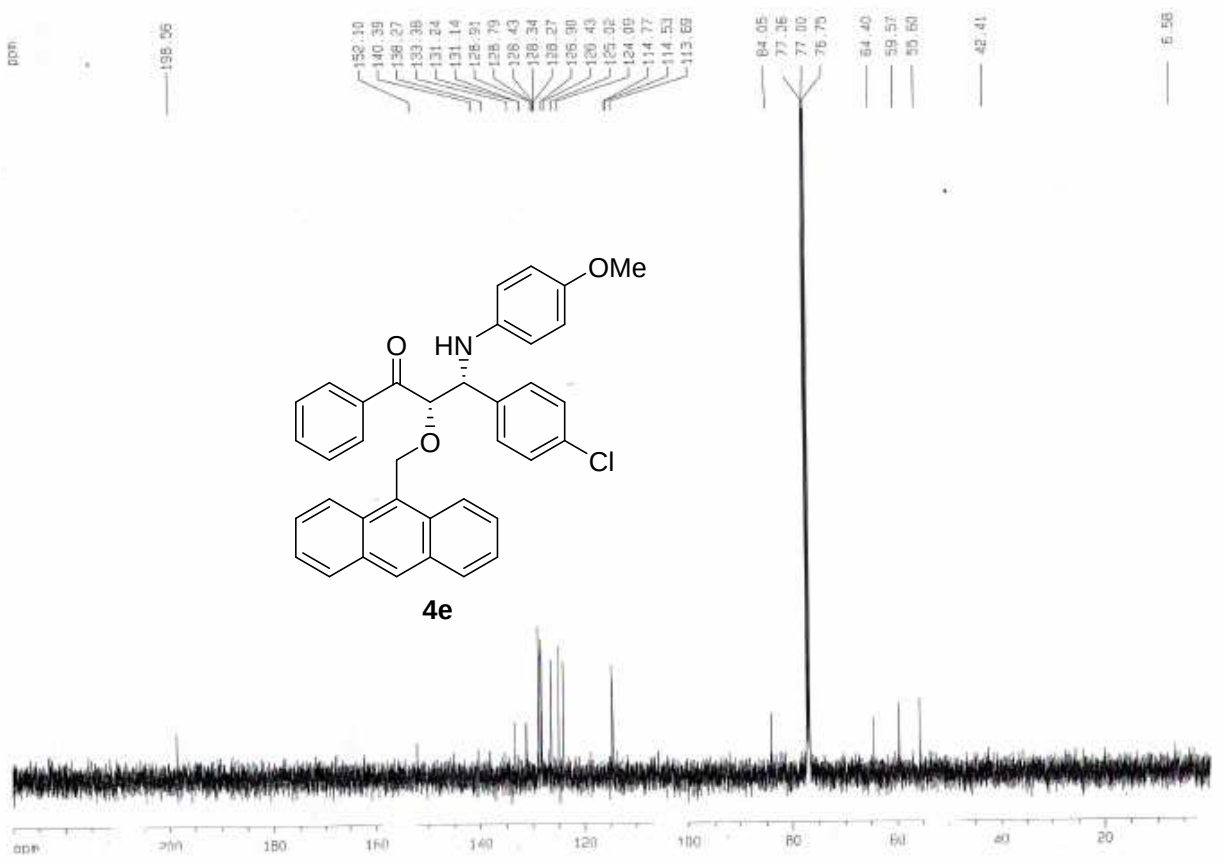

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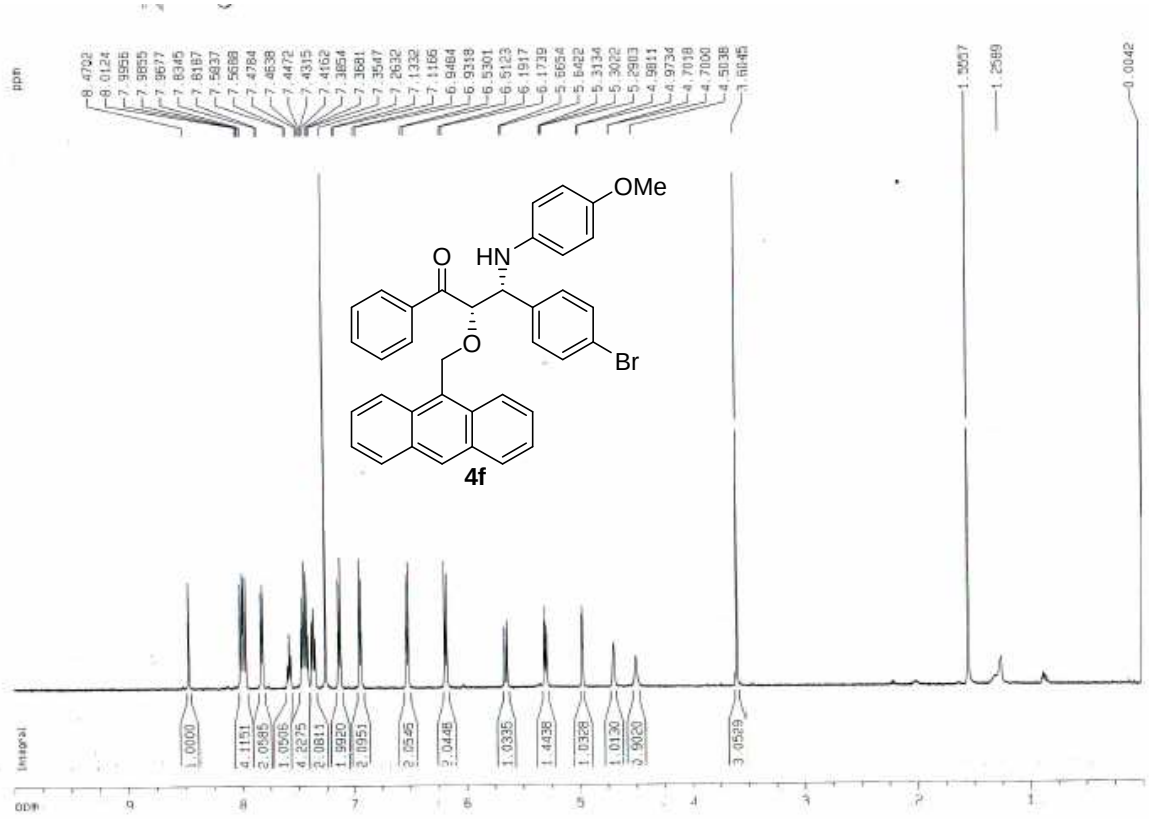
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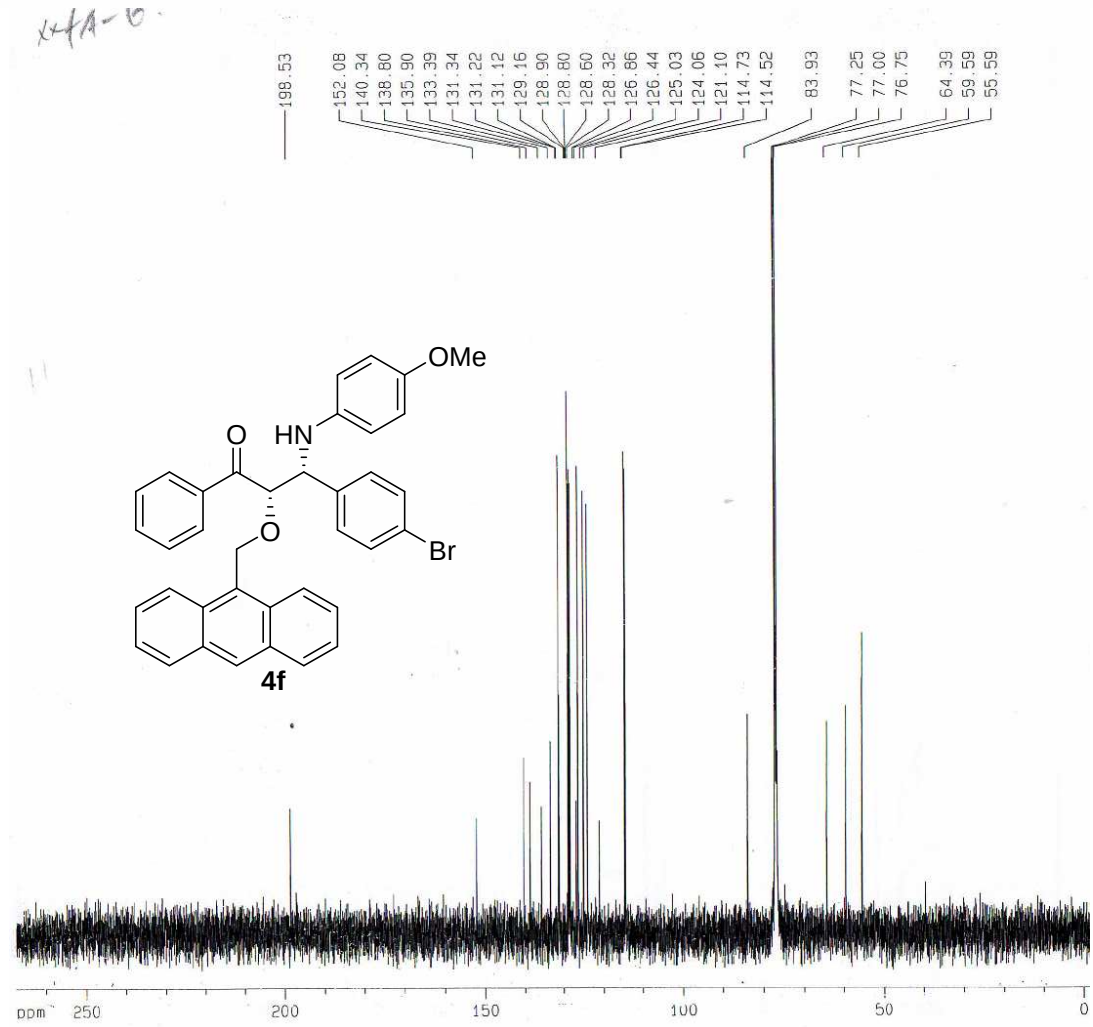
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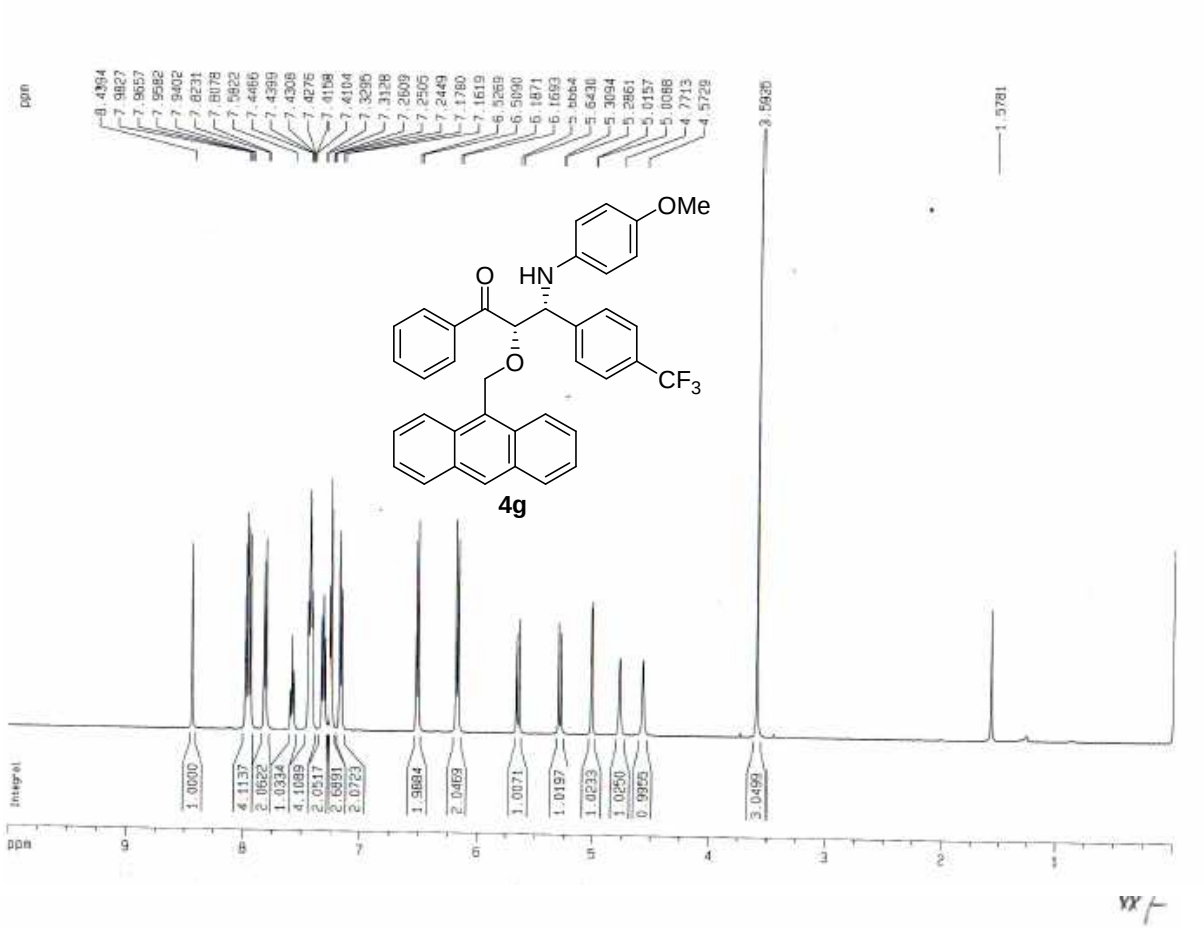
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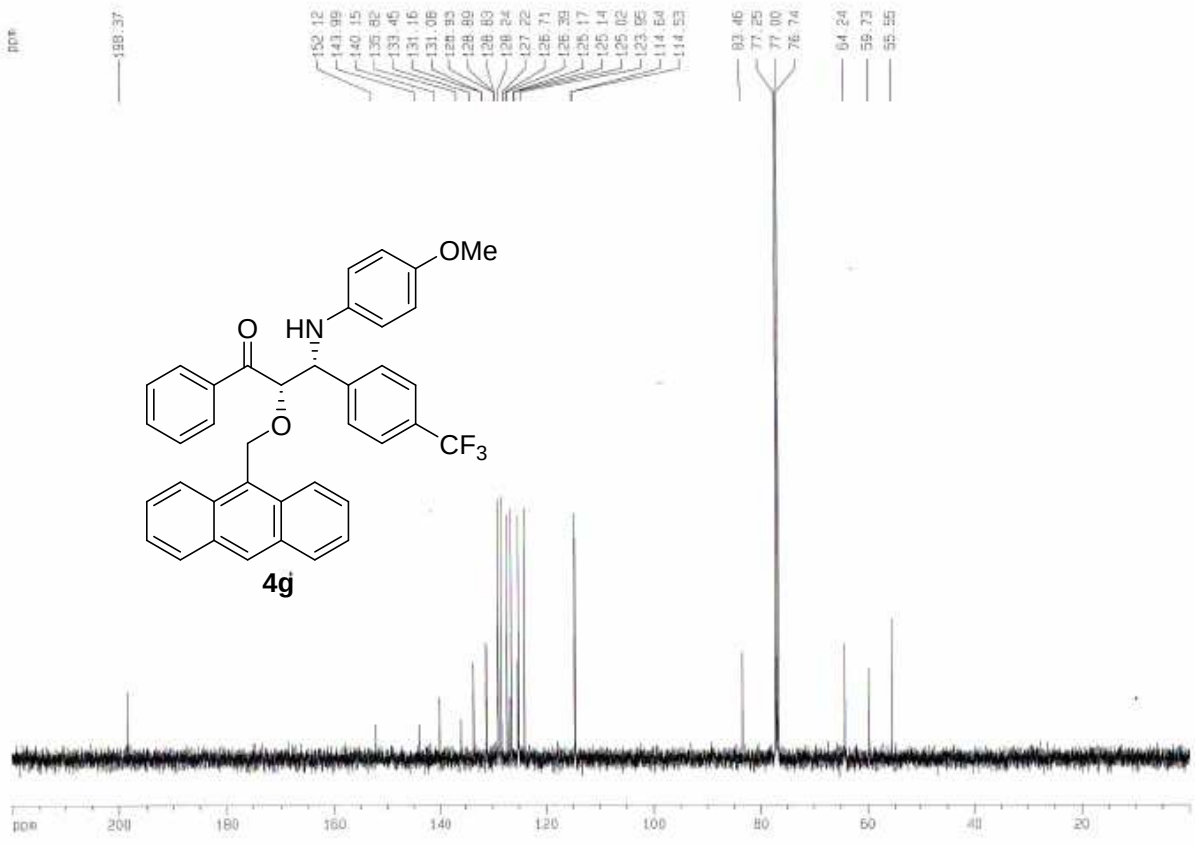
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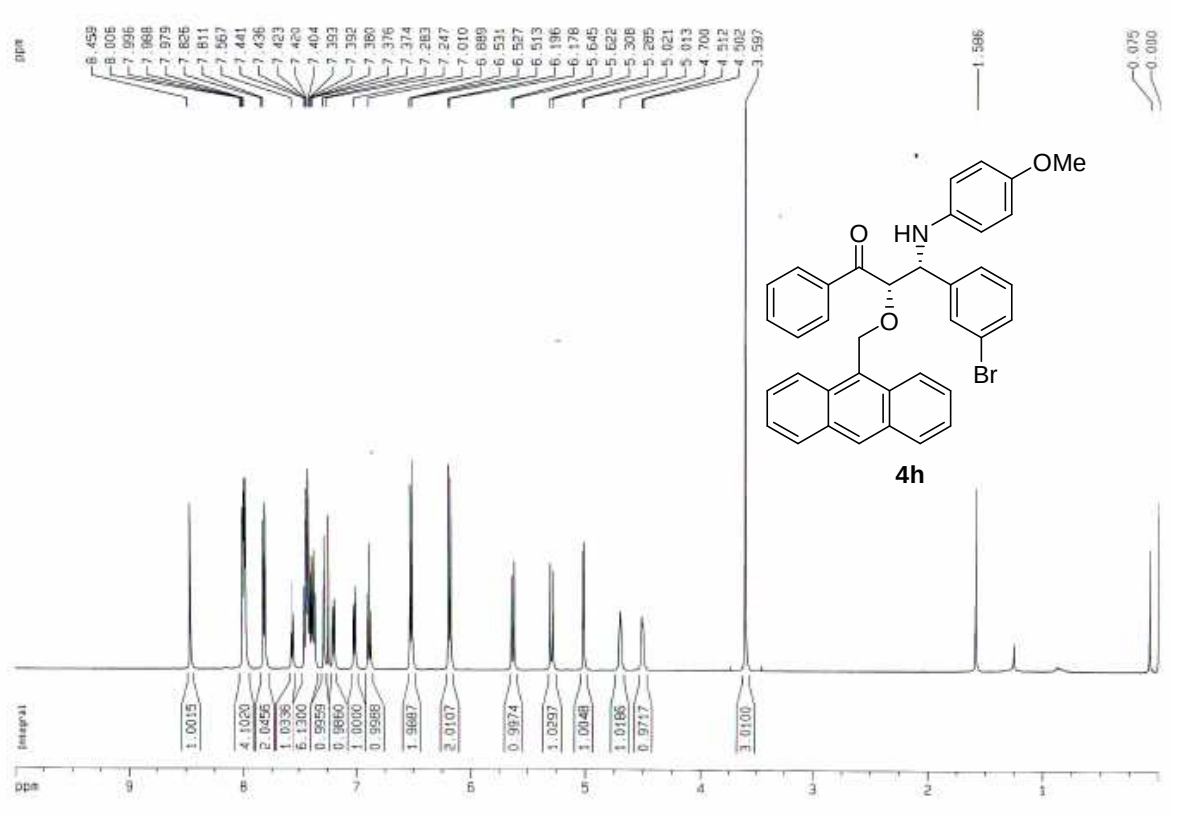

