

Supporting Information

Cooperative Catalysis with Chiral Brønsted Acid-Rh₂(OAc)₄: Highly Enantioselective Three-Component Reactions of Diazo compounds with Alcohols and Imines

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General: HRMS (ESI) Mass spectra were recorded on a Bio TOF IIIQ from Bruker Daltonics Inc. at Chengdu Institute of Biology (CIB), the Chinese Academy of Sciences (CAS). NMR spectra were recorded on a Brucker-400 MHz spectrometer. HPLC analysis was performed on Waters-Breeze (2487 Dual Absorbance Detector and 1525 Binary HPLC Pump) & Shimadzu (SPD-20AV UV-VIS Detector and LC-20AT Liquid Chromatograph Pump). Chiraldak OD, AD, 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.

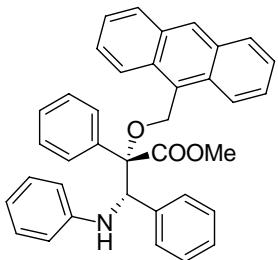
Materials: Dichloromethane was distilled over calcium hydride. Aldimines **3** and **6** were prepared from condensation of the corresponding aldehydes with amines according to the literature method.^[1] Chiral phosphoric acid **5a-e** were prepared according to the literature procedure.^[2] Solvents for the column chromatography were distilled before use. All reactions were carried out under an argon atmosphere in a well-dried glassware.

[†] East China Normal University

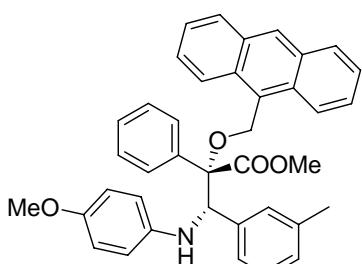
[‡] University of Science and Technology of China

General Procedure for the Enantioselective Three-Component Reactions (Table 2):

A solution of $\text{Rh}_2(\text{OAc})_4$ (0.005 mmol), chiral phosphoric acid **5b** (0.005 mmol), alcohol **2e** (0.25 mmol) , imine **3** or **6** (0.275 mmol) and 4 \AA MS (0.1 g) in 2 mL CH_2Cl_2 under an argon atmosphere was cooled to -20°C . Diazo compound **1** (0.25 mmol) in 1 mL CH_2Cl_2 was then added over 1 h period of time via a syringe pump. 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 ~1:20) to give the pure products.

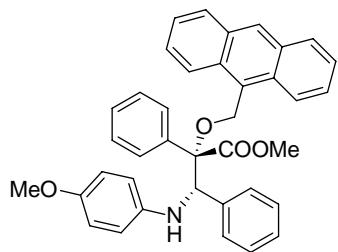


(2S,3S)-methyl 2-(9-anthrylmethoxy)-2,3-diphenyl-3-(phenylamino)propanoate (4e): yield 86%; $[\alpha]_D^{20} = +67.3^\circ$ ($c = 1$, EtOAc); 93% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol = 97 : 3, 254nm, Retention time: $t_{\text{major}} = 7.3$ min, and $t_{\text{minor}} = 9.4$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.70 (s, 3H), 4.87 (d, $J = 9.6$ Hz, 1H), 5.17 (d, $J = 9.6$ Hz, 1H), 5.21 (d, $J = 10.3$ Hz, 1H), 5.76 (d, $J = 10.3$ Hz, 1H), 6.41 (dd, $J_1 = 1.0$ Hz, $J_2 = 8.6$ Hz, 2H), 6.65 (m, 1H), 6.97 (m, 2H), 7.13 (m, 5H), 7.47-7.54 (m, 7H), 7.82 (m, 2H), 8.06 (m, 2H), 8.30 (m, 2H), 8.53 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.93, 60.60, 64.30, 87.90, 114.04, 117.63, 125.07, 125.11, 126.10, 127.59, 127.62, 128.37, 128.50, 128.96, 129.02, 129.15, 129.26, 131.17, 131.65, 136.41, 136.74, 146.25, 171.69; HRMS (EI) calcd for $\text{C}_{37}\text{H}_{31}\text{NO}_3$ (M^+) 537.2304, found 537.2300;



(2S,3S)-methyl 2-(9-anthrylmethoxy)-2-phenyl-3-(4-methoxyphenylamino)-3-m-tolylpropanoate (7a): yield 95%; $[\alpha]_D^{20} = +49.2^\circ$ ($c = 1$, EtOAc); 90% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol = 80 : 20, 254nm, Retention time: $t_{\text{major}} = 4.9$ min, and $t_{\text{minor}} = 5.6$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 2.05 (s, 3H), 3.57 (s, 3H), 3.67 (s, 3H), 4.48 (bs, 1H), 5.02 (bs, 1H), 5.18 (d, $J = 10.3$ Hz, 1H), 5.68 (d, $J = 10.3$ Hz, 1H), 6.33 (d, $J = 9.0$ Hz, 2H), 6.53 (d, $J = 9.0$ Hz, 2H), 6.86-6.93 (m, 4H), 7.41-7.48 (m, 7H), 7.79 (m, 2H), 7.99 (d, $J = 9.5$ Hz, 2H), 8.26

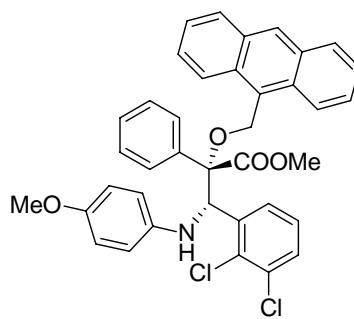
(d, $J = 9.5$ Hz, 2H), 8.45 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 21.35, 51.85, 55.63, 60.44, 65.38, 87.94, 114.63, 115.44, 125.03, 126.06, 126.14, 127.24, 128.16, 128.26, 128.41, 128.84, 128.90, 129.12, 129.41, 129.84, 131.13, 131.60, 136.43, 138.64, 140.55, 152.14, 171.73; HRMS (ESI) calcd for $\text{C}_{39}\text{H}_{36}\text{NO}_4$ ($\text{M}+\text{H}$) $^+$ 582.2639, found 582.2617.



(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)

-2,3-diphenylpropanoate (7b): yield 83%; $[\alpha]_D^{20} = +57.6^\circ$ ($c = 1$, EtOAc); 94% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{major}} = 6.9$ min, and $t_{\text{minor}} = 8.4$ min.); ^1H

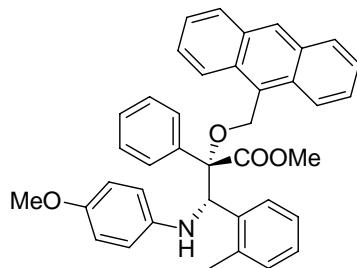
NMR (CDCl_3 , 400 MHz) δ (ppm) 3.57 (s, 3H), 3.67 (s, 3H), 4.52 (bs, 1H), 5.06 (bs, 1H), 5.17 (d, $J = 10.3$ Hz, 1H), 5.68 (d, $J = 10.3$ Hz, 1H), 6.33 (d, $J = 9.0$ Hz, 2H), 6.53 (d, $J = 9.0$ Hz, 2H), 7.04 (m, 5H), 7.41-7.49 (m, 7H), 7.80 (m, 2H), 8.01 (d, $J = 9.5$ Hz, 2H), 8.27 (d, $J = 9.5$ Hz, 2H), 8.46(s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.90, 55.63, 60.55, 65.49, 87.96, 114.63, 115.45, 125.07, 126.04, 127.50, 128.24, 128.44, 128.77, 128.92, 129.09, 129.35, 129.47, 131.12, 131.61, 136.38, 138.76, 10.44, 152.20, 171.70; HRMS (ESI) calcd for $\text{C}_{38}\text{H}_{34}\text{NO}_4$ ($\text{M}+\text{H}$) $^+$ 568.2482, found 568.2489.



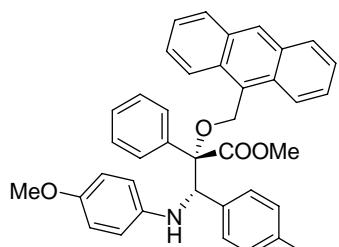
(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-3-(2,3-dichlorophenyl)-2-phenylpropanoate (7c): yield

95%; $[\alpha]_D^{20} = +18.8^\circ$ ($c = 1$, EtOAc); 93% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / EtOH / DEA = 90 : 10 : 0.1, 254nm, Retention time: $t_{\text{minor}} = 5.6$ min, and $t_{\text{major}} = 7.4$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm)

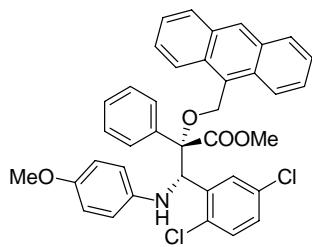
3.66 (s, 3H), 3.71 (s, 3H), 4.71 (d, $J = 10.5$ Hz, 1H), 5.15 (d, $J = 10.5$ Hz, 1H), 5.86 (d, $J = 10.5$ Hz, 1H), 5.89 (d, $J = 10.5$ Hz, 1H), 6.44 (d, $J = 8.5$ Hz, 2H), 6.63 (d, $J = 8.5$ Hz, 2H), 6.93 (m, 1H), 7.29(m, 2H), 7.53-7.58 (m, 7H), 7.97 (m, 2H), 8.09 (m, 2H), 8.35 (m, 2H), 8.55 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 29.62, 51.66, 55.48, 60.05, 60.40, 87.28, 114.62, 115.12, 124.79, 124.98, 126.03, 126.99, 128.32, 128.38, 128.42, 128.62, 128.80, 129.04, 129.12, 129.53, 131.10, 131.47, 132.28, 132.68, 136.51, 139.31, 140.04, 152.29, 171.43; HRMS (ESI) calcd for $\text{C}_{38}\text{H}_{31}\text{Cl}_2\text{KNO}_4$ ($\text{M}+\text{K}$) $^+$ 674.1262, found 674.1228.



(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-2-phenyl-3-o-tolylpropanoate (7d): yield 95%; $[\alpha]_D^{20} = +33.2^\circ$ ($c = 1$, EtOAc); 93% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol = 95 : 5, 254nm, Retention time: $t_{\text{major}} = 5.8$ min, and $t_{\text{minor}} = 7.7$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 2.10 (s, 3H), 3.60 (s, 3H), 3.72 (s, 3H), 4.26 (bs, 1H), 5.20 (d, $J = 10.3$ Hz, 1H), 5.49 (bs, 1H), 5.53 (d, $J = 10.3$ Hz, 1H), 6.36 (d, $J = 8.9$ Hz, 2H), 6.55 (d, $J = 8.9$ Hz, 2H), 6.80-6.85 (m, 4H), 7.43-7.50 (m, 7H), 7.78 (m, 2H), 8.00 (d, $J = 9.0$ Hz, 2H), 8.30 (d, $J = 9.0$ Hz, 2H) 8.46 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 19.95, 51.98, 55.65, 60.61, 60.94, 88.47, 114.64, 115.95, 125.06, 125.09, 125.33, 126.04, 127.11, 128.10, 128.52, 128.83, 128.93, 129.62, 129.82, 131.10, 131.58, 136.24, 137.21, 137.46, 140.63, 152.50, 171.85; HRMS (ESI) calcd for $\text{C}_{39}\text{H}_{35}\text{KNO}_4$ ($\text{M}+\text{K}$) $^+$ 620.2198, found 620.2175.

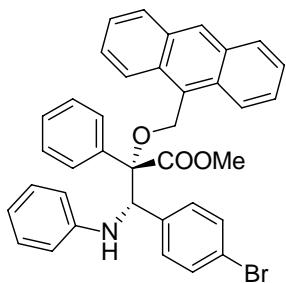


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-2-phenyl-3-p-tolylpropanoate (7e): yield 92%; $[\alpha]_D^{20} = +84.7^\circ$ ($c = 1$, EtOAc); 98% ee, determined by HPLC (Daicel Chirapak IA, flow rate 0.9 mL/min, hexane / isopropanol / EtOH / TFA = 425 : 25 : 25 : 1, Retention time: $t_{\text{major}} = 7.0$ min, and $t_{\text{minor}} = 8.2$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 2.19 (s, 3H), 3.57 (s, 3H), 3.68 (s, 3H), 4.52 (bs, 1H), 5.03 (bs, 1H) 5.16 (d, $J = 10.4$ Hz, 1H), 5.69 (d, $J = 10.4$ Hz, 1H), 6.32 (d, $J = 8.9$ Hz, 2H), 6.53 (d, $J = 8.9$ Hz, 2H), 6.85 (d, $J = 8.0$ Hz, 2H), 6.94 (d, $J = 8.0$ Hz, 2H), 7.41-7.50 (m, 7H), 7.82 (m, 2H), 8.00 (d, $J = 9.2$ Hz, 2H), 8.27 (d, $J = 9.2$ Hz, 2H), 8.46 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 21.17, 51.88, 55.64, 60.53, 65.14, 88.01, 114.63, 115.46, 125.06, 125.11, 126.02, 128.23, 128.27, 128.41, 128.86, 128.91, 128.94, 129.14, 129.39, 131.14, 131.61, 135.71, 136.54, 136.98, 140.54, 152.14, 171.78; HRMS (ESI) calcd for $\text{C}_{39}\text{H}_{35}\text{KNO}_4$ ($\text{M}+\text{K}$) $^+$ 620.2198, found 620.2215.