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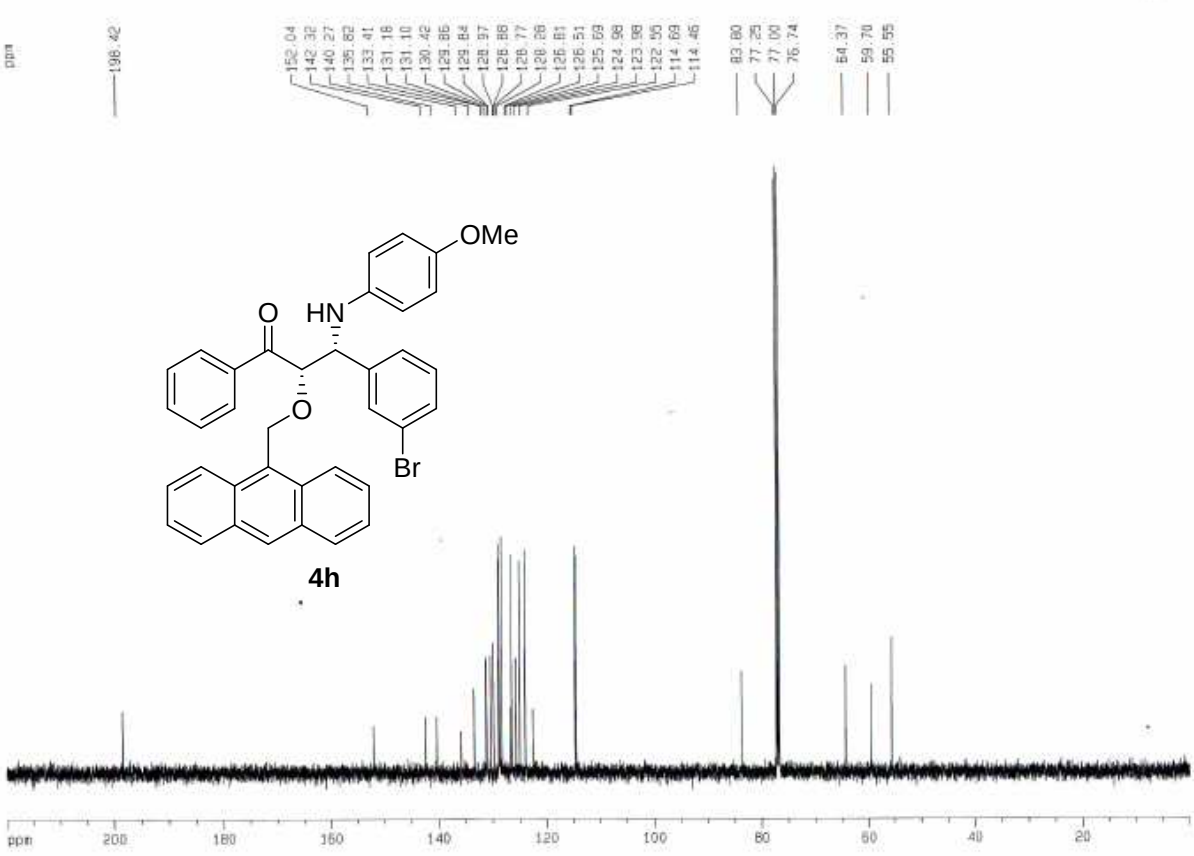

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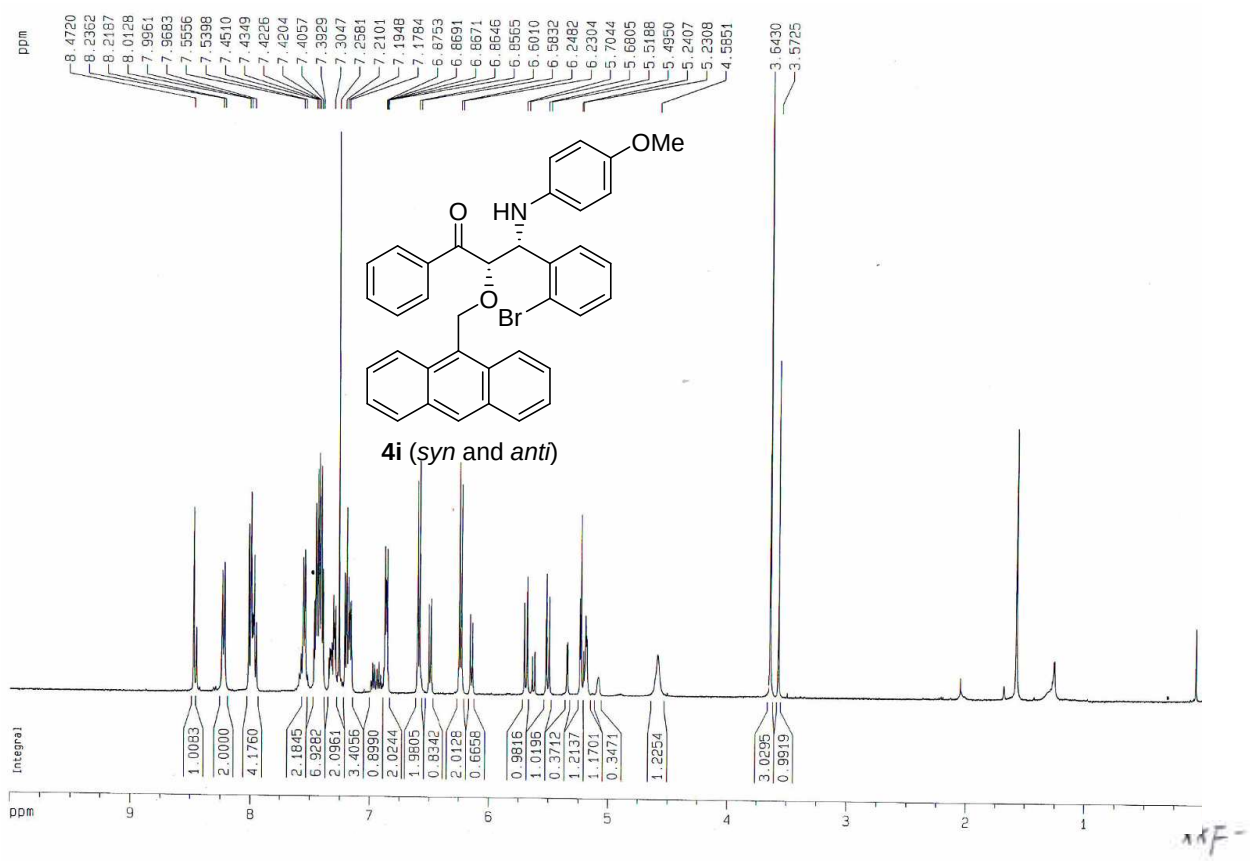

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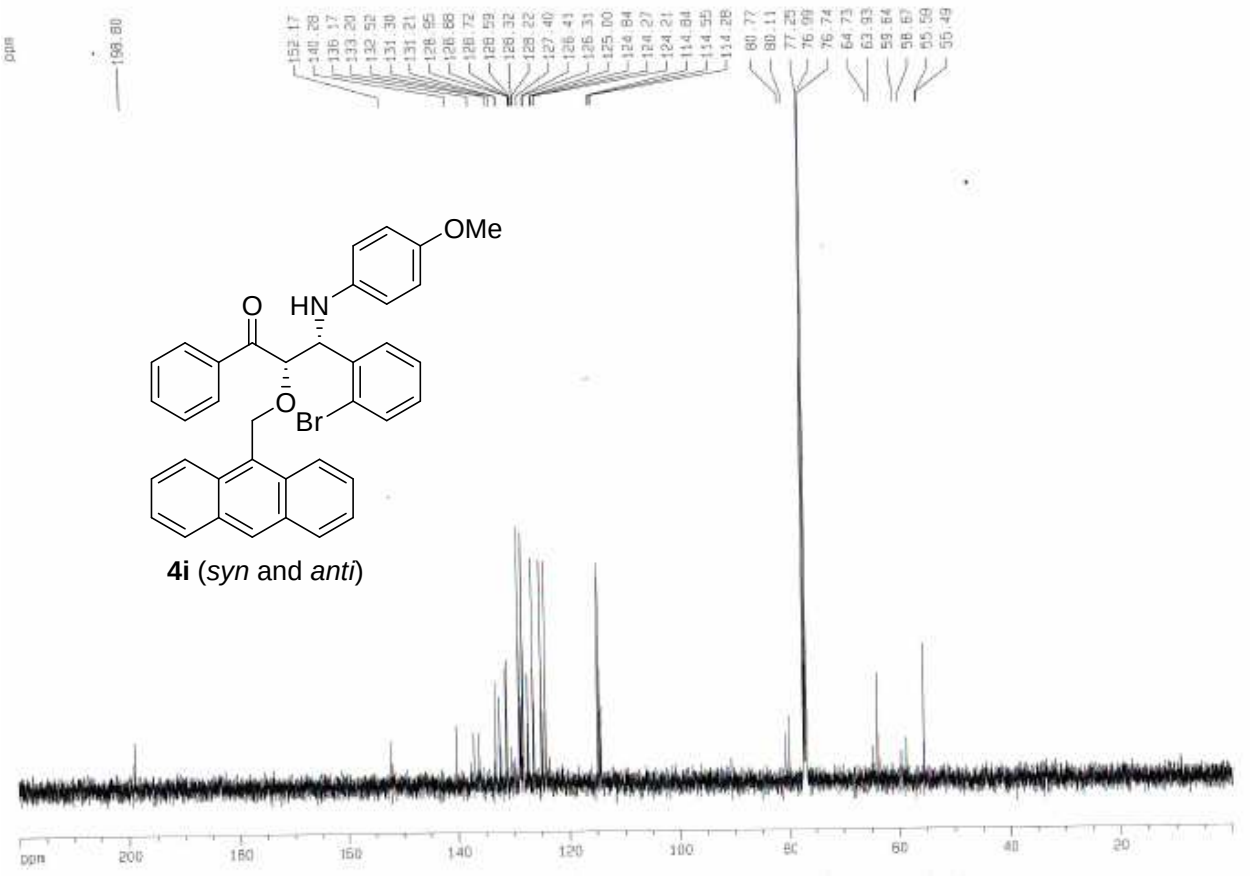

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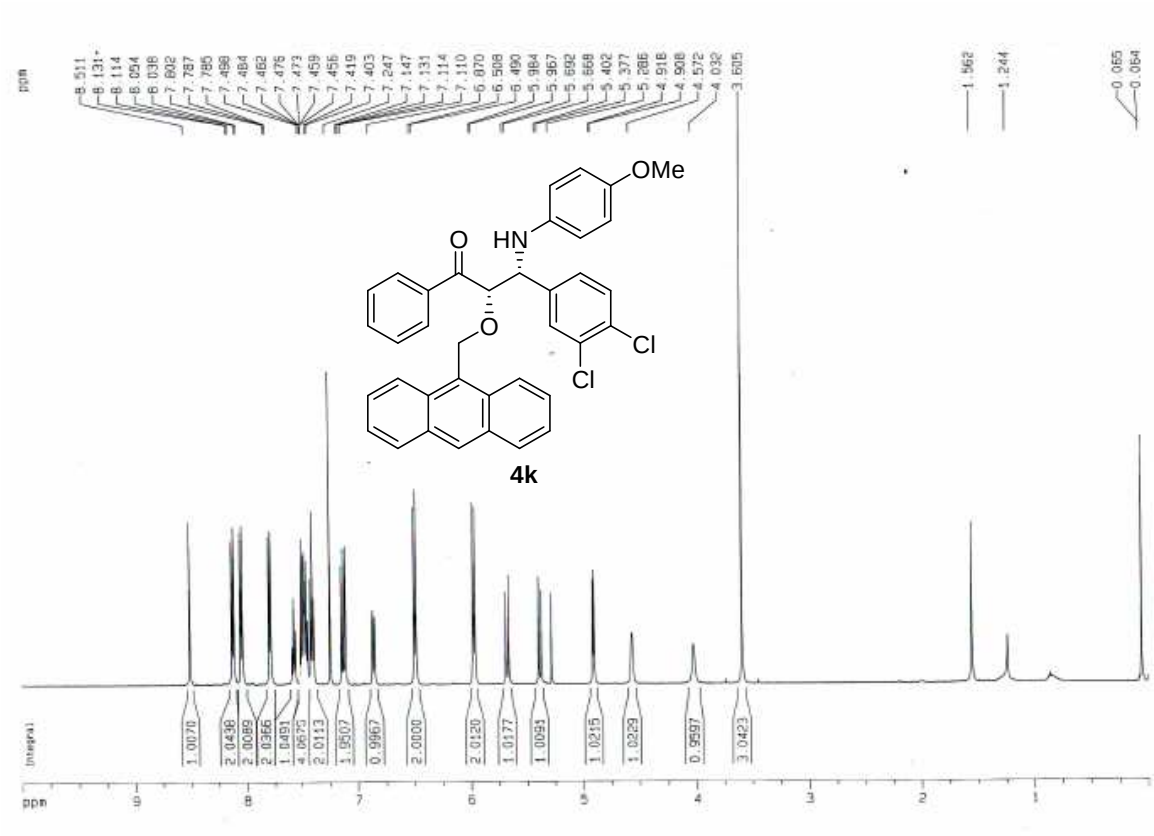
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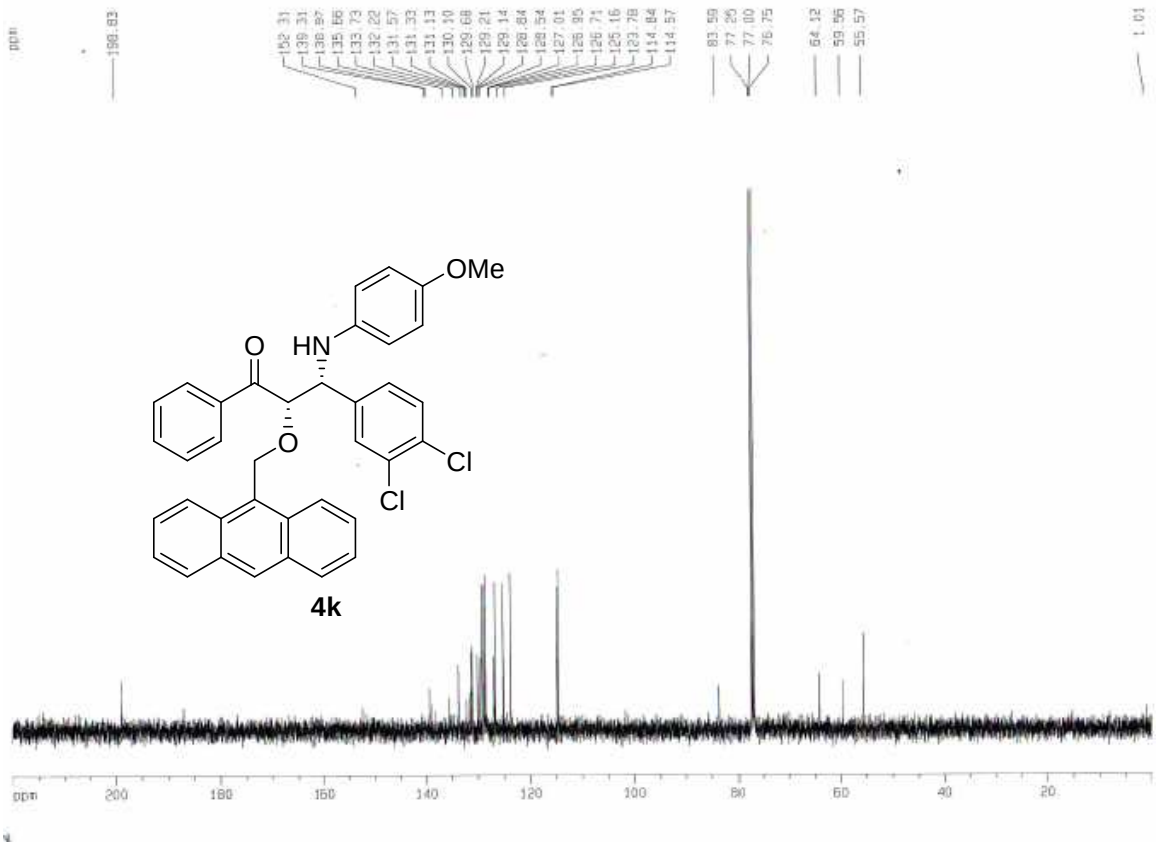
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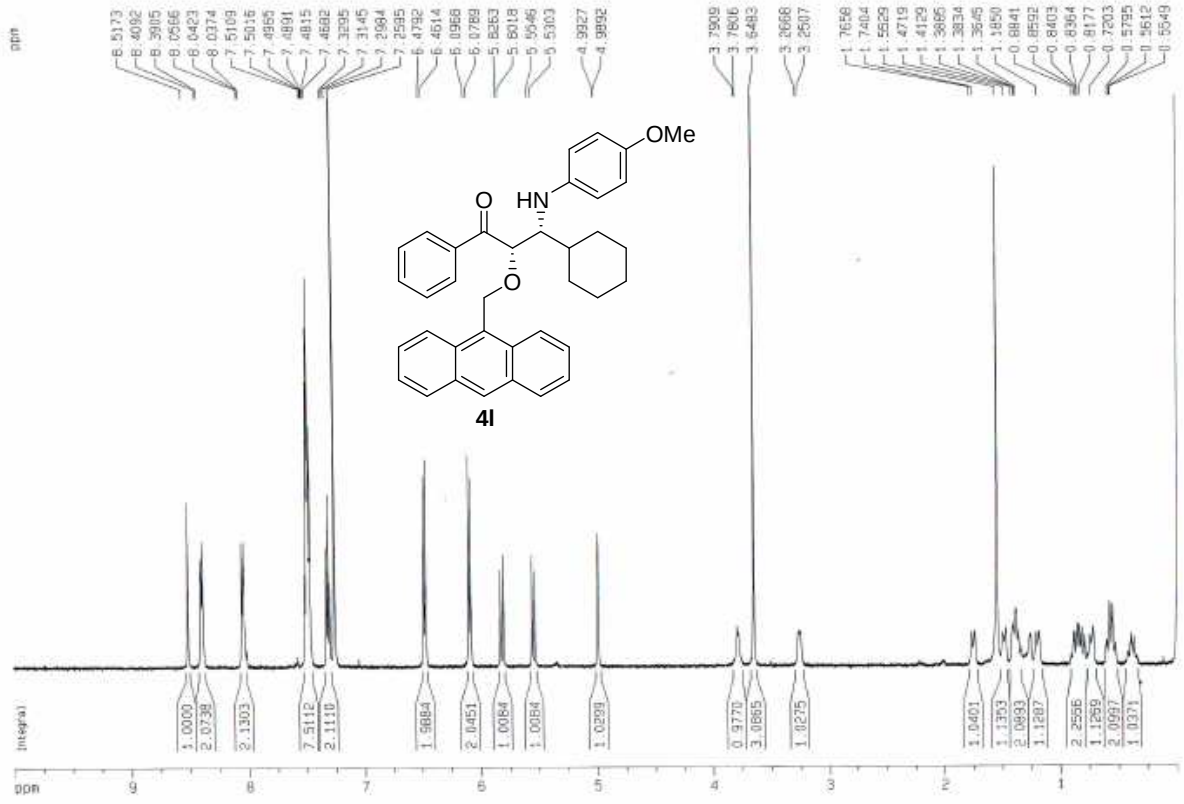
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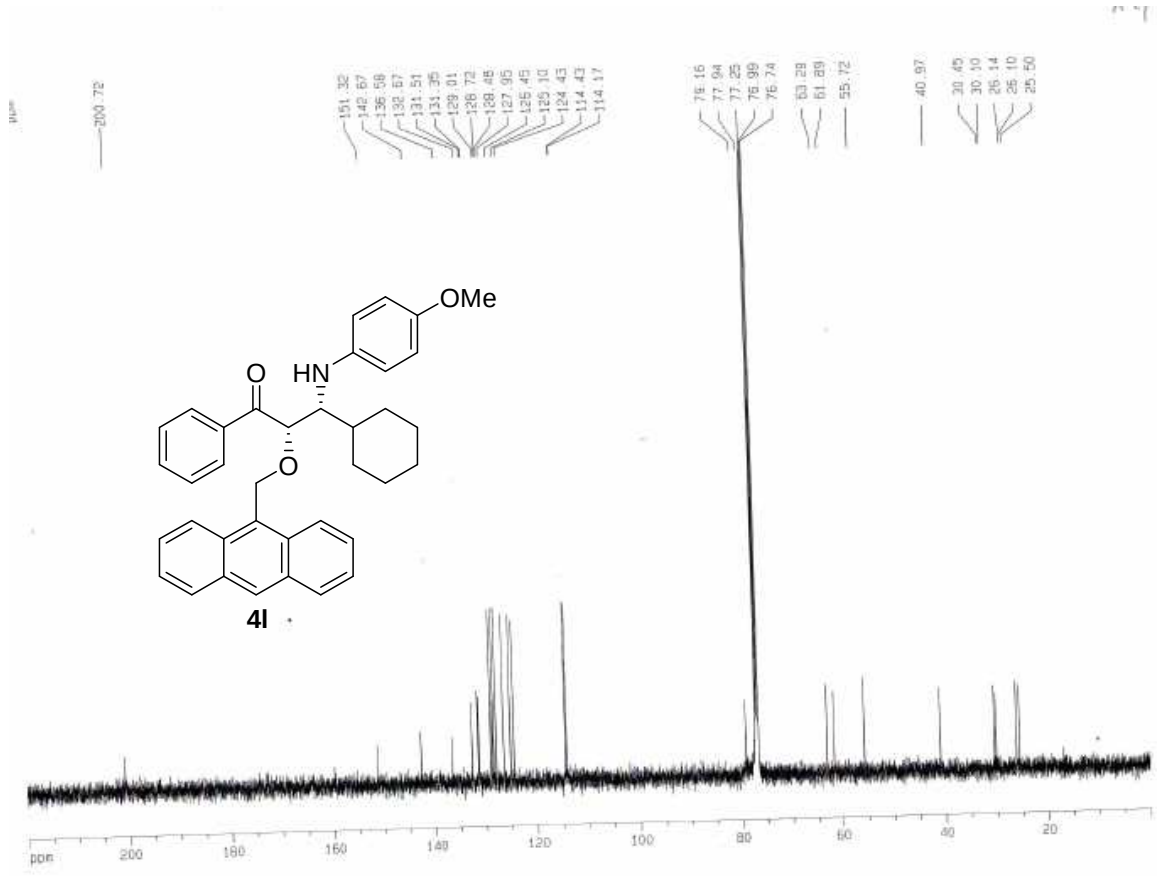
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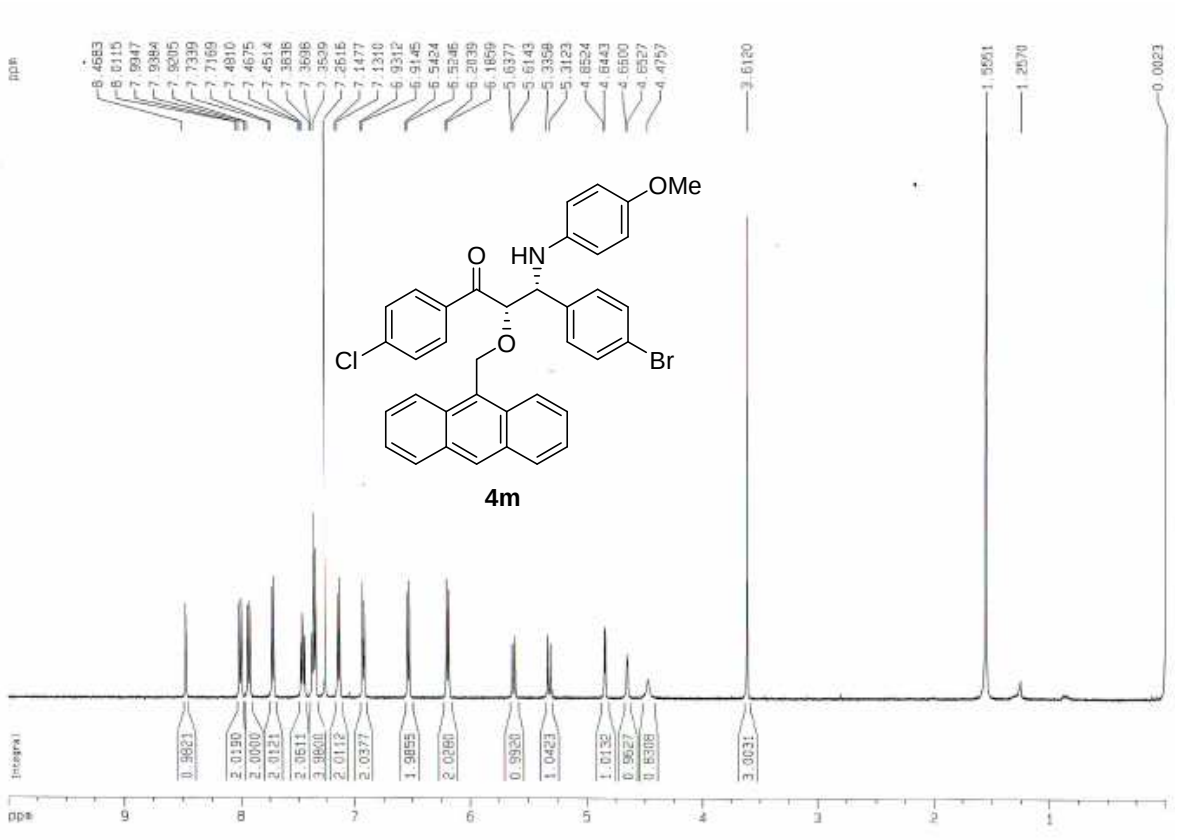