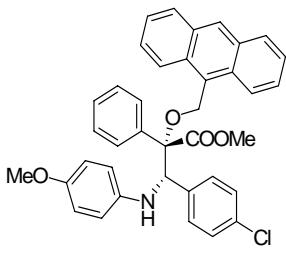


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-3-(2,5-dichlorophenyl)-2-phenylpropanoate (7f): yield 95%; $[\alpha]_D^{20} = +95.5^\circ$ ($c = 1$, EtOAc); 93% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol = 95 : 5,

254nm, Retention time: $t_{\text{major}} = 7.0$ min, and $t_{\text{minor}} = 9.0$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 2.18 (s, 3H), 3.79 (s, 3H), 4.72 (d, $J = 10.0$ Hz, 1H), 5.20 (d, $J = 10.0$ Hz, 1H), 5.26 (d, $J = 10.5$ Hz, 1H), 5.78 (d, $J = 10.5$ Hz, 1H), 6.37 (d, $J = 8.5$ Hz, 2H), 6.86 (d, $J = 8.5$ Hz, 2H), 7.03 (d, $J = 8.5$ Hz, 2H), 7.26 (d, $J = 8.5$ Hz, 2H), 7.52-7.61 (m, 7H), 7.87 (m, 2H), 8.11 (d, $J = 3.5$ Hz, 2H), 8.34 (d, $J = 3.5$ Hz, 2H), 8.56 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 20.23, 51.94, 60.50, 63.88, 87.50, 114.15, 121.50, 124.78, 124.99, 126.02, 127.08, 128.29, 128.45, 128.69, 128.88, 128.99, 129.12, 129.49, 130.56, 130.68, 130.98, 131.49, 135.85, 137.77, 143.42, 171.41; HRMS (ESI) calcd for $\text{C}_{38}\text{H}_{31}\text{Cl}_2\text{KNO}_4$ ($\text{M}+\text{K}$) $^+$ 674.1262, found 674.1228.

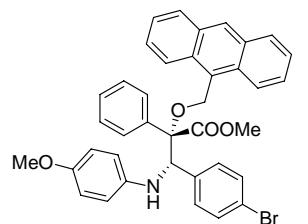


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-bromophenyl)-2-phenyl-3-(phenylamino)propanoate (7g): yield 87%; $[\alpha]_D^{20} = +86.6^\circ$ ($c = 1$, EtOAc); 92% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol = 95 : 5, 254nm, Retention time: $t_{\text{major}} = 7.2$ min, and $t_{\text{minor}} = 9.6$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.728 (s, 3H), 4.77 (d, $J = 9.5$ Hz, 1H), 5.13 (d, $J = 9.5$ Hz, 1H), 5.20 (d, $J = 10.5$ Hz, 1H), 5.73 (d, $J = 10.5$ Hz, 1H), 6.37 (m, 2H), 6.58 (m, 1H), 6.98 (m, 4H) 7.19 (m, 2H), 7.46-7.54 (m, 7H), 7.79 (m, 2H), 8.06 (d, $J = 9.0$ Hz, 2H), 8.26 (d, $J = 9.0$ Hz, 2H), 8.52 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.98, 60.56, 63.67, 87.53, 114.01, 117.91, 121.59, 124.79, 125.04, 126.07, 128.36, 128.50, 128.69, 128.93, 128.99, 129.06, 129.09, 130.63, 130.72, 131.02, 131.55, 135.84, 137.71, 145.79, 171.38; HRMS (ESI) calcd for $\text{C}_{37}\text{H}_{30}\text{BrKNO}_3$ ($\text{M}+\text{K}$) $^+$ 654.1041, found 654.1031.

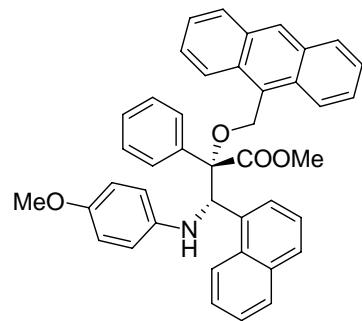


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-chlorophenyl)-2-phenyl-3-(4-methoxyphenylamino)propanoate (7h): yield 83%; $[\alpha]_D^{20} = +86.1^\circ$ ($c = 1$, EtOAc); 93% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol = 90 : 10, 254nm, Retention time: $t_{\text{major}} = 6.3$ min, and $t_{\text{minor}} = 8.3$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.70 (s, 3H), 3.84 (s, 3H), 4.59 (bs, 1H), 5.19 (bs, 1H), 5.32 (d, $J = 11.0$ Hz, 1H), 5.81 (d, $J = 11.0$ Hz, 1H), 6.45 (d, $J = 9.0$ Hz, 2H), 6.68 (d, $J = 9.0$ Hz, 2H), 7.15 (m, 4H), 7.57-7.65 (m, 7H), 7.93 (m, 2H), 8.14 (d, $J = 8.0$ Hz, 2H), 8.41 (d, $J = 9.0$ Hz, 2H), 8.58 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.90, 53.35, 55.45, 60.45, 64.76, 87.60, 114.52, 115.47,

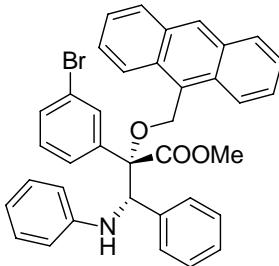
124.77, 124.96, 125.98, 127.57, 128.21, 128.41, 128.69, 128.86, 128.93, 129.16, 130.29, 130.94, 131.46, 133.14, 135.82, 137.21, 139.90, 152.29, 171.37; HRMS (ESI) calcd for $C_{38}H_{32}ClNNaO_4$ ($M+Na$)⁺ 624.1912, found 624.1906.



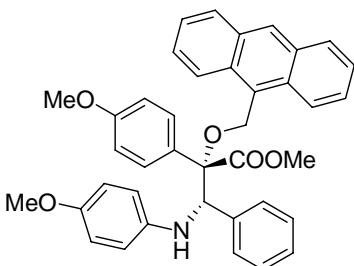
(2S,3S)-methyl 2-(9-anthryl)methoxy-3-(4-bromophenyl)-2-phenyl-3-(4-methoxyphenylamino)propanoate (7i): yield 82%; $[\alpha]_D^{20} = +92.8^\circ$ ($c = 1$, EtOAc); 94% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol = 90 : 10, 254nm, Retention time: $t_{\text{major}} = 6.6$ min, and $t_{\text{minor}} = 8.7$ min.); 1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 3.69 (s, 3H), 3.81 (s, 3H), 4.53 (bs, 1H), 5.13 (bs, 1H), 5.27 (d, $J = 10.5$ Hz, 1H), 5.76 (d, $J = 10.5$ Hz, 1H), 6.40 (d, $J = 9.0$ Hz, 2H), 6.64 (d, $J = 9.0$ Hz, 2H), 7.00 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.52-7.62 (m, 7H), 7.87 (m, 2H), 8.12 (d, $J = 8.5$ Hz, 2H), 8.35 (d, $J = 8.5$ Hz, 2H), 8.57 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm) 51.96, 55.52, 60.50, 64.85, 87.57, 114.55, 115.49, 121.47, 124.80, 125.00, 126.02, 128.24, 128.45, 128.71, 128.89, 128.97, 129.18, 130.54, 130.57, 130.97, 131.49, 135.80, 137.74, 139.91, 152.33, 171.39; HRMS (ESI) calcd for $C_{38}H_{33}BrNO_4$ ($M+H$)⁺ 646.1587, found 646.1589.



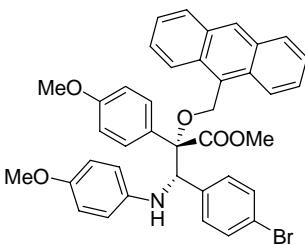
(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-3-(1-naphthalenyl)-2-phenylpropanoate (7j): yield 88%; $[\alpha]_D^{20} = +80.3^\circ$ ($c = 1$, EtOAc); 95% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol = 90 : 10, 254nm, Retention time: $t_{\text{minor}} = 8.6$ min, and $t_{\text{major}} = 12.0$ min.); 1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 3.59 (s, 3H), 3.69 (s, 3H), 4.70 (bs, 1H), 5.27 (bs, 1H), 5.30 (d, $J = 10.5$ Hz, 1H), 5.80 (d, $J = 10.5$ Hz, 1H), 6.43 (d, $J = 9.0$ Hz, 2H), 6.57 (d, $J = 9.0$ Hz, 2H), 7.27 (m, 1H), 7.41 (m, 2H), 7.49-7.59 (m, 10H), 7.65 (m, 1H), 7.91 (m, 2H), 8.09 (m, 2H), 8.36 (m, 2H), 8.54 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm) 51.94, 53.44, 55.49, 60.45, 65.50, 87.99, 114.51, 115.46, 124.96, 125.02, 125.56, 125.68, 126.09, 127.01, 127.44, 128.00, 128.25, 128.29, 128.44, 128.89, 128.93, 129.32, 131.07, 131.53, 132.74, 136.40, 140.25, 152.10, 171.65; HRMS (ESI) calcd for $C_{42}H_{36}NO_4$ ($M+H$)⁺ 618.2639, found 618.2635.



(2S,3S)-methyl 2-(9-anthrylmethoxy)-2-(3-bromophenyl)-3-phenyl-3-(phenylamino)propanoate (7k): yield 98%; $[\alpha]_D^{20} = +124.0^\circ$ ($c = 1$, EtOAc); 84% ee, determined by HPLC (Daicel Chirapak OD-H, flow rate 0.6 mL/min, hexane / isopropanol / EtOH / TFA = 500 : 5 : 5 : 1, 254nm, Retention time: $t_{\text{major}} = 15.0$ min, and $t_{\text{minor}} = 16.2$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.73 (s, 3H), 4.66 (d, $J = 9.5$ Hz, 1H), 5.03 (d, $J = 9.5$ Hz, 1H), 5.21 (d, $J = 10.0$ Hz, 1H), 5.70 (d, $J = 10.5$ Hz, 1H), 6.36 (m, 2H), 6.54 (m, 1H), 6.94 (m, 2H), 7.06-7.28 (m, 6H), 7.49-7.56 (m, 5H), 7.75 (m, 1H), 7.90 (s, 1H), 8.05 (d, $J = 8.0$ Hz, 2H), 8.27 (d, $J = 8.0$ Hz, 2H), 8.52 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 52.17, 60.75, 64.96, 87.48, 114.11, 117.86, 122.45, 124.81, 125.08, 126.31, 127.61, 127.73, 127.76, 128.45, 128.65, 128.94, 129.70, 131.00, 131.50, 131.94, 132.25, 138.02, 138.45, 146.13, 170.93; HRMS (ESI) calcd for $\text{C}_{37}\text{H}_{30}\text{BrKNO}_3$ ($\text{M}+\text{K}$) $^+$ 654.1041, found 654.1041.

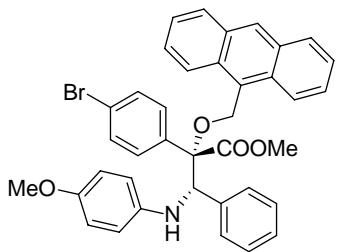


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-2-(4-methoxyphenyl)-3-phenylpropanoate (7l): yield 98%; $[\alpha]_D^{20} = +36.4^\circ$ ($c = 1$, EtOAc); >99% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 490 : 10 : 10 : 1, 254nm, Retention time: $t_{\text{major}} = 33.0$ min, and $t_{\text{minor}} = 41.7$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.62 (s, 3H), 3.70 (s, 3H), 3.90 (s, 3H), 4.60 (bs, 1H), 5.07 (bs, 1H), 5.20 (d, $J = 10.5$ Hz, 1H), 5.70 (d, $J = 10.5$ Hz, 1H), 6.37 (d, $J = 9.0$ Hz, 2H), 6.58 (d, $J = 8.5$ Hz, 2H), 6.98 (d, $J = 8.5$ Hz, 2H), 7.07-7.15 (m, 5H), 7.50-7.55 (m, 4H), 7.78 (d, $J = 8.5$ Hz, 2H), 8.06 (d, $J = 8$ Hz, 2H), 8.33 (d, $J = 8.5$ Hz, 2H), 8.51 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.78, 55.31, 55.58, 60.27, 65.43, 87.54, 113.47, 114.58, 115.38, 124.99, 125.04, 125.95, 127.42, 128.30, 128.43, 128.84, 129.05, 129.15, 130.67, 131.07, 131.54, 138.80, 140.40, 152.09, 159.82, 171.83; HRMS (ESI) calcd for $\text{C}_{39}\text{H}_{35}\text{KNO}_5$ ($\text{M}+\text{K}$) $^+$ 636.2147, found 636.2151.