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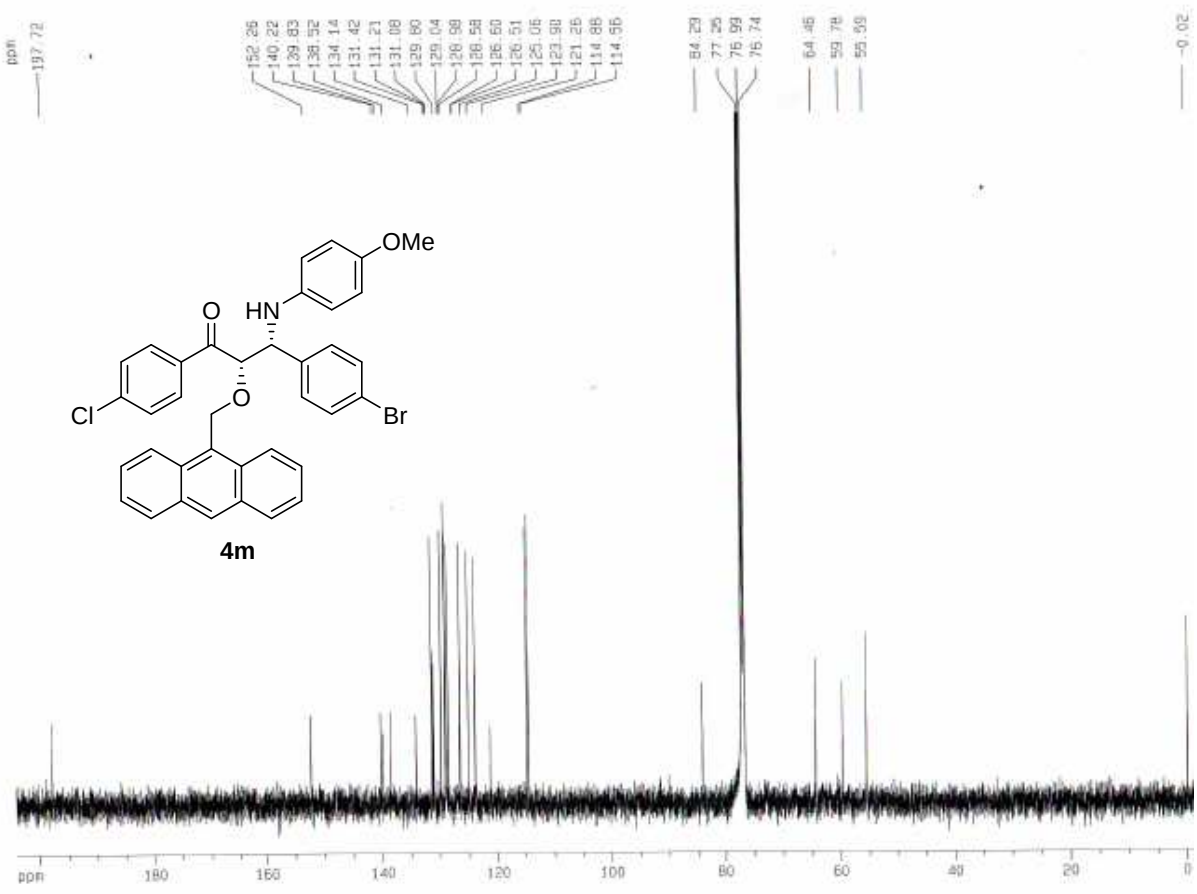