(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-3-(4-bromophenyl)-2-(4-methoxyphenyl)propanoate (7m): yield 97%; $[\alpha]_D^{20} = +53.5^\circ$ ($c = 1$, EtOAc); 95% ee, determined by HPLC

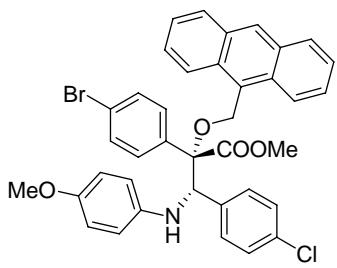
(Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{major}} = 11.8$ min, and $t_{\text{minor}} = 18.6$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.63 (s, 3H), 3.73 (s, 3H), 3.90 (s, 3H), 4.50 (bs, 1H), 5.03 (bs, 1H), 5.20 (d, $J = 10.5$ Hz, 1H), 5.68 (d, $J = 10.5$ Hz, 1H), 6.34 (d, $J = 8.5$ Hz, 2H), 6.59 (d, $J = 8.5$ Hz, 2H), 6.95 (d, $J = 8.5$ Hz, 2H), 6.80 (d, $J = 9.0$ Hz, 2H), 7.20 (d, $J = 8.5$ Hz, 2H), 7.50-7.56 (m, 4H), 7.74 (d, $J = 8.5$ Hz, 2H), 8.06 (d, $J = 8$ Hz, 2H), 8.30 (d, $J = 8.5$ Hz, 2H), 8.51 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 51.93, 55.33, 55.57, 60.31, 64.91, 87.25, 113.56, 114.61, 115.49, 121.46, 124.86, 125.02, 126.03, 127.90, 128.41, 128.90, 130.56, 130.61, 130.73, 131.02, 131.54, 137.89, 139.98, 152.32, 159.93, 171.63; HRMS (ESI) calcd for $\text{C}_{39}\text{H}_{34}\text{BrKNO}_5$ ($\text{M}+\text{K}$) $^+$ 714.1252, found 714.1243.



(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)

-2-(4-bromophenyl)-3-phenylpropanoate (7n): yield 84%; $[\alpha]_D^{20} = +14.0^\circ$ ($c = 1$, EtOAc); 94% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 490 : 10 : 10 : 1, 254nm, Retention time: $t_{\text{major}} = 17.0$ min,

and $t_{\text{minor}} = 31.2$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.60 (s, 3H), 3.70 (s, 3H), 4.36 (d, $J = 9.0$ Hz, 1H), 5.00 (d, $J = 9.0$ Hz, 1H), 5.19 (d, $J = 10.5$ Hz, 1H), 5.66 (d, $J = 10.5$ Hz, 1H), 6.34 (d, $J = 9.0$ Hz, 2H), 6.55 (d, $J = 9.0$ Hz, 2H), 7.01-7.11 (m, 5H), 7.48-7.53 (m, 6H), 7.66 (d, $J = 8.5$ Hz, 2H), 8.05 (d, $J = 8$ Hz, 2H), 8.25 (d, $J = 8.5$ Hz, 2H), 8.50 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 29.68, 52.03, 55.57, 60.67, 65.67, 87.57, 114.57, 115.64, 123.15, 124.82, 125.03, 126.06, 127.52, 127.59, 128.52, 128.61, 128.93, 130.94, 131.07, 131.18, 131.52, 135.31, 138.18, 140.17, 171.22; HRMS (ESI) calcd for $\text{C}_{38}\text{H}_{32}\text{BrKNO}_4$ ($\text{M}+\text{K}$) $^+$ 684.1146, found 684.1122.

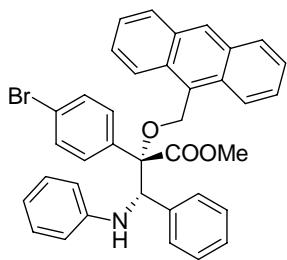


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)

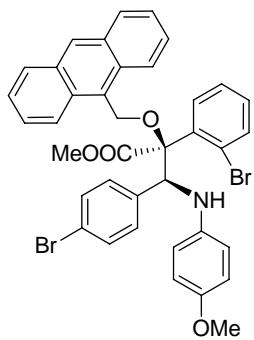
-2-(4-bromophenyl)-3-(4-chlorophenyl)propanoate (7o): yield

95%; $[\alpha]_D^{20} = +45.2^\circ$ ($c = 1$, EtOAc); 92% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 490 : 10 : 10 : 1, 254nm, Retention time: $t_{\text{major}} = 14.0$ min, and $t_{\text{minor}} = 22.6$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.64 (s, 3H), 3.78 (s, 3H), 4.35 (d, $J = 9.5$ Hz, 1H), 5.06 (d, $J = 9.5$ Hz, 1H), 5.23 (d, $J = 10.5$ Hz, 1H), 5.70 (d, $J = 10.5$ Hz, 1H),

6.37 (d, $J = 9.0$ Hz, 2H), 6.60 (d, $J = 8.5$ Hz, 2H), 7.00 (d, $J = 8.5$ Hz, 2H), 7.08 (d, $J = 8.5$ Hz, 2H), 7.52-7.61 (m, 6H), 7.71 (d, $J = 8.5$ Hz, 2H), 8.07 (d, $J = 8.0$ Hz, 2H), 8.29 (d, $J = 9.0$ Hz, 2H), 8.52 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 52.10, 55.46, 60.64, 64.99, 87.28, 114.57, 115.71, 123.29, 124.61, 125.01, 126.09, 127.68, 128.33, 128.58, 128.94, 130.22, 130.85, 130.96, 131.26, 131.46, 133.33, 134.86, 136.75, 139.70, 152.53, 170.98; HRMS (ESI) calcd for $\text{C}_{38}\text{H}_{31}\text{BrClKNO}_4$ ($\text{M}+\text{K}$) $^+$ 718.0757, found 718.0751

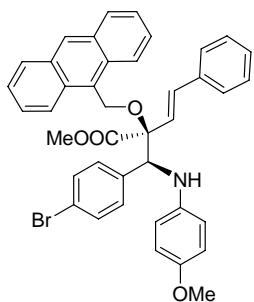


(2S,3S)-methyl 2-(9-anthrylmethoxy)-2-(4-bromophenyl)-3-phenyl-3-(phenylamino)propanoate (7p): yield 84%; $[\alpha]_D^{20} = +21.7^\circ$ ($c = 1$, EtOAc); 92% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{major}} = 7.5$ min, and $t_{\text{minor}} = 10.1$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.66 (s, 3H), 4.69 (d, $J = 10.0$ Hz, 1H), 5.07 (d, $J = 10.0$ Hz, 1H), 5.17 (d, $J = 10.5$ Hz, 1H), 5.68 (d, $J = 10.5$ Hz, 1H), 6.37 (d, $J = 8.0$ Hz, 2H), 6.55 (m, 1H), 6.96 (m, 2H), 7.07-7.13 (m, 5H), 7.47-7.53 (m, 6H), 7.66 (d, $J = 8.5$ Hz, 2H), 8.02 (d, $J = 8.5$ Hz, 2H), 8.23 (d, $J = 8.5$ Hz, 2H), 8.48 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 52.01, 60.66, 64.44, 87.45, 114.07, 117.87, 123.21, 124.76, 125.02, 126.08, 127.56, 127.66, 128.54, 128.93, 128.95, 130.93, 131.27, 131.49, 135.31, 138.12, 145.99, 171.12; HRMS (ESI) calcd for $\text{C}_{37}\text{H}_{30}\text{BrKNO}_3$ ($\text{M}+\text{K}$) $^+$ 654.1041, found 654.1070.

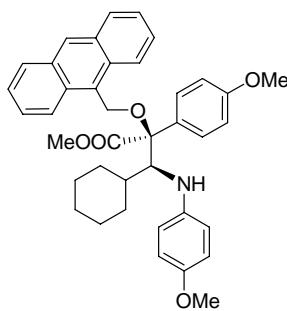


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-2-(2-bromophenyl)-3-(4-bromophenyl)propanoate (7q): yield 91%; 83% ee, determined by HPLC (Daicel Chirapak IA, flow rate 0.7 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{minor}} = 10.8$ min, and $t_{\text{major}} = 13.8$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.61 (s, 3H), 3.71 (s, 3H), 4.60 (s, 1H), 5.19 (s, 1H), 5.47 (d, $J = 10.5$ Hz, 1H), 5.76 (d, $J = 10.5$ Hz, 1H), 6.20 (d, $J = 8.5$ Hz, 2H), 6.52 (d, $J = 9.0$ Hz, 2H), 7.04 (d, $J = 8.0$ Hz, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 7.26 (m, 1H), 7.29 (m, 1H), 7.48-7.53 (m, 4H), 7.69 (d, $J = 8.0$ Hz, 1H), 8.06 (d, $J = 8.0$ Hz, 2H), 8.11 (d, $J = 8.0$ Hz, 1H), 8.19 (d, $J = 8.0$ Hz, 2H), 8.54 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 52.31, 55.60, 61.38, 64.10, 86.91, 114.66, 115.01, 121.37, 123.94, 124.73, 125.09, 126.25, 127.75, 128.15, 128.77, 128.97, 130.17, 130.49,

131.02, 131.37, 131.57, 131.65, 134.70, 137.48, 138.32, 139.85, 152.20, 170.10.

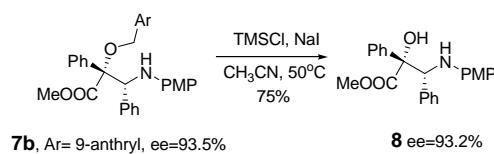


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-2-(*trans*-styryl)-3-(4-bromophenyl)propanoate (7r): yield 43%; $[\alpha]_D^{20} = +62.0^\circ$ ($c = 1$, EtOAc); 95% ee, determined by HPLC (Daicel Chirapak IA, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{major}} = 10.8$ min, and $t_{\text{minor}} = 12.7$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 1.30 (t, $J = 7.5$ Hz, 3H), 3.61 (s, 3H), 4.21-4.28 (m, 2H), 4.51 (d, $J = 8.5$ Hz, 1H), 4.68 (d, $J = 8.0$ Hz, 1H), 5.64 (dd, $J_1 = 10.5$ Hz, $J_2 = 22.0$ Hz, 2H), 6.29 (d, $J = 8.5$ Hz, 2H), 6.54 (d, $J = 9.0$ Hz, 2H), 6.83 (m, 2H), 7.14 (d, $J = 8.5$ Hz, 2H), 7.28 (d, $J = 8.5$ Hz, 2H), 7.38-7.46 (m, 5H), 7.50-7.52 (m, 4H), 8.06 (m, 2H), 8.44 (m, 2H), 8.52 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 14.15, 55.60, 59.94, 61.72, 65.96, 86.15, 114.57, 115.20, 121.63, 124.80, 125.06, 126.16, 126.94, 128.48, 128.76, 128.96, 128.99, 130.71, 130.82, 130.97, 131.54, 135.29, 135.89, 137.63, 140.69, 152.18, 170.84.

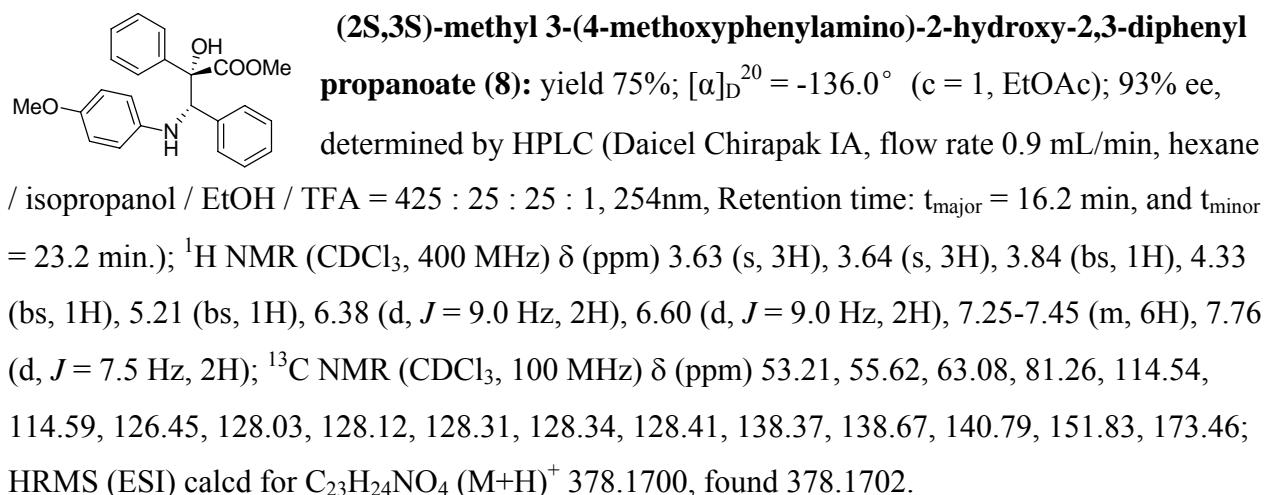


(2S,3S)-methyl 2-(9-anthrylmethoxy)-3-(4-methoxyphenylamino)-3-cyclohexyl-2-(4-methoxyphenyl)propanoate (7s): yield 34%; 49% ee, determined by HPLC (Daicel Chirapak AD-H, flow rate 1.0 mL/min, hexane / isopropanol / EtOH / TFA = 450 : 25 : 25 : 1, 254nm, Retention time: $t_{\text{major}} = 5.3$ min, and $t_{\text{minor}} = 10.1$ min.); ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 0.64-0.66 (m, 1H), 0.84-1.07 (m, 6H), 1.34-1.50 (m, 4H), 3.43 (d, $J = 10.5$ Hz, 1H), 3.67 (s, 3H), 3.81 (s, 3H), 3.87 (s, 3H), 4.01 (d, $J = 10.5$ Hz, 1H), 5.31 (d, $J = 10.5$ Hz, 1H), 5.51 (d, $J = 10.5$ Hz, 1H), 6.39 (d, $J = 4.5$ Hz, 2H), 6.60 (d, $J = 4.0$ Hz, 2H), 6.85 (d, $J = 9.0$ Hz, 2H), 7.44-7.50 (m, 4H), 7.60 (d, $J = 9.0$ Hz, 2H), 8.00 (d, $J = 8.0$ Hz, 2H), 8.32 (d, $J = 8.5$ Hz, 2H), 8.46 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) 26.14, 26.18, 26.94, 27.82, 33.88, 41.25, 51.88, 55.29, 55.73, 60.56, 65.28, 88.53, 113.31, 114.18, 114.61, 124.93, 125.15, 125.76, 128.23, 128.39, 128.83, 129.38, 129.98, 130.89, 131.51, 142.73, 151.38, 159.45, 172.93.

Deprotection of three-component product **7b**^[3]



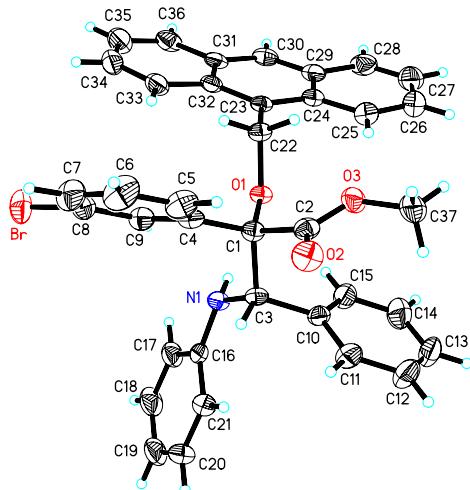
To a solution of compound **7b** (0.05 mmol) and NaI (15 mg, 2eq) in 0.5 mL of CH₃CN was added 15 μ L TMSCl (2 ~ 2.5eq) via a syringe pump at room temperature under an argon atmosphere. The reaction temperature was warmed to 50 °C and stirred over night. The reaction mixture was poured into water and stirred for 10 min. The aqueous phase was extracted with EtOAc. The organic phase was separated, washed with saturated aqueous Na₂S₂O₃, and dried over anhydrous MgSO₄. After evaporating the solvents, the crude product was purified by flash chromatography on silica gel (eluent: EtOAc/light petroleum ether = 1:30~1:20) to give β -amino- α -hydroxyl carboxylate compound **8**.



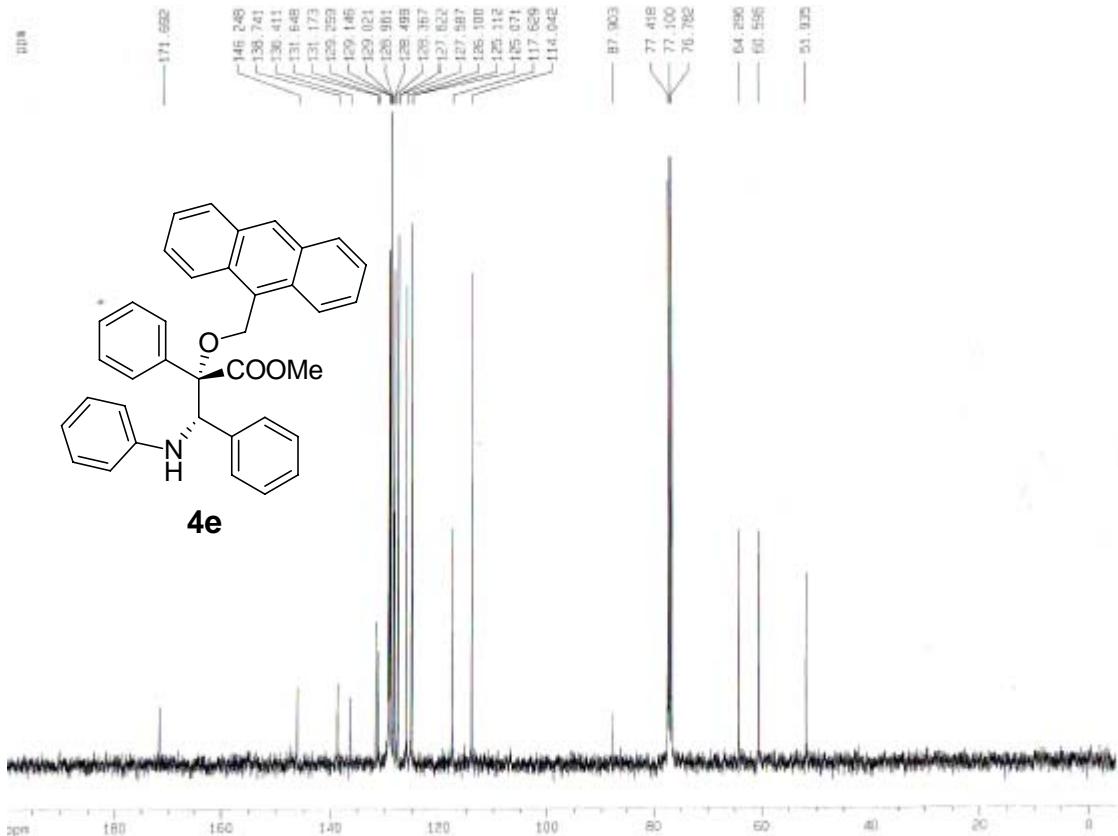
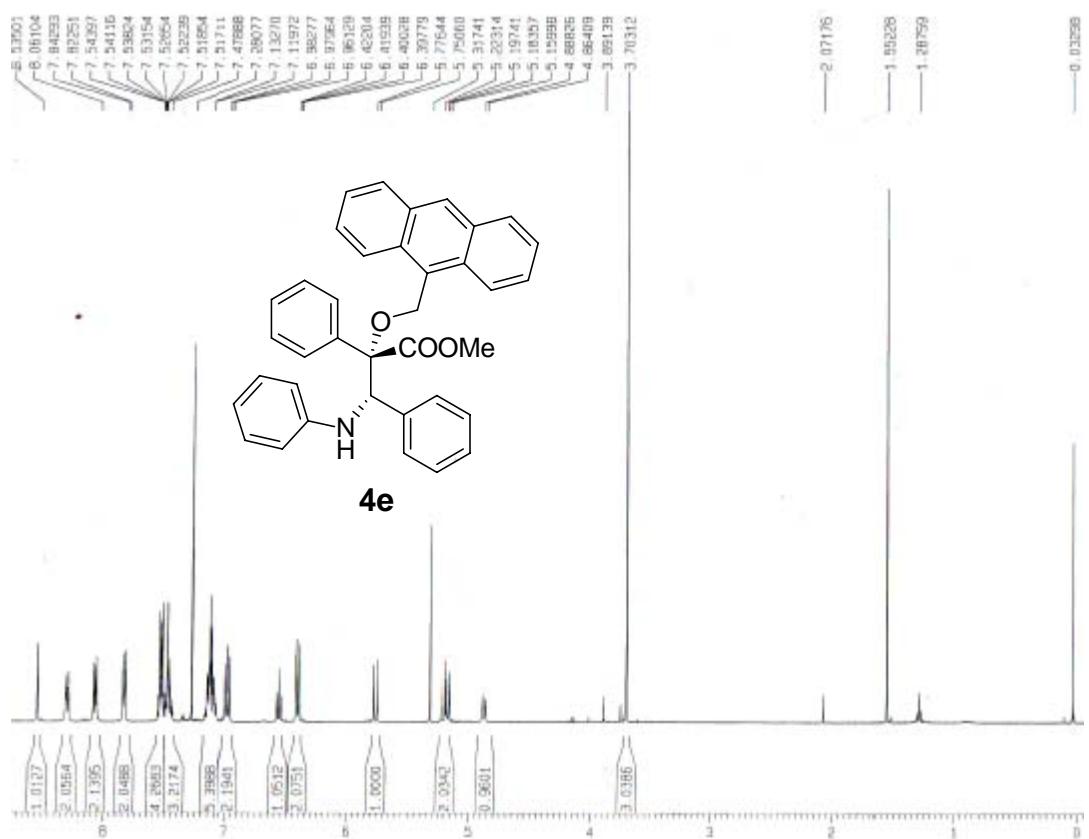
Reference and notes:

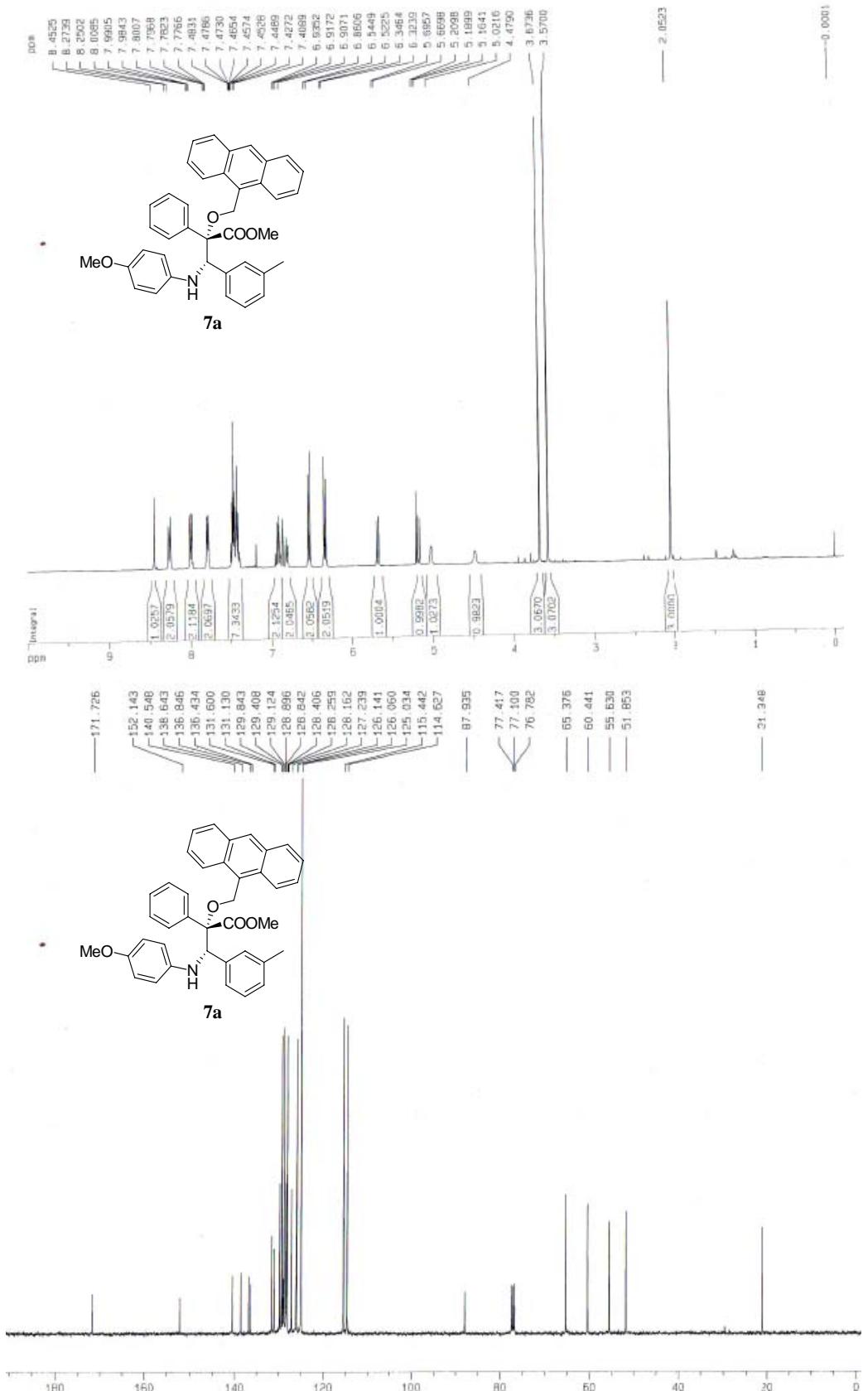
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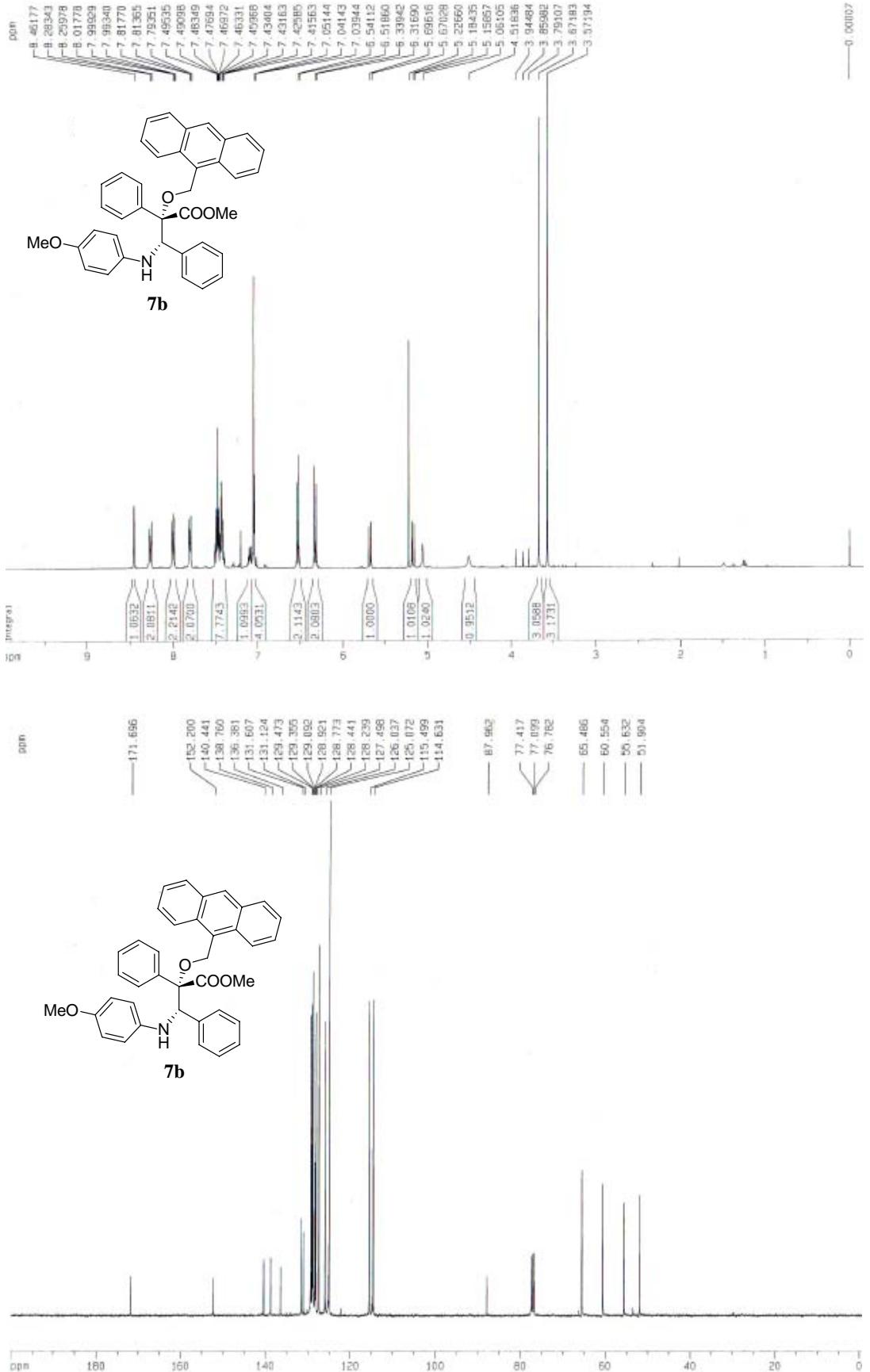
X-ray data of **7k**

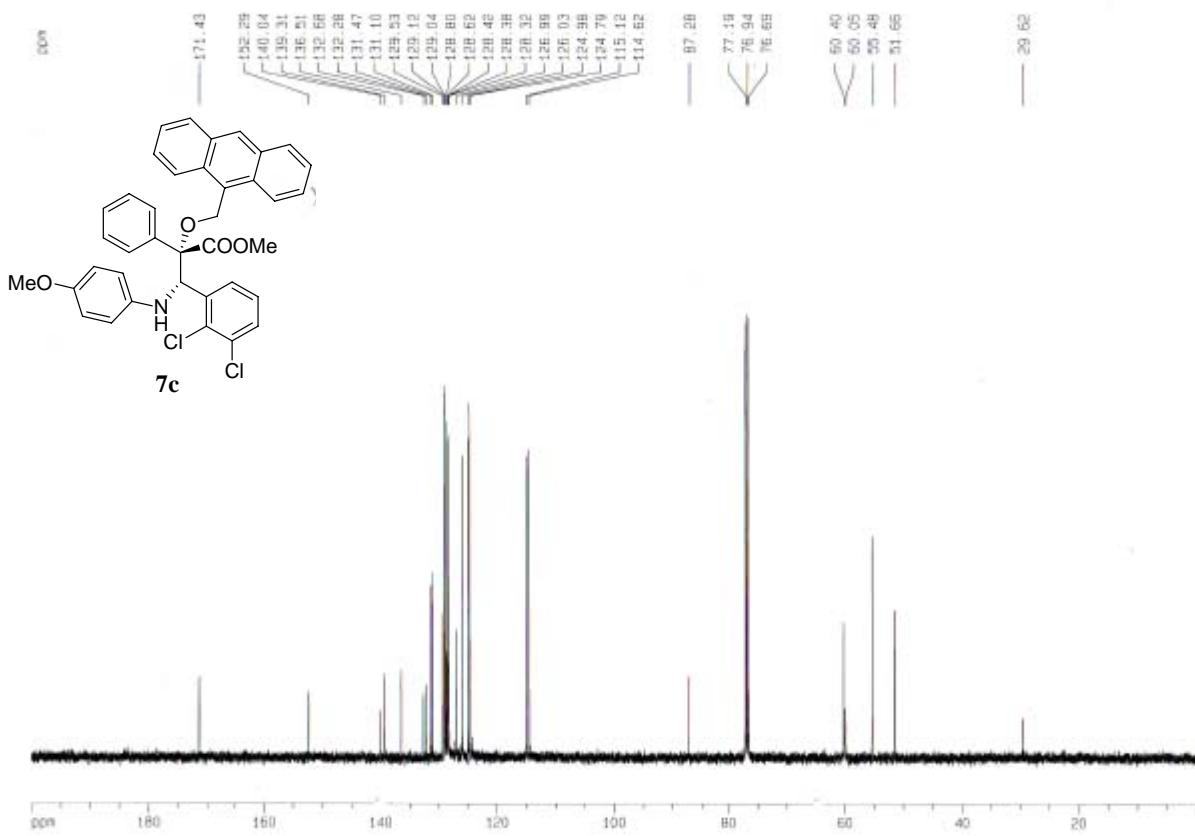
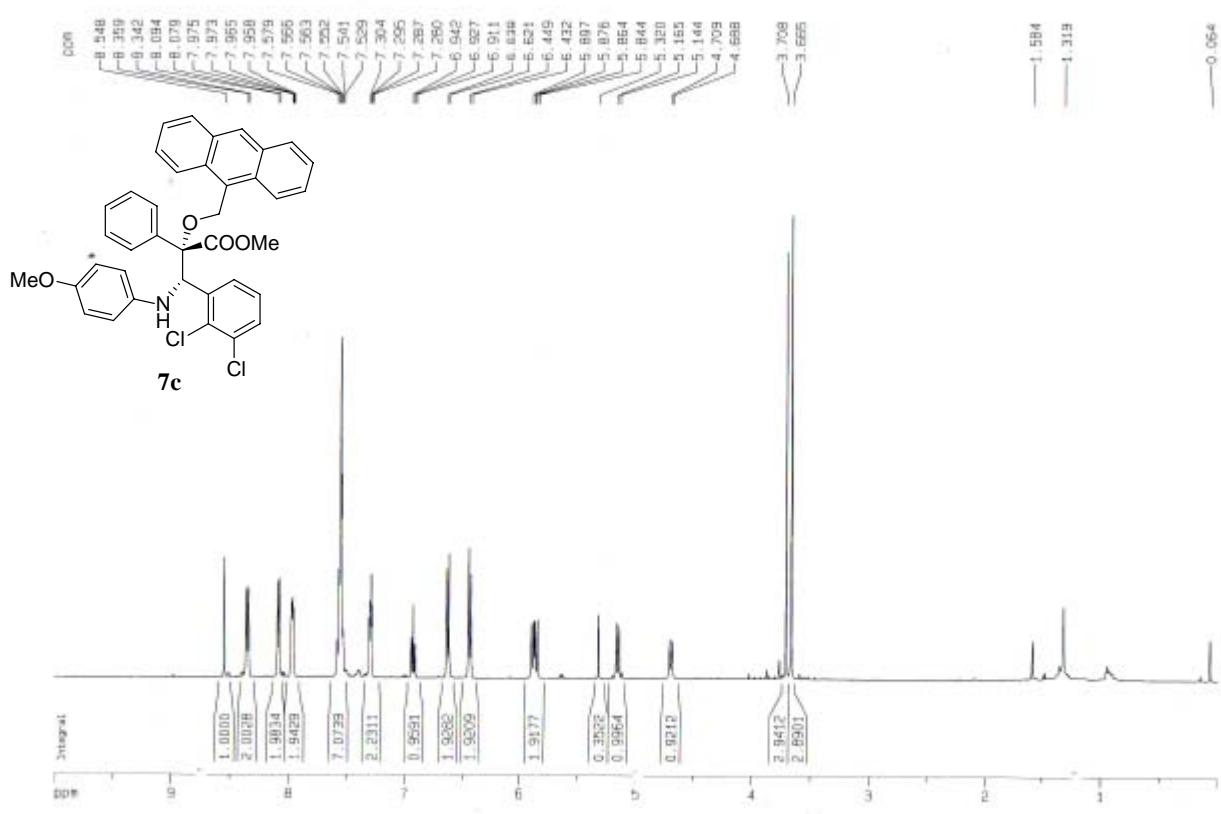