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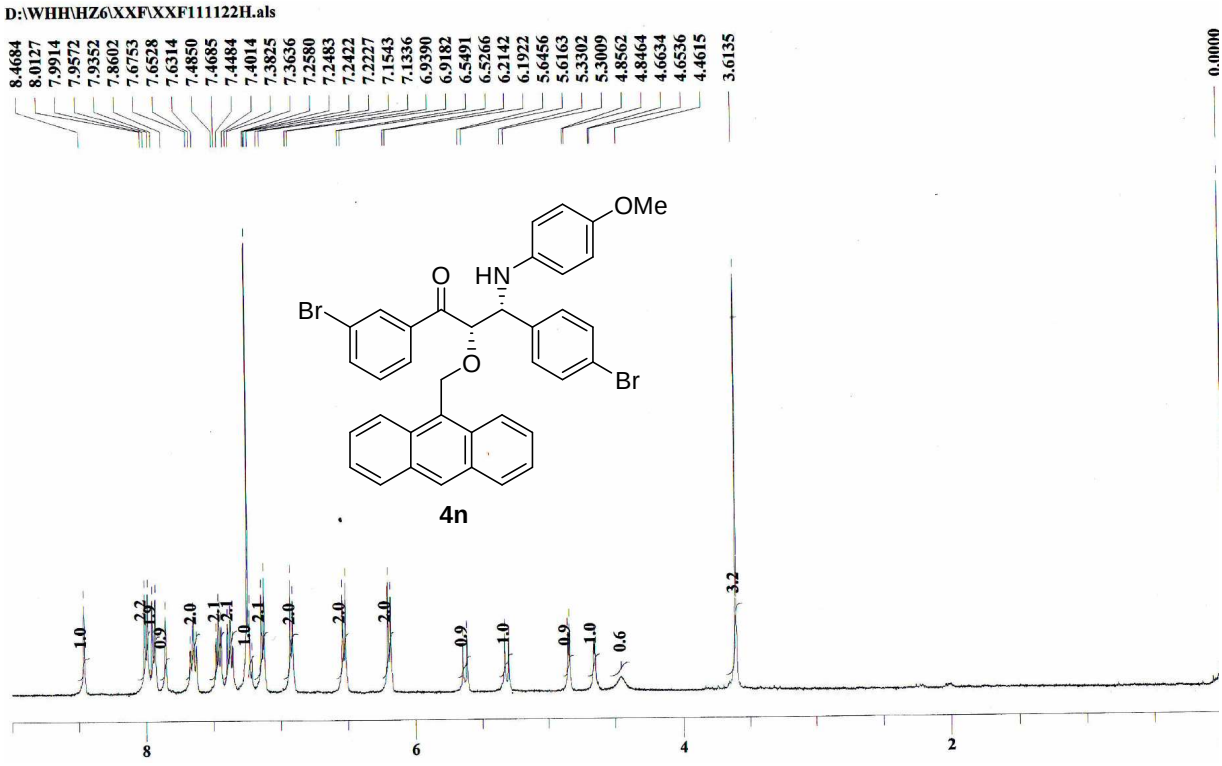
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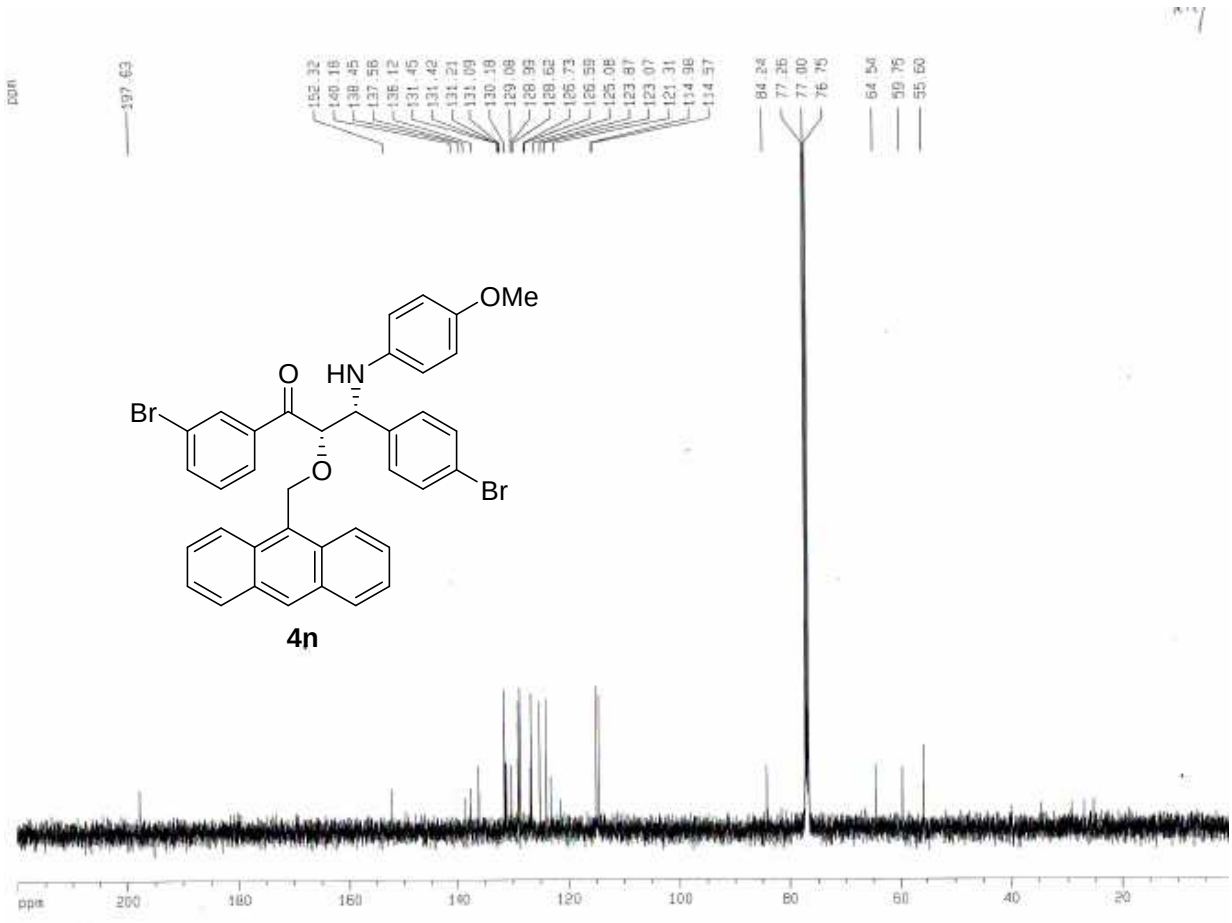
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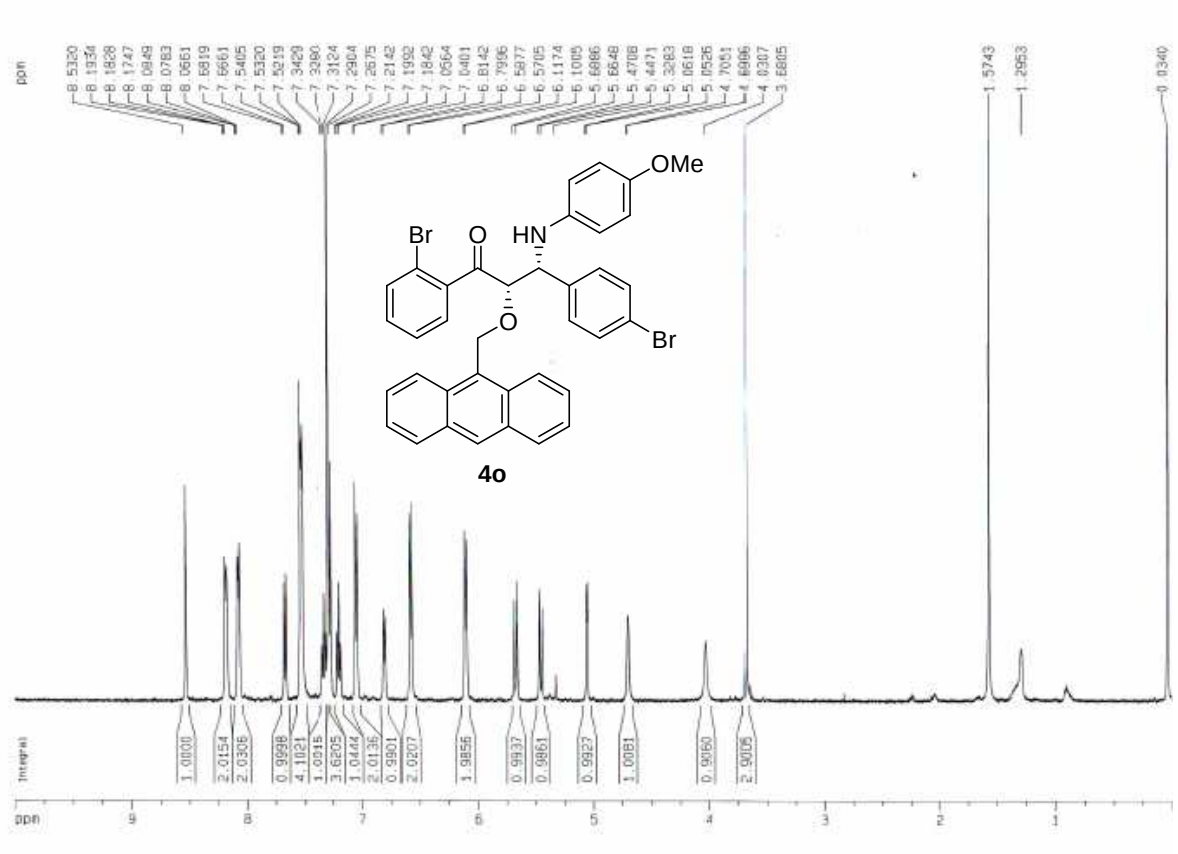
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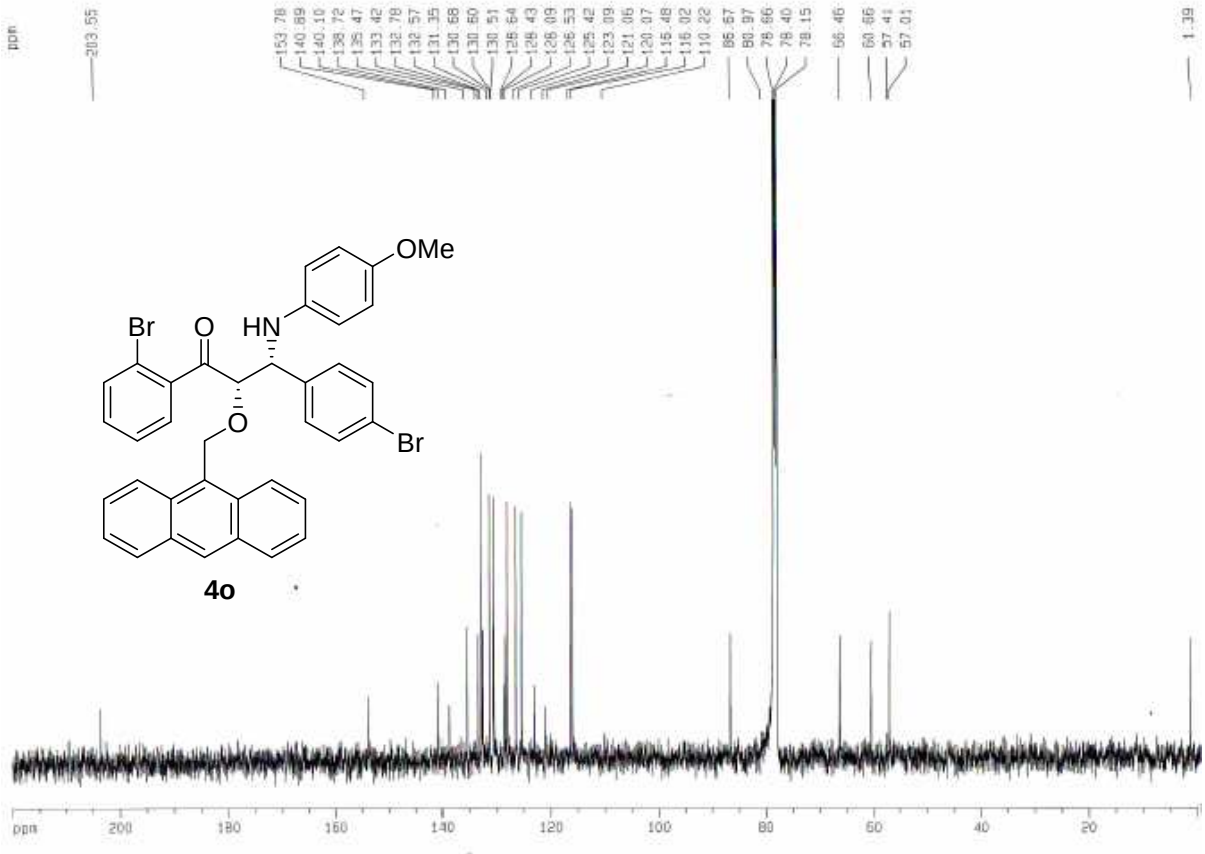
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xxfA-1-race

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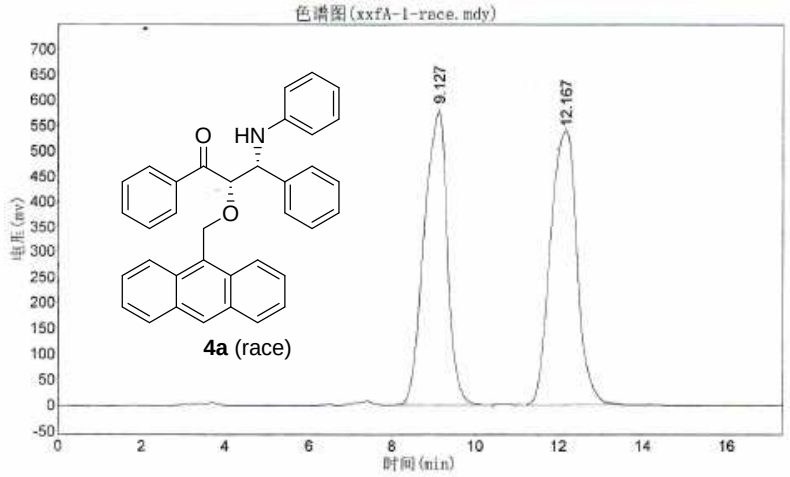


分析结果表

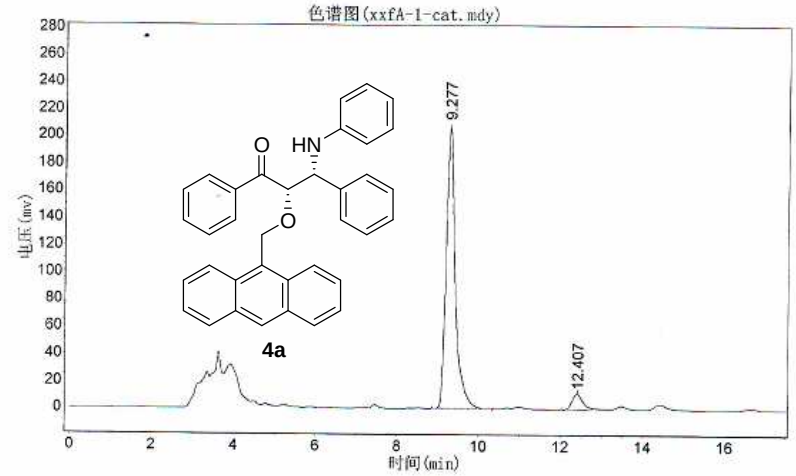
峰号	峰名	保留时间	峰高	峰面积	含量
1		9.127	574347.875	22463364.000	48.7741
2		12.167	538043.500	23592538.000	51.2259
总计			1112391.375	46055902.000	100.0000

xxfA-1-cat

实验单位：ecnu
实验时间：2009-10-11,18:04:05
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-1-cat.mdy

实验者：xxf
报告时间：2009-10-12,21:13:42
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex/i-prOH:EtOH=450：25:25
Detection:254nm
flow:1.0ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.277	205792.047	3239726.000	95.8144
2		12.407	11293.880	199999.500	4.1856
总计			217085.927	3439725.500	100.0000

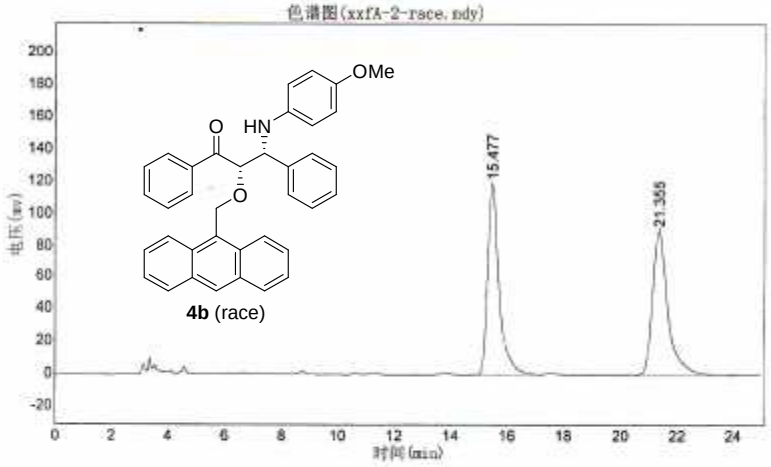
xxfA-2-race

实验单位：ecnu
实验时间：2009-10-12,20:55:43
谱图文件:C:\湖大智达\N2000\ecnu\xxf\xxfA-2-race.mdy

实验者：xxf
报告时间：2009-10-12,20:55:45
积分方法:面积归一法

实验内容简介：
column:AD-H
M.P.:n-hex/i-prOH:EtOH=450：25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-2-race.mdy)



4b (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.477	118859.219	3307132.000	49.8177
2		21.355	89624.461	3331333.750	50.1823
总计			208483.680	6638465.750	100.0000