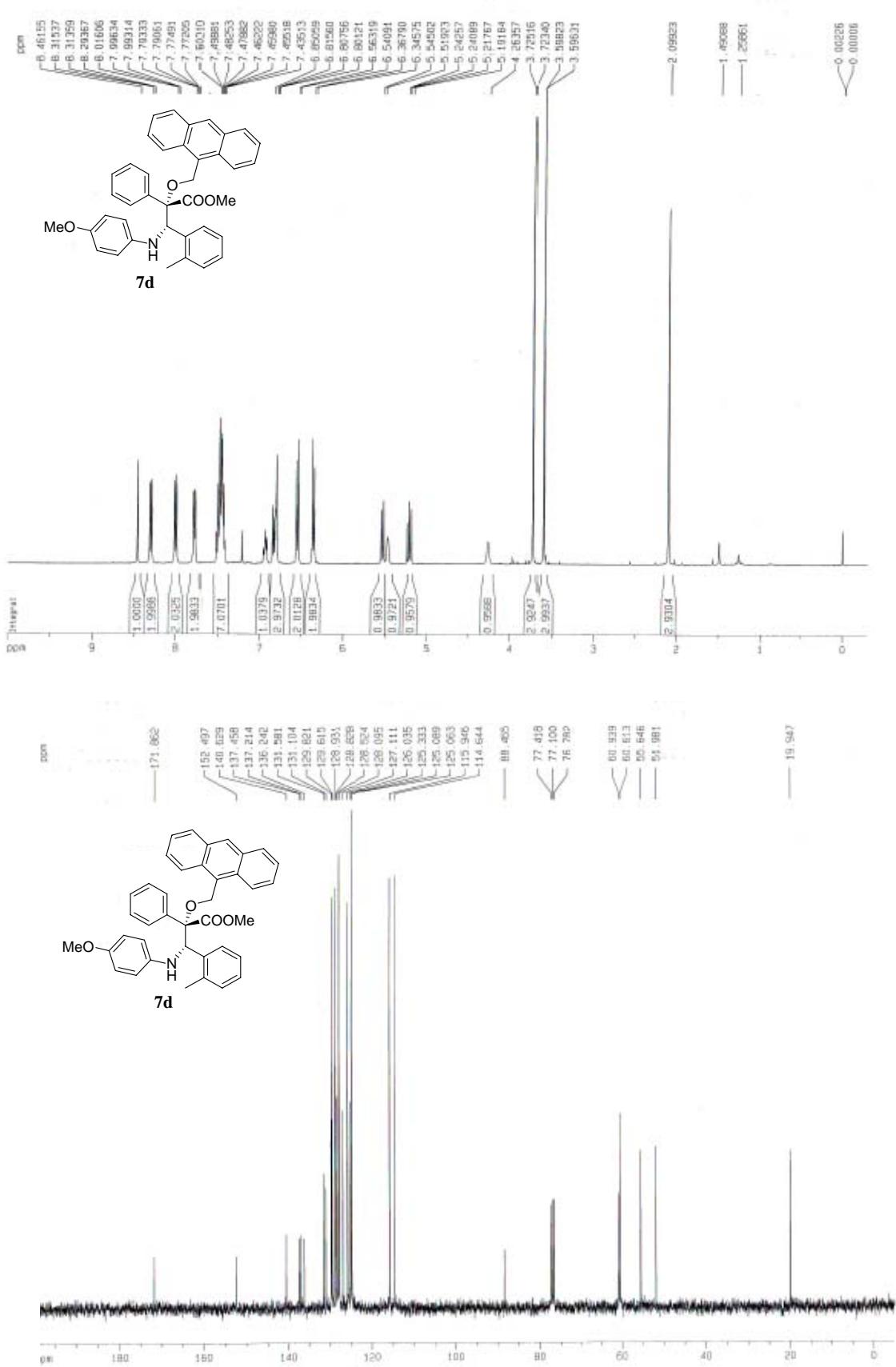
Identification code	cd2859
Empirical formula	C37 H30 Br N 03
Formula weight	616.53
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 6.6746(5) Å alpha = 90 deg. b = 20.6662(16) Å beta = 90 deg. c = 22.0533(18) Å gamma = 90 deg.
Volume	3042.0(4) Å^3
Z, Calculated density	4, 1.346 Mg/m^3
Absorption coefficient	1.389 mm^-1
F(000)	1272
Crystal size	0.421 x 0.237 x 0.180 mm
Theta range for data collection	1.35 to 27.00 deg.
Limiting indices	-8<=h<=8, -22<=k<=26, -28<=l<=25
Reflections collected / unique	18175 / 6601 [R(int) = 0.0908]
Completeness to theta = 27.00	99.6 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.77861
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6601 / 1 / 384
Goodness-of-fit on F^2	0.954
Final R indices [I>2sigma(I)]	R1 = 0.0532, wR2 = 0.0962
R indices (all data)	R1 = 0.0799, wR2 = 0.1048
Absolute structure parameter	-0.012(8)
Largest diff. peak and hole	0.331 and -0.345 e. Å^-3