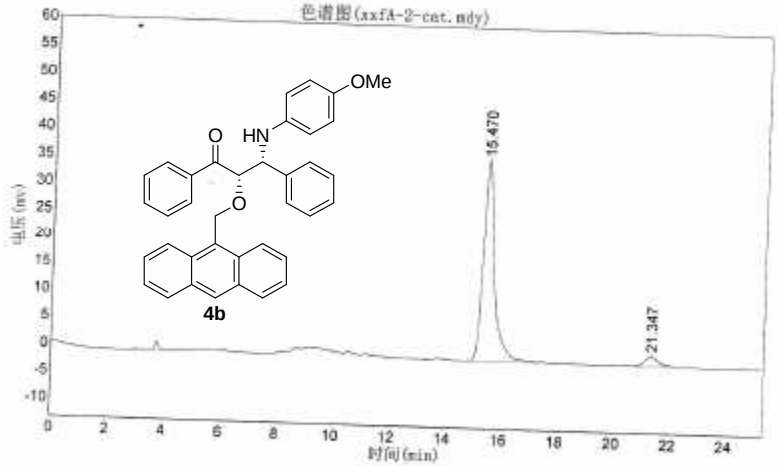
xxfA-2-cat

实验单位：ecnu
实验时间：2009-10-12,20:30:55
谱图文件:C:\湖大智达\N2000\ecnu\xxf\xxfA-2-cat.mdy

实验者：xxf
报告时间：2009-10-12,20:32:48
积分方法:面积归一法

实验内容简介：
column:AD-H
M.P.:n-hex/i-prOH:EtOH=450：25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-2-cat.mdy)



4b

分析结果表

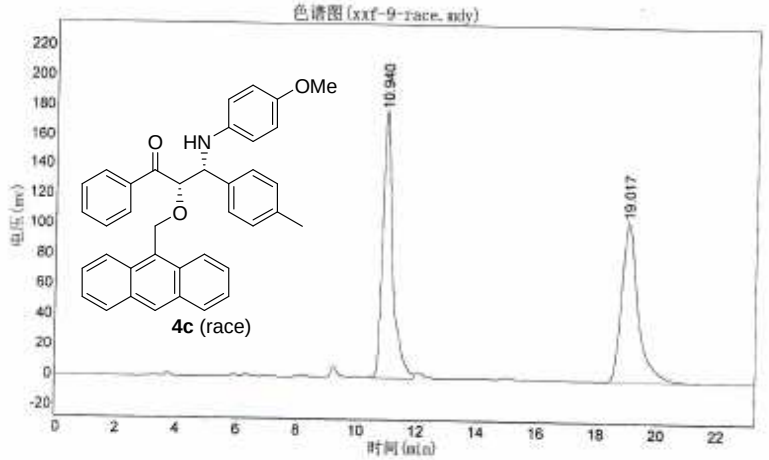
峰号	峰名	保留时间	峰高	峰面积	含量
1		15.470	36902.020	1016982.438	95.2449
2		21.347	1623.470	50772.902	4.7551
总计			38525.489	1067755.340	100.0000

xxfA-9-race

实验单位：ecnu
实验时间：2009-10-15,19:29:11
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-9-race.mdy

实验者：xxf
报告时间：2009-10-15,19:29:13
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex/i-prOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min



分析结果表

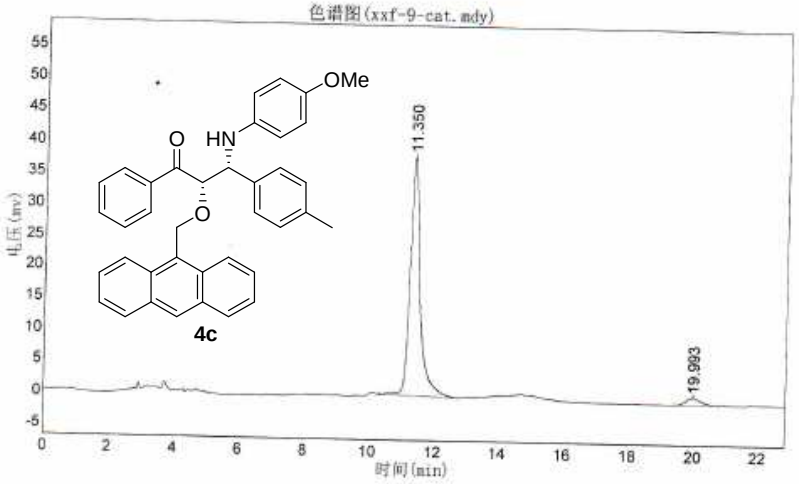
峰号	峰名	保留时间	峰高	峰面积	含量
1		10.940	177097.875	4135777.750	50.4552
2		19.017	105085.758	4061160.000	49.5448
总计			282183.633	8196937.750	100.0000

xxfA-9-cat

实验单位：ecnu
实验时间：2009-10-15,20:14:28
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-9-cat.mdy

实验者：xxf
报告时间：2009-10-15,20:14:33
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex/i-prOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.350	38039.211	827199.125	96.0360
2		19.993	1094.299	34144.000	3.9640
总计			39133.510	861343.125	100.0000

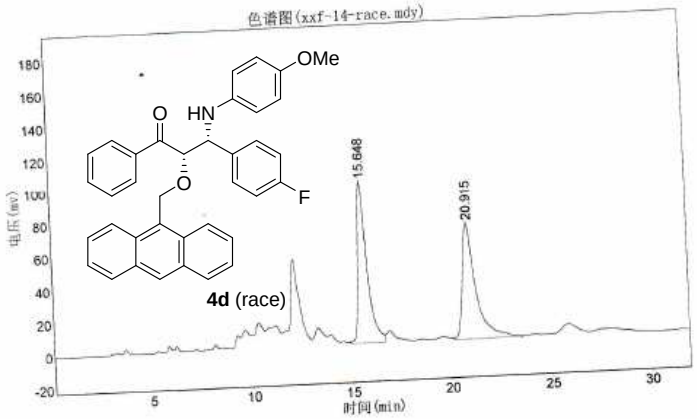
N2000 数据工作站

xxfA-14-race

实验单位：ecnu
实验时间：2009-10-21,21:33:01
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-14-race.mdy

实验者：xxf
报告时间：2009-10-21,21:34:28
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min



分析结果表

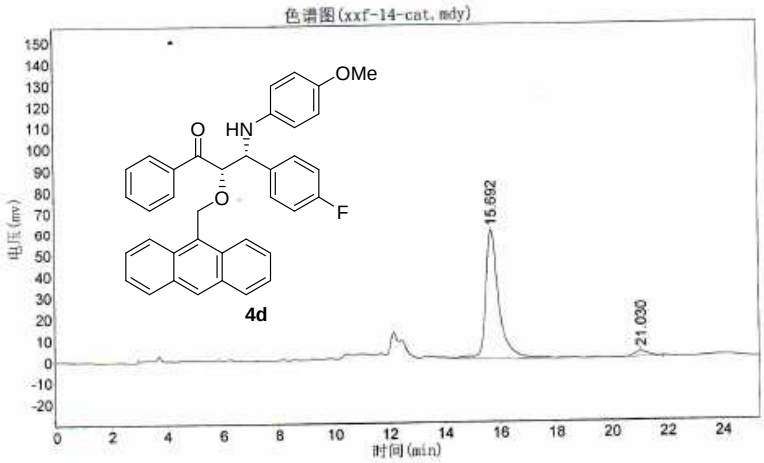
峰号	峰名	保留时间	峰高	峰面积	含量
1		15.648	99388.836	3258567.500	49.4157
2		20.915	69900.914	3335621.250	50.5843
总计			169289.750	6594188.750	100.0000

xxfA-14-cat

实验单位：ecnu
实验时间：2009-10-21,21:36:29
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-14-cat.mdy

实验者：xxf
报告时间：2009-10-21,21:36:34
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min



分析结果表

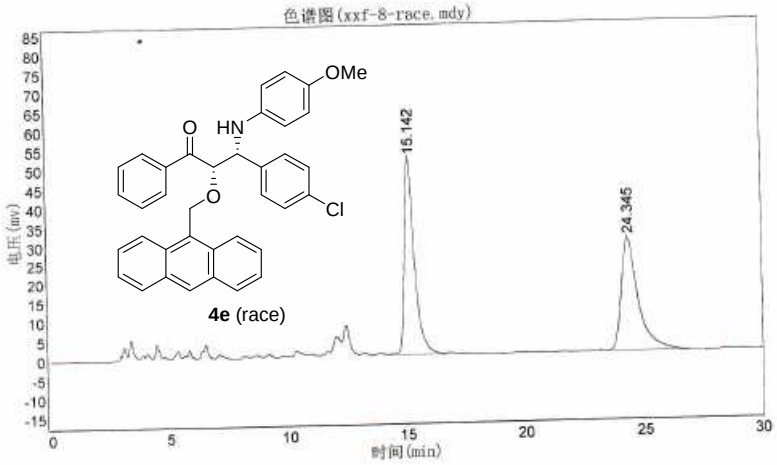
峰号	峰名	保留时间	峰高	峰面积	含量
1		15.692	60346.598	1930923.625	95.9030
2		21.030	2424.204	82488.500	4.0970
总计			62770.802	2013412.125	100.0000

xxfA-8-race

实验单位：ecnu
实验时间：2009-10-21,21:31:52
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-8-race.mdy

实验者：xxf
报告时间：2009-10-21,21:40:25
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
Flow:1.0ml/min



分析结果表

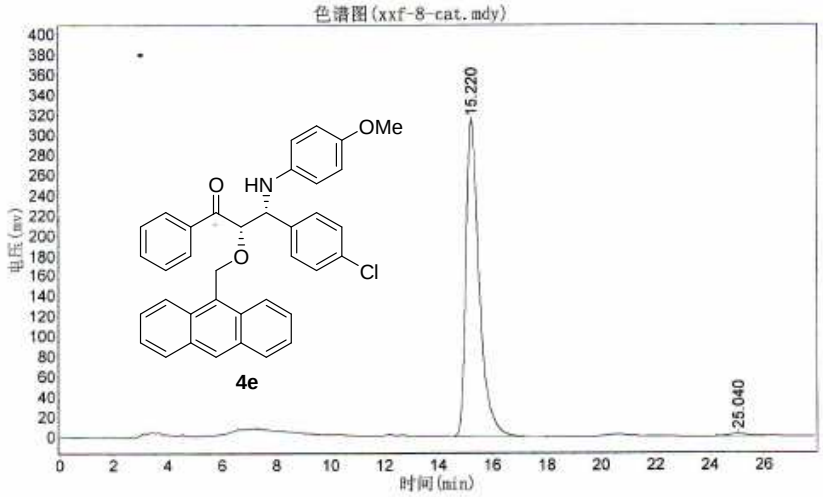
峰号	峰名	保留时间	峰高	峰面积	含量
1		15.142	51238.605	1509629.125	50.8091
2		24.345	29662.867	1461551.125	49.1909
总计			80901.473	2971180.250	100.0000

xxfA-8-cat

实验单位：ecnu
实验时间：2009-10-21,22:14:21
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-8-cat.mdy

实验者：xxf
报告时间：2009-10-21,22:14:25
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.220	312615.281	10765735.000	98.8979
2		25.040	2354.734	119968.547	1.1021
总计			314970.016	10885703.547	100.0000

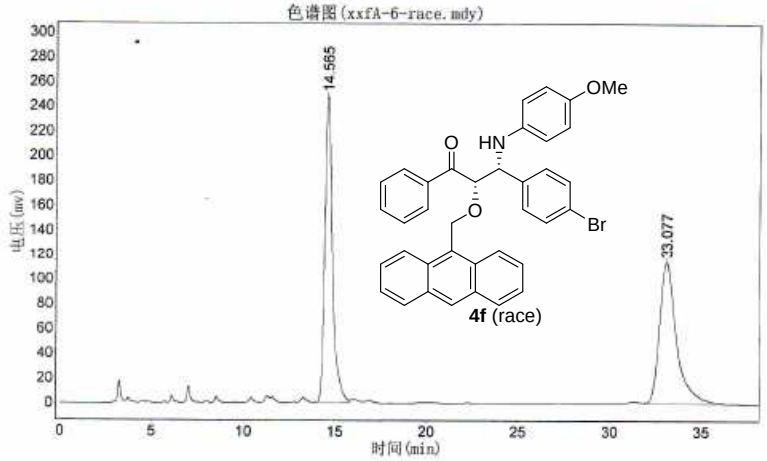
xxfA-6-race

实验单位：ecnu
实验时间：2009-10-14,21:44:44
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-6-race.mdy

实验者：xxf
报告时间：2009-10-14,22:18:04
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex/i-prOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-6-race.mdy)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.565	247906.078	7125321.500	49.3430
2		33.077	114085.984	7315081.000	50.6570
总计			361992.063	14440402.500	100.0000

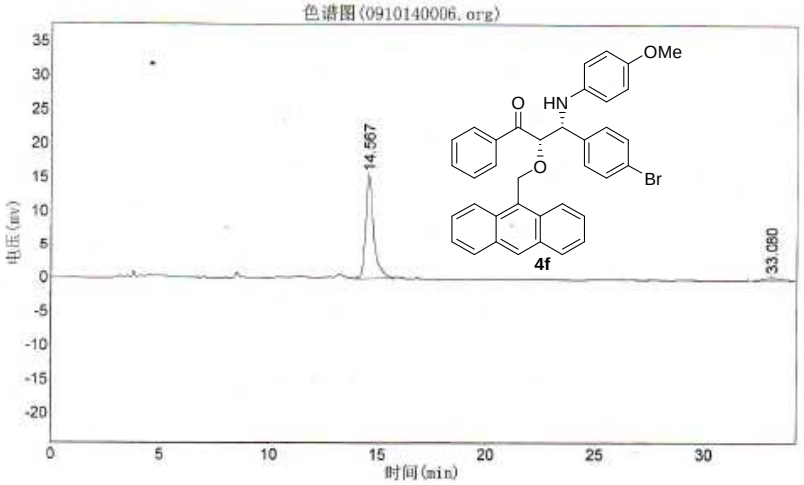
xxfA-6-cat

实验单位：ecnu
实验时间：2009-10-14,22:17:54
谱图文件:C:\浙大智达\N2000\ecnu\xxf\0910140006.org

实验者：xxf
报告时间：2009-10-14,23:04:39
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex/i-prOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(0910140006.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.567	15251.572	396888.250	96.4136
2		33.080	312.681	14763.301	3.5864
总计			15564.253	411651.551	100.0000