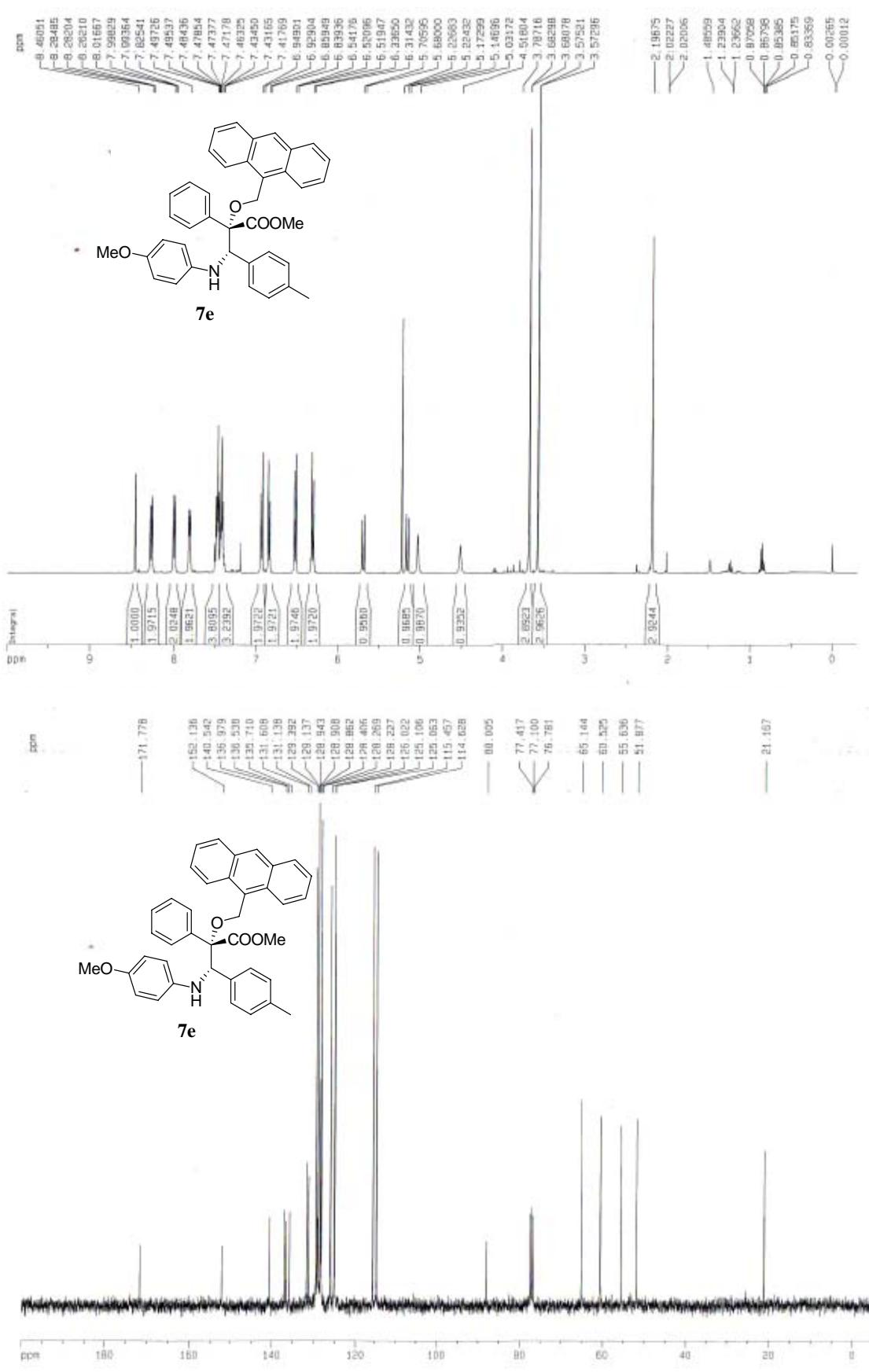


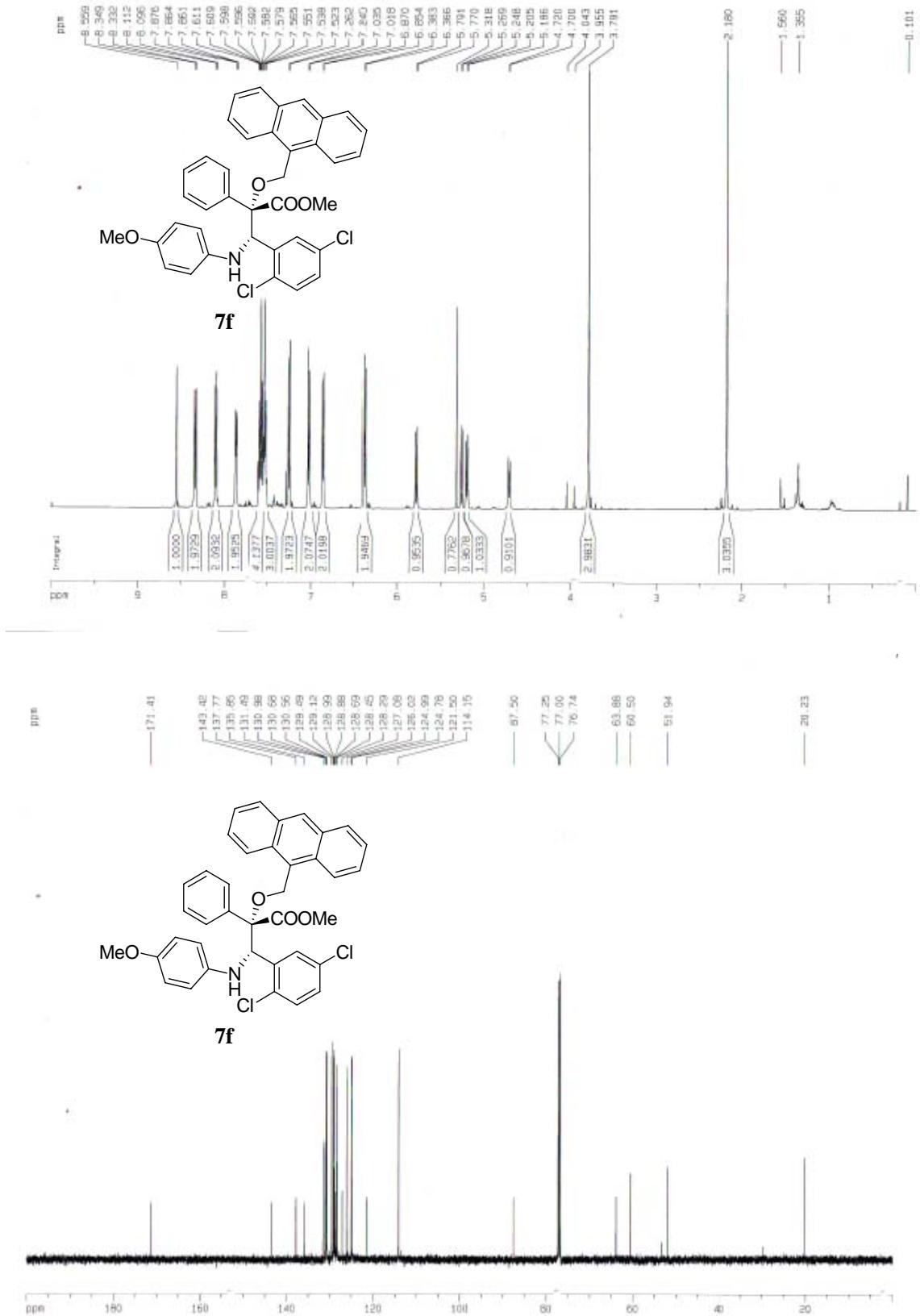


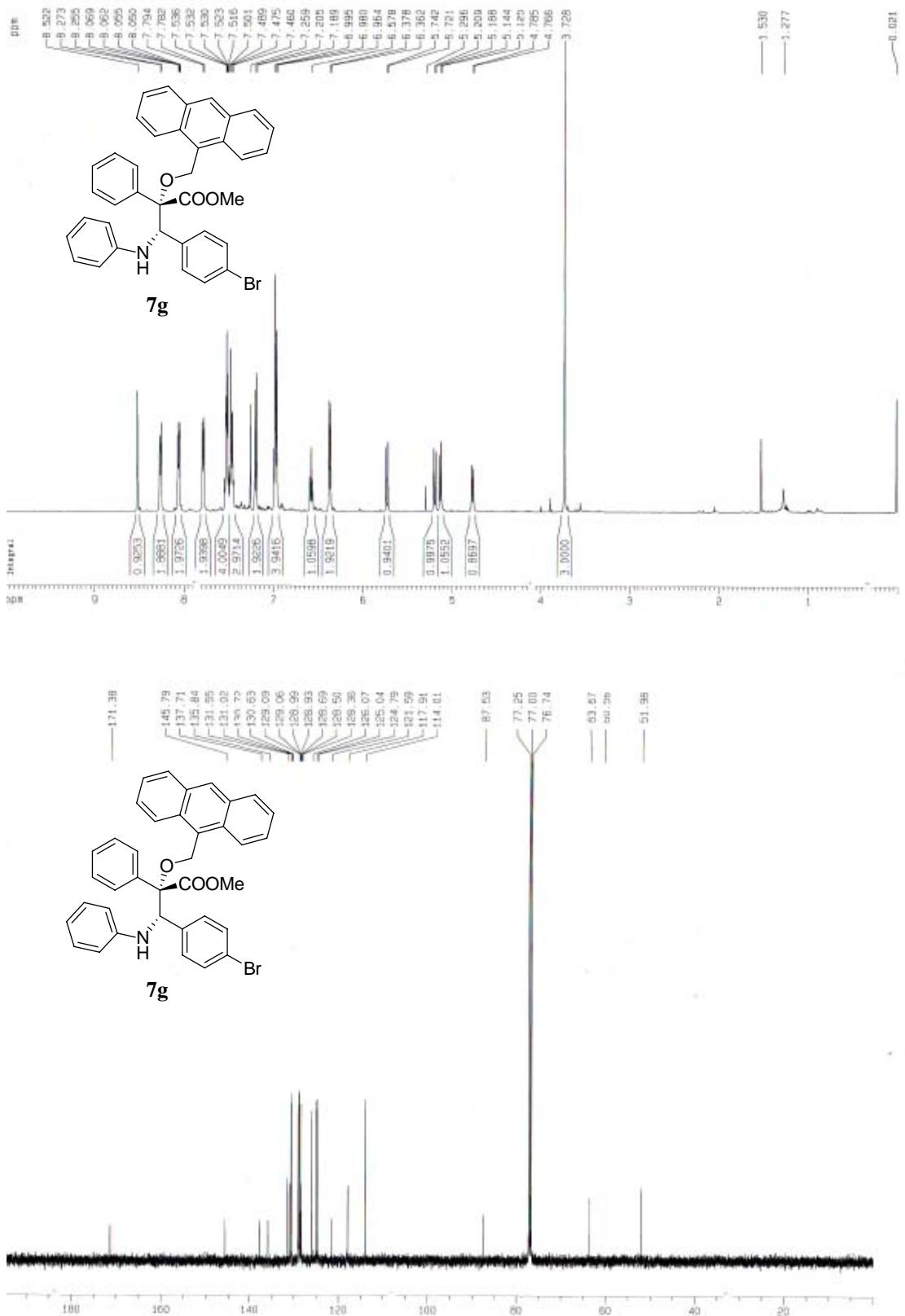


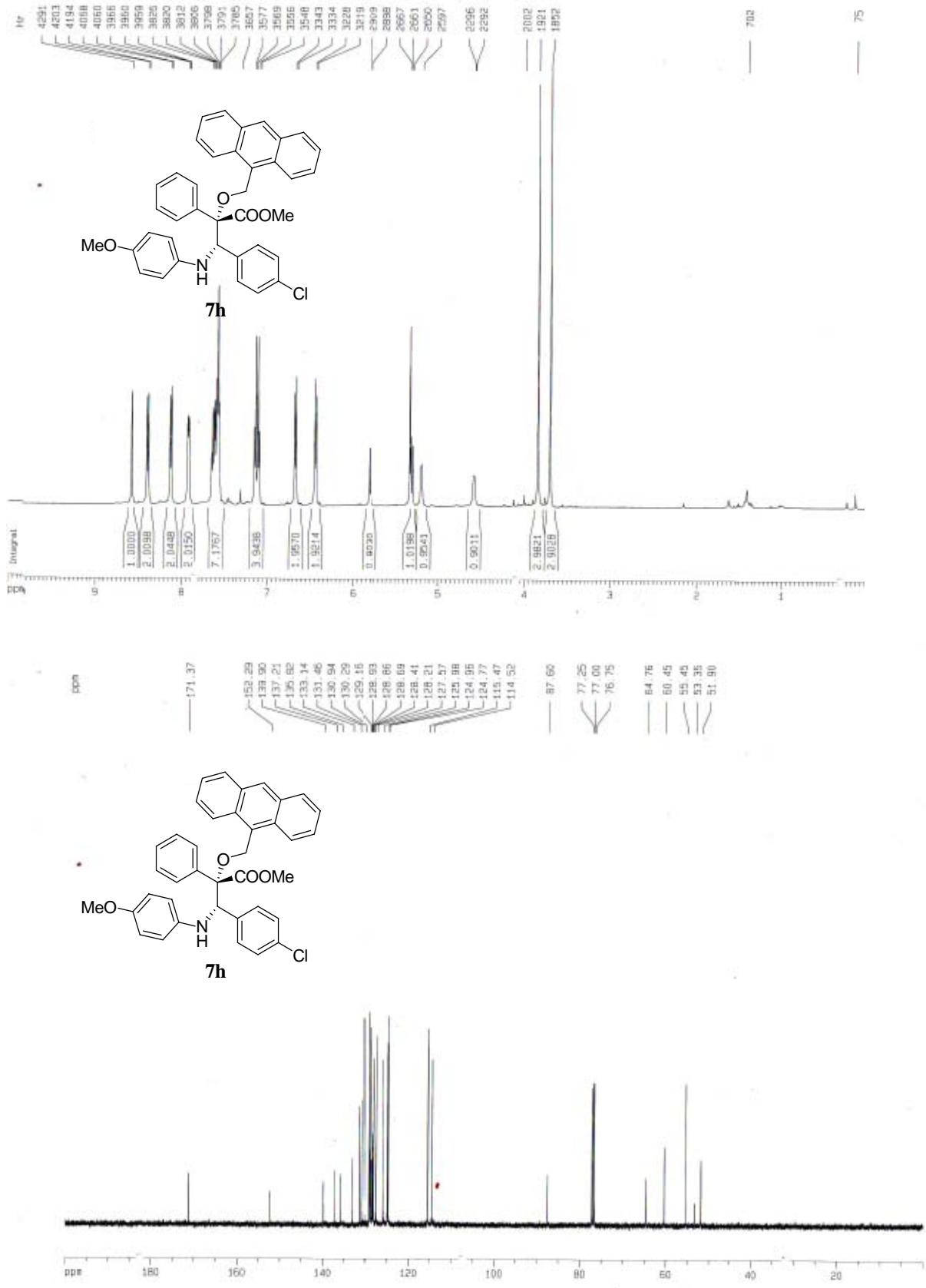


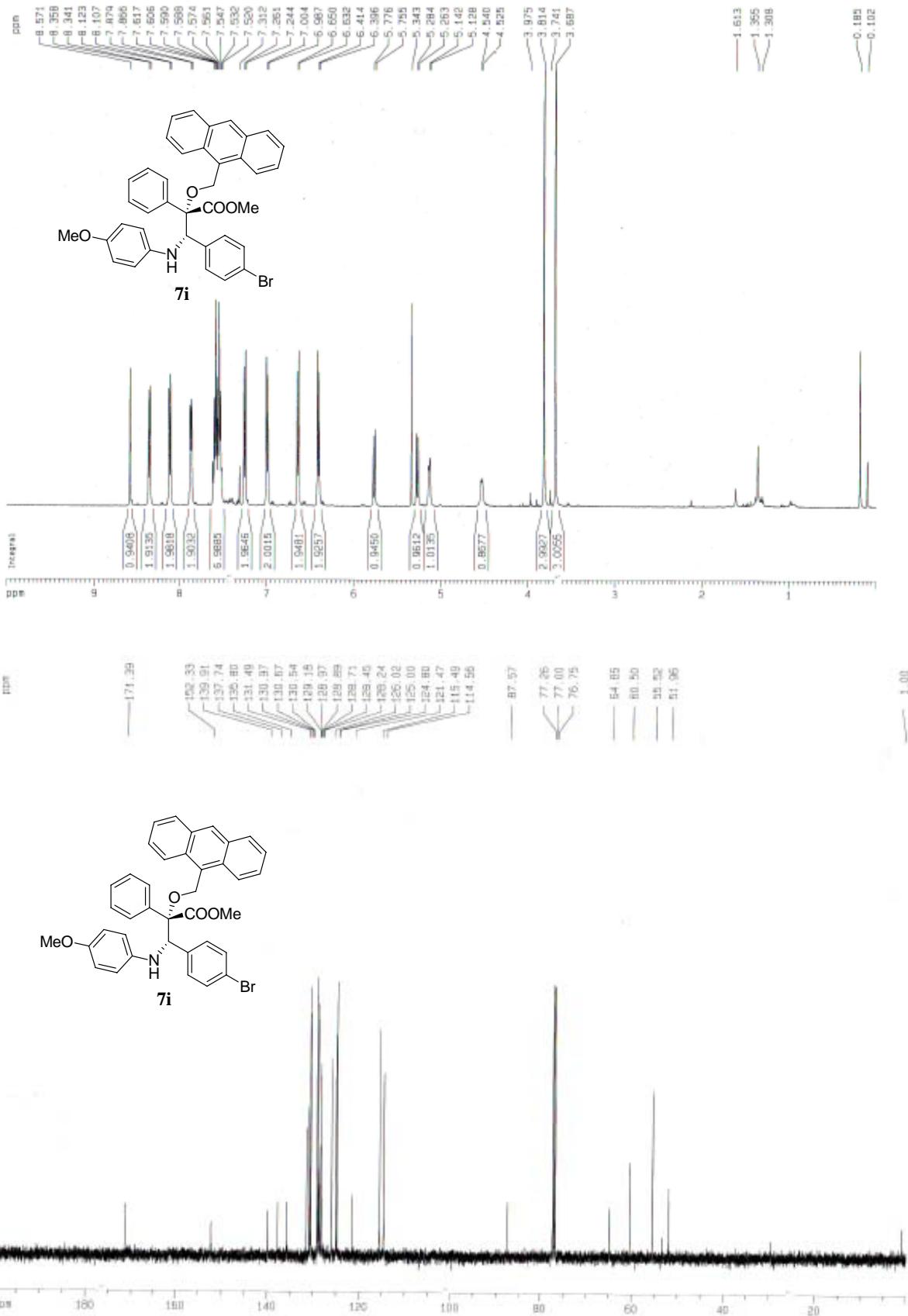


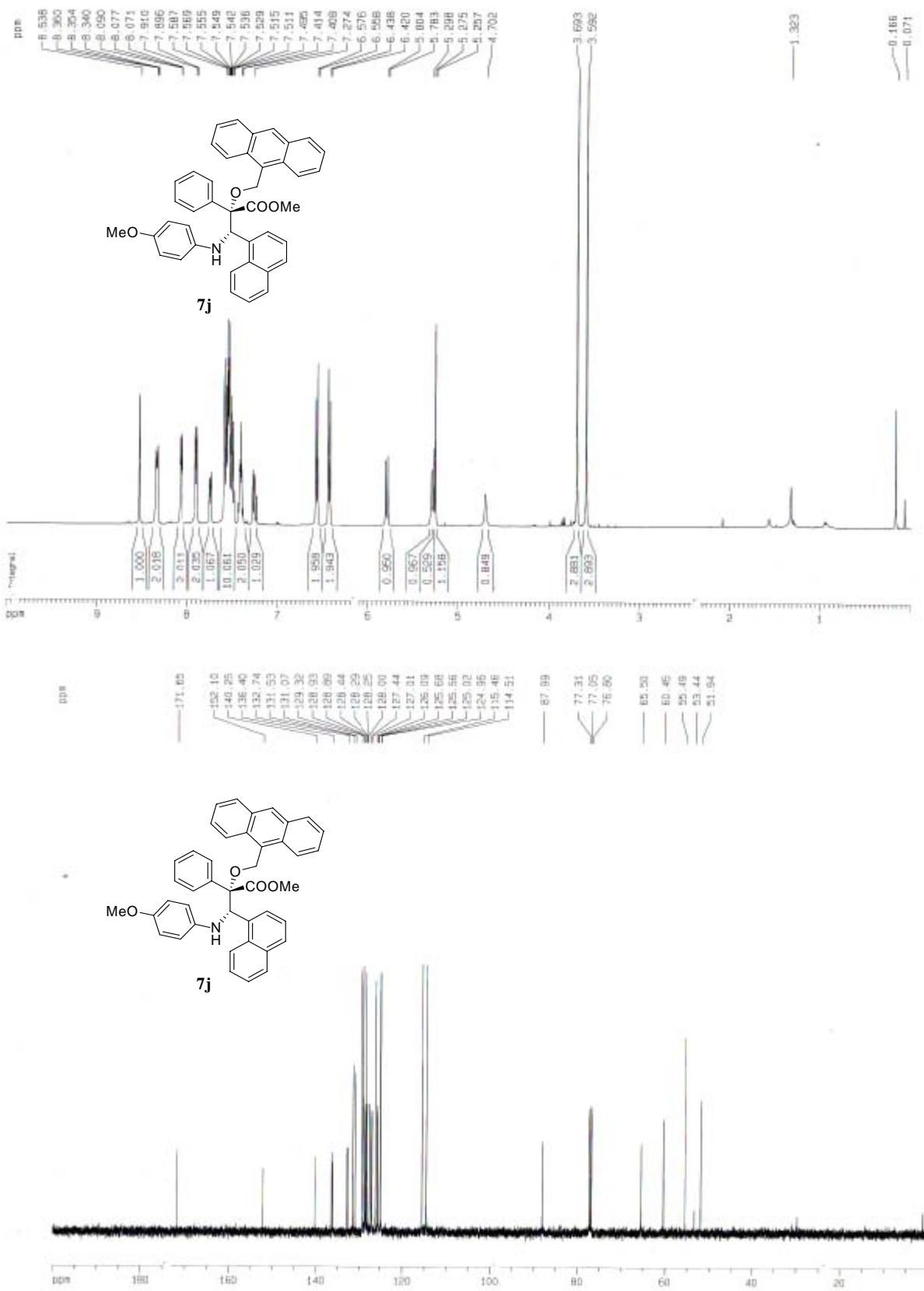


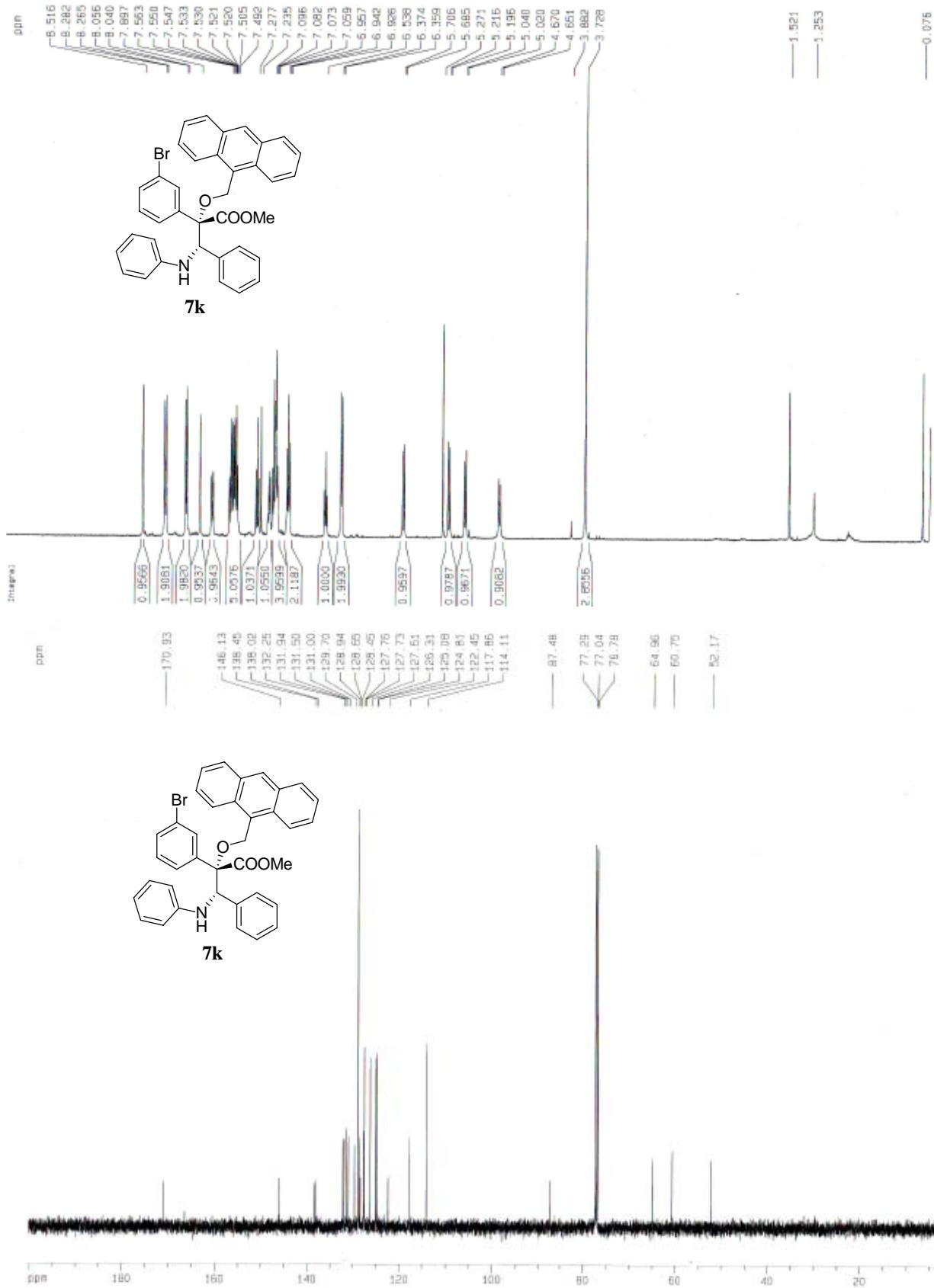


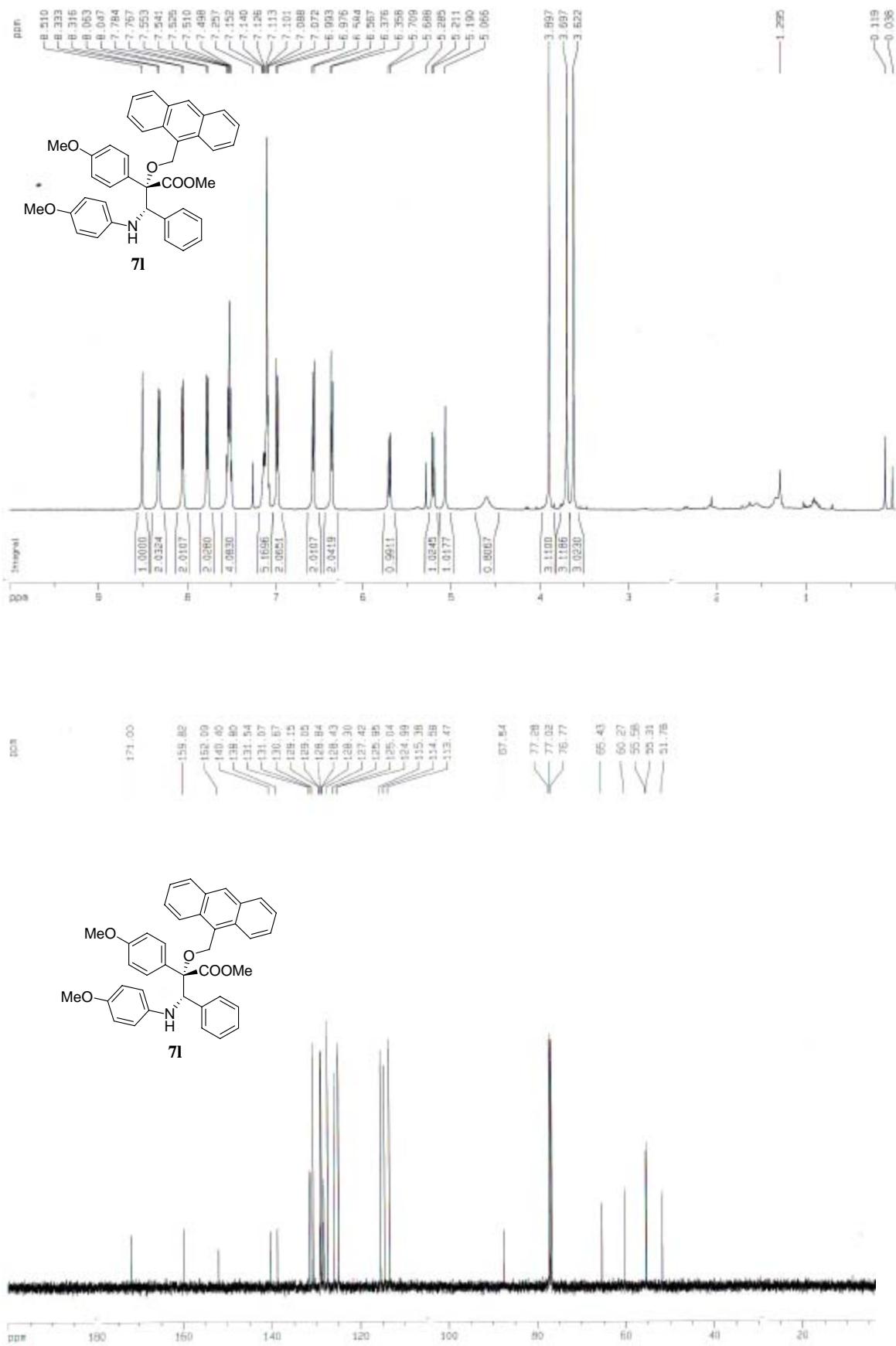


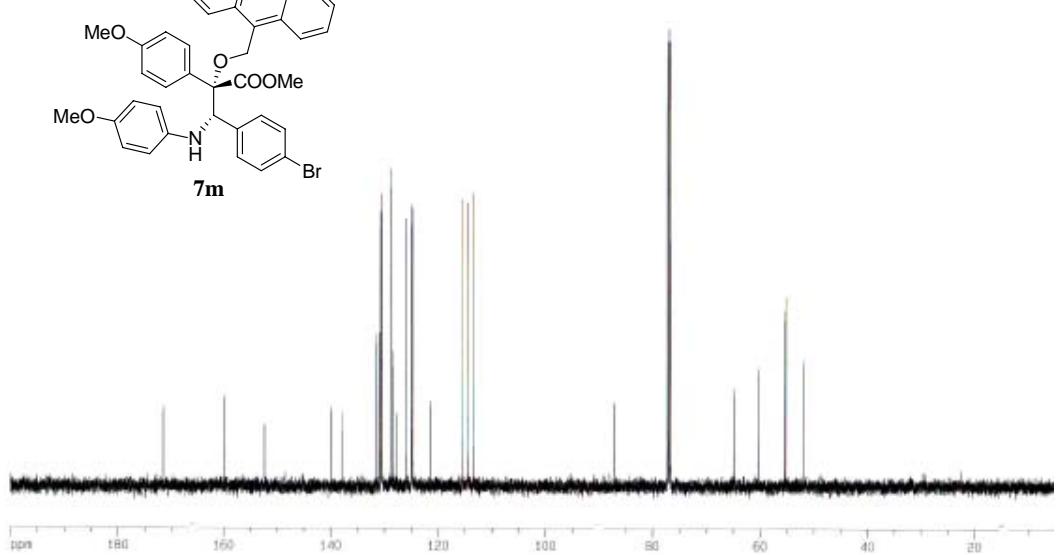
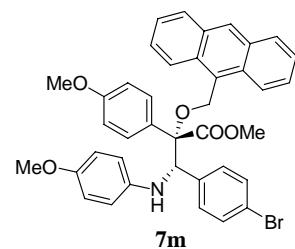
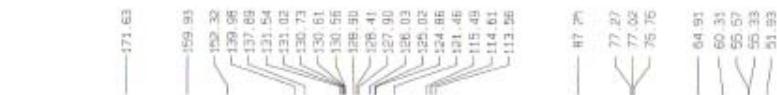
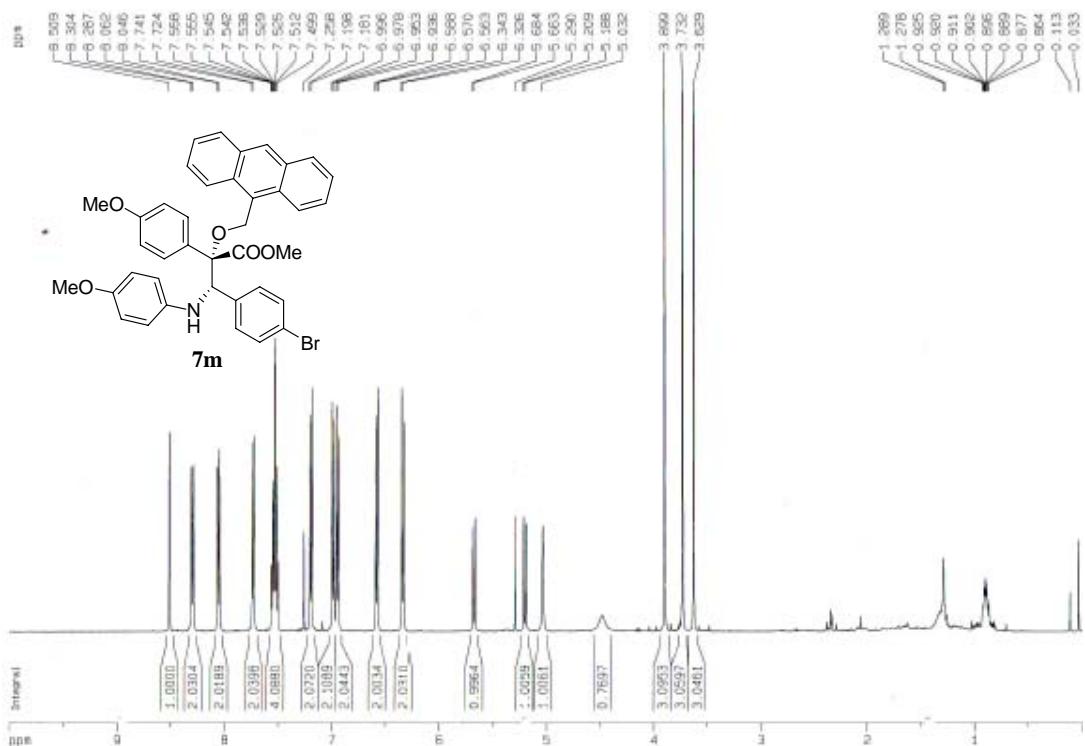