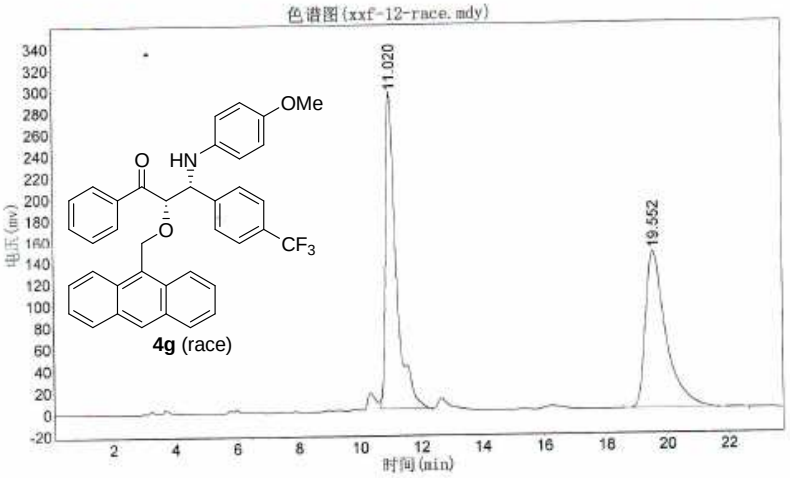
xxf-12-race

实验单位：ecnu
实验时间：2009-10-20,21:55:44
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-12-race.mdy

实验者：xxf
报告时间：2009-10-20,21:56:04
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxf-12-race.mdy)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.020	289926.469	6978408.500	50.9691
2		19.552	143872.297	6713035.000	49.0309
总计			433798.766	13691443.500	100.0000

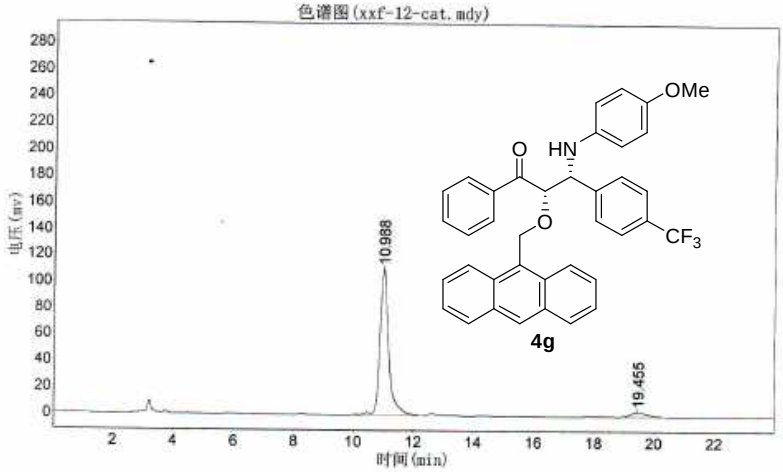
xxfA-12-cat

实验单位：ecnu
实验时间：2009-10-20,21:18:00
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxf-12-cat.mdy

实验者：xxf
报告时间：2009-10-20,21:18:29
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxf-12-cat.mdy)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.988	110609.195	2259188.000	94.9826
2		19.455	3185.675	119339.547	5.0174
总计			113794.870	2378527.547	100.0000

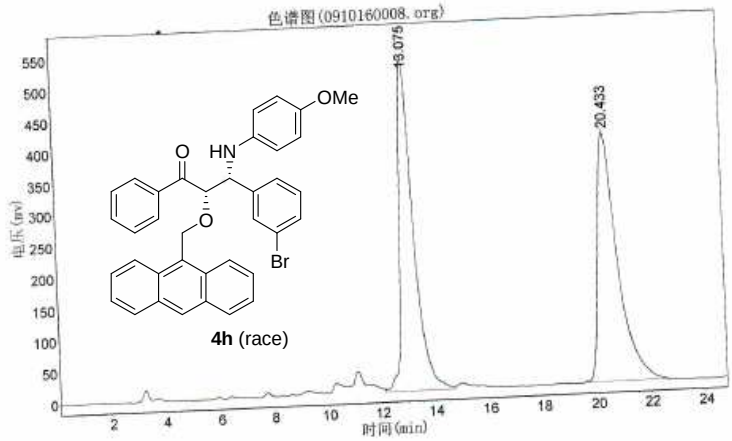
N2000 数据工作站

xxfA-10-race

实验单位：ecnu
实验时间：2009-10-19,18:53:13
谱图文件:C:\浙大智达\N2000\ecnu\xxf\0910160008.org

实验者：xxf
报告时间：2009-10-19,18:57:40
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min



4h (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.075	531837.938	19222812.000	49.0038
2		20.433	396326.000	20004406.000	50.9962
总计			928163.938	39227218.000	100.0000

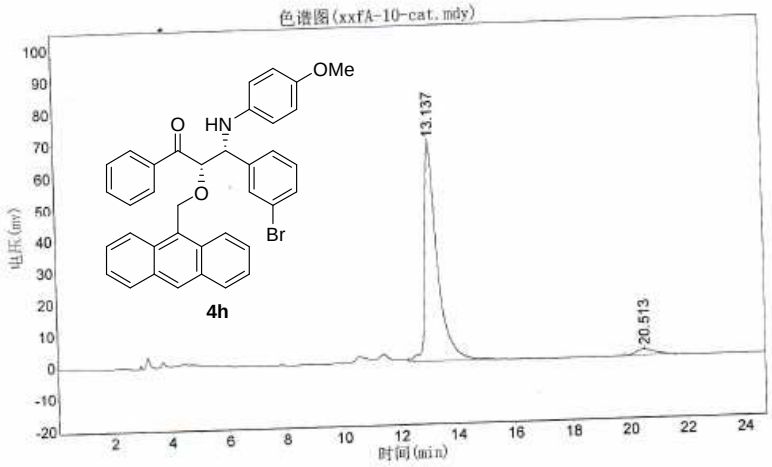
N2000 数据工作站

xxfA-10-cat

实验单位：ecnu
实验时间：2009-10-19,19:44:35
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-10-cat.mdy

实验者：xxf
报告时间：2009-10-19,19:44:40
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:EtOH:iPrOH=450:25:25
Detection:254nm
flow:1.0ml/min



4h

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.137	69002.773	2084069.125	96.0782
2		20.513	1832.017	85069.805	3.9218
总计			70834.790	2169138.930	100.0000

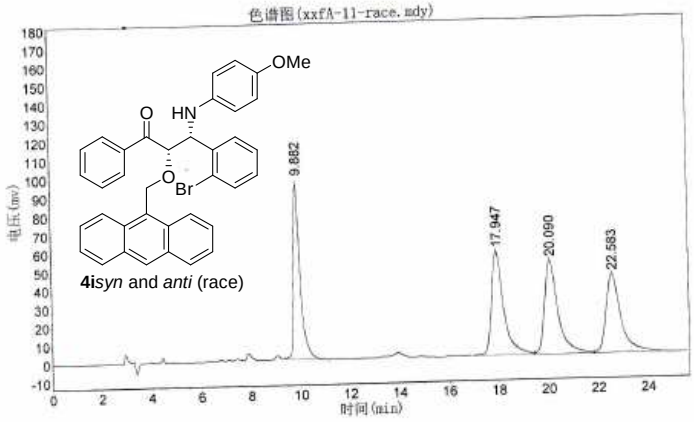
xxfA-11-race

实验单位：ecnu
实验时间：2009-10-22,21:06:30
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-11-race.mdy

实验者：xxf
报告时间：2009-10-23,19:36:48
积分方法:面积归一法

实验内容简介：
column:1A
M.P.:n-hex:iPrOH:TFA=90:10:0.1
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-11-race.mdy)



4i*syn* and *anti* (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.882	94783.313	1744212.625	24.0207
2		17.947	56171.496	1884958.750	25.9590
3		20.090	50645.625	1880226.750	25.8938
4		22.583	42464.711	1751895.125	24.1265
总计			244065.145	7261293.250	100.0000

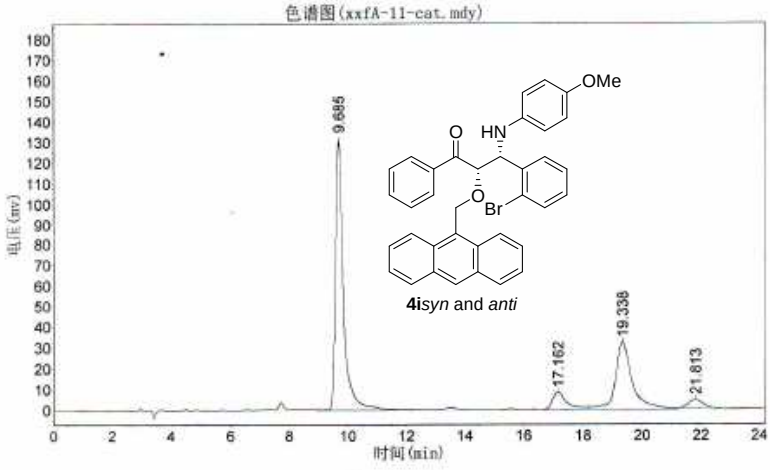
xxfA-11-cat

实验单位：ecnu
实验时间：2009-10-23,19:44:15
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-11-cat.mdy

实验者：xxf
报告时间：2009-10-23,19:44:17
积分方法:面积归一法

实验内容简介：
column:1A
M.P.:n-hex:iPrOH:TFA=90:10:0.1
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-11-cat.mdy)



4i*syn* and *anti*

分析结果表

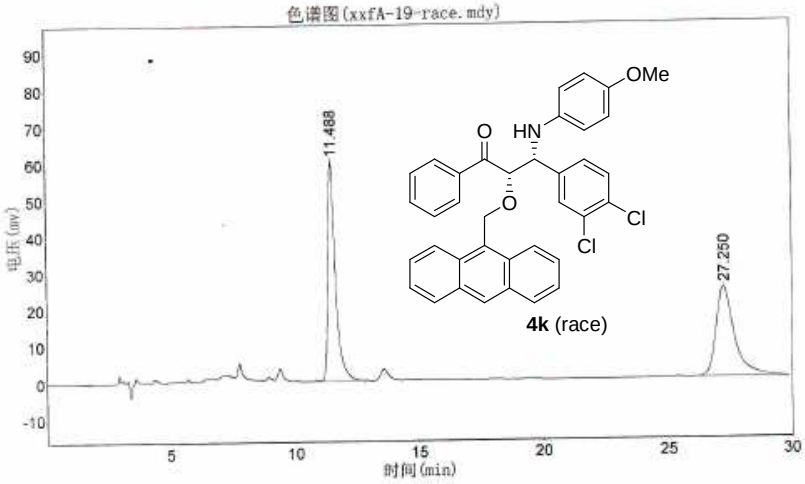
峰号	峰名	保留时间	峰高	峰面积	含量
1		9.685	130138.070	2442453.500	59.4209
2		17.162	8421.843	287229.281	6.9878
3		19.338	32260.693	1253795.250	30.5028
4		21.813	4015.497	126949.805	3.0885
总计			174836.104	4110427.836	100.0000

xxfA-19-race

实验单位：ecnu
实验时间：2009-10-23,21:33:41
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-19-race.mdy

实验者：xxf
报告时间：2009-10-23,21:33:43
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:iPrOH:TFA=90:10:0.1
Detection:254nm
flow:1.0ml/min



4k (race)

分析结果表

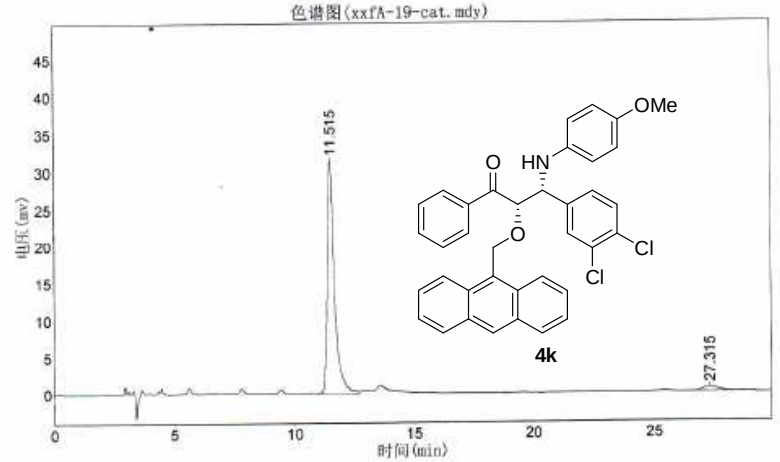
峰号	峰名	保留时间	峰高	峰面积	含量
1		11.488	59867.754	1269871.750	50.4522
2		27.250	24396.568	1247107.500	49.5478
总计			84264.322	2516979.250	100.0000

xxfA-19-cat

实验单位：ecnu
实验时间：2009-10-23,22:04:49
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-19-cat.mdy

实验者：xxf
报告时间：2009-10-23,22:04:51
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:iPrOH:TFA=90:10:0.1
Detection:254nm
flow:1.0ml/min



4k

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.515	31436.916	669981.438	96.3954
2		27.315	589.046	25052.900	3.6046
总计			32025.962	695034.338	100.0000

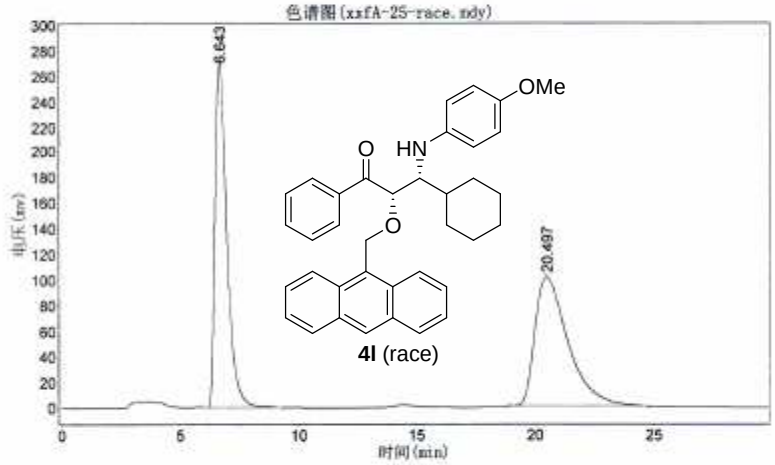
xxfA-24-race

实验单位：ecnu
实验时间：2009-12-02,20:35:57
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-25-race.mdy

实验者：xxf
报告时间：2009-12-02,20:35:59
积分方法:面积归一法

实验内容简介：
column:AD-H
M.P.:n-hex:iPrOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-25-race.mdy)



4l (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		6.643	269927.031	9459776.000	49.0358
2		20.497	100988.125	9831780.000	50.9642
总计			370915.156	19291556.000	100.0000

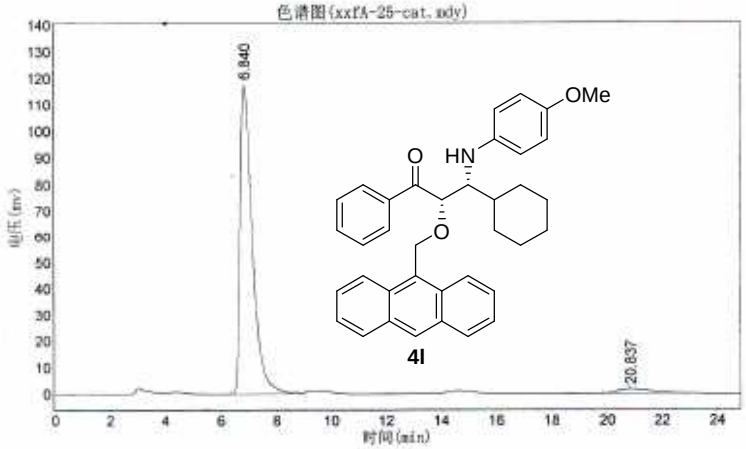
xxfA-24-cat

实验单位：ecnu
实验时间：2009-12-02,20:13:59
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-25-cat.mdy

实验者：xxf
报告时间：2009-12-02,20:28:28
积分方法:面积归一法

实验内容简介：
column:AD-H
M.P.:n-hex:iPrOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-25-cat.mdy)



4l

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		6.840	116156.195	3568235.000	96.6638
2		20.837	1514.125	123152.297	3.3362
总计			117670.321	3691387.297	100.000

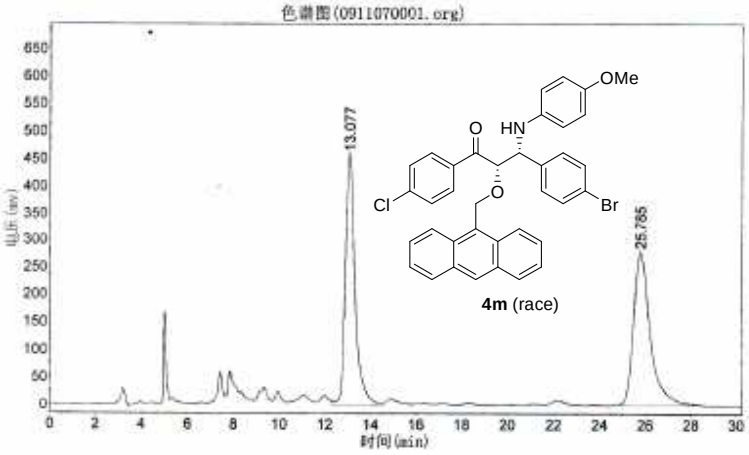
xxfA-20-1-race

实验单位：ecnu
实验时间：2009-11-08,13:02:28
谱图文件:C:\浙大智达\N2000\ecnu\xxf\0911070001.org

实验者：xxf
报告时间：2009-11-08,13:14:25
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:i-PrOH:TFA=90：10：0.1
Detection:254nm
flow:1.0ml/min

色谱图(0911070001.org)



4m (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.077	455736.875	13487067.000	49.1845
2		25.785	274832.844	13934325.000	50.8155
总计			730569.719	27421392.000	100.0000

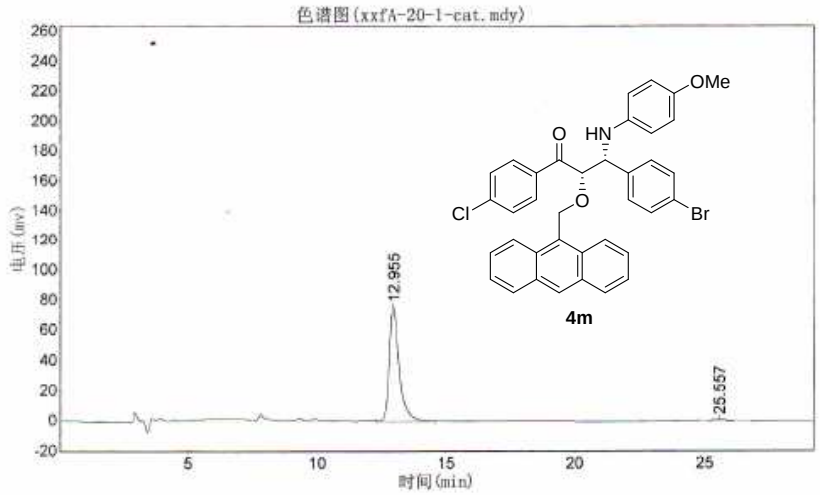
xxfA-20-1-cat

实验单位：ecnu
实验时间：2009-11-08,14:11:33
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-20-1-cat.mdy

实验者：xxf
报告时间：2009-11-08,14:11:36
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:i-PrOH:TFA=90：10：0.1
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-20-1-cat.mdy)



4m

分析结果表

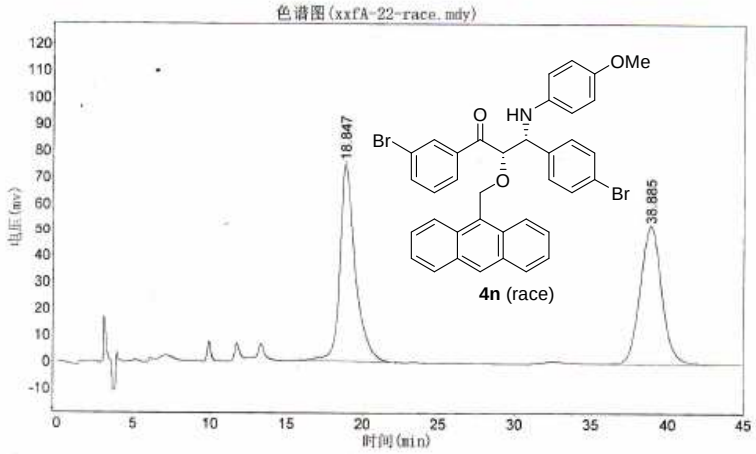
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.955	76136.648	2056869.000	97.0438
2		25.557	1381.758	62657.402	2.9562
总计			77518.407	2119526.402	100.0000

xxfA-22-race

实验单位：ecnu
实验时间：2009-11-08,17:08:33
谱图文件:C:\新大智达\N2000\ecnu\xxf\xxfA-22-race.mdy

实验者：xxf
报告时间：2009-11-08,17:08:35
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:i-PrOH:EtOH:TFA=500：20：20:1
Detection:254nm
flow:1.0ml/min



4n (race)

分析结果表

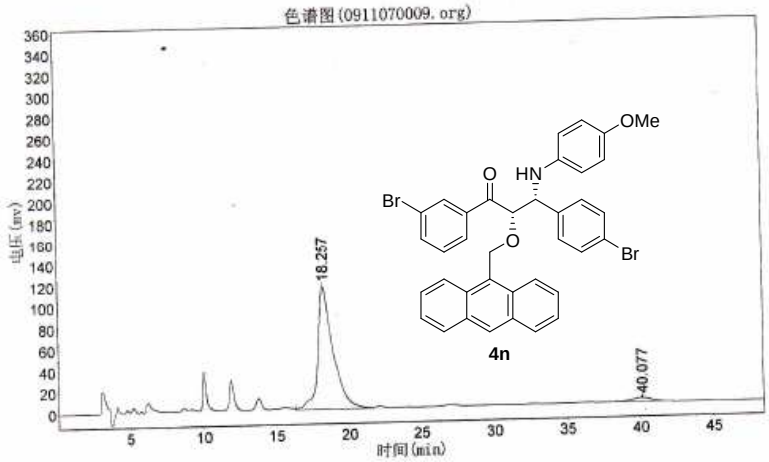
峰号	峰名	保留时间	峰高	峰面积	含量
1		18.847	74562.047	5235161.000	50.6721
2		38.885	51818.406	5096290.000	49.3279
总计			126380.453	10331451.000	100.0000

xxfA-22-cat

实验单位：ecnu
实验时间：2009-11-08,18:38:24
谱图文件:C:\新大智达\N2000\ecnu\xxf\0911070009.org

实验者：xxf
报告时间：2009-11-08,18:41:26
积分方法:面积归一法

实验内容简介：
column:IA
M.P.:n-hex:i-PrOH:EtOH:TFA=500：20：20:1
Detection:254nm
flow:1.0ml/min



4n

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		18.257	115746.578	8385583.000	95.9101
2		40.077	3582.833	357589.688	4.0899
总计			119329.411	8743172.688	100.0000

N2000 数据工作站

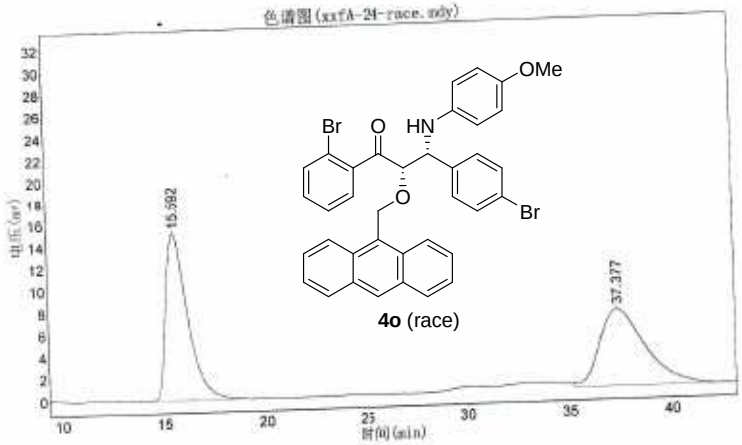
xxfA-24-race

实验单位：ecnu
实验时间：2009-12-01,21:48:50
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-24-race.mdy

实验者：xxf
报告时间：2009-12-01,21:51:08
积分方法:面积归一法

实验内容简介:
column:AD-H
M.P.:n-hex:iPrOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-24-race.mdy)



4o (race)

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.592	15129.708	1120284.000	50.9295
2		37.377	6995.193	1079390.375	49.0705
总计			22124.901	2199674.375	100.0000

N2000 数据工作站

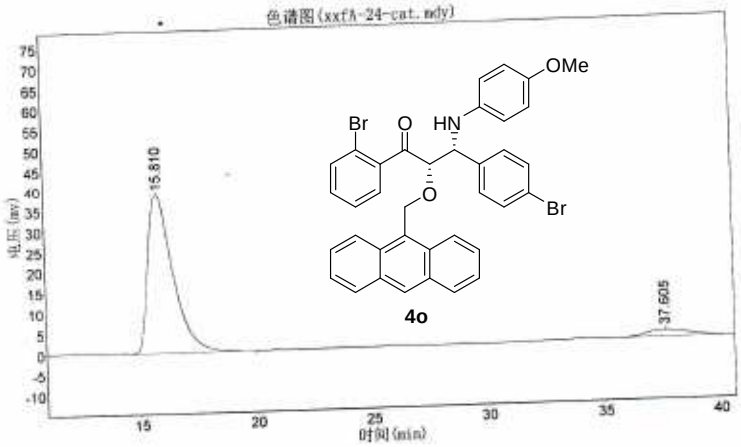
xxfA-24-cat

实验单位：ecnu
实验时间：2009-12-01,22:33:53
谱图文件:C:\浙大智达\N2000\ecnu\xxf\xxfA-24-cat.mdy

实验者：xxf
报告时间：2009-12-01,22:33:57
积分方法:面积归一法

实验内容简介:
column:AD-H
M.P.:n-hex:iPrOH:EtOH=450:25:25
Detection:254nm
flow:1.0ml/min

色谱图(xxfA-24-cat.mdy)



4o

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.810	38832.625	2891198.250	93.5806
2		37.605	1607.032	198328.297	6.4194
总计			40439.657	3089526.547	100.0000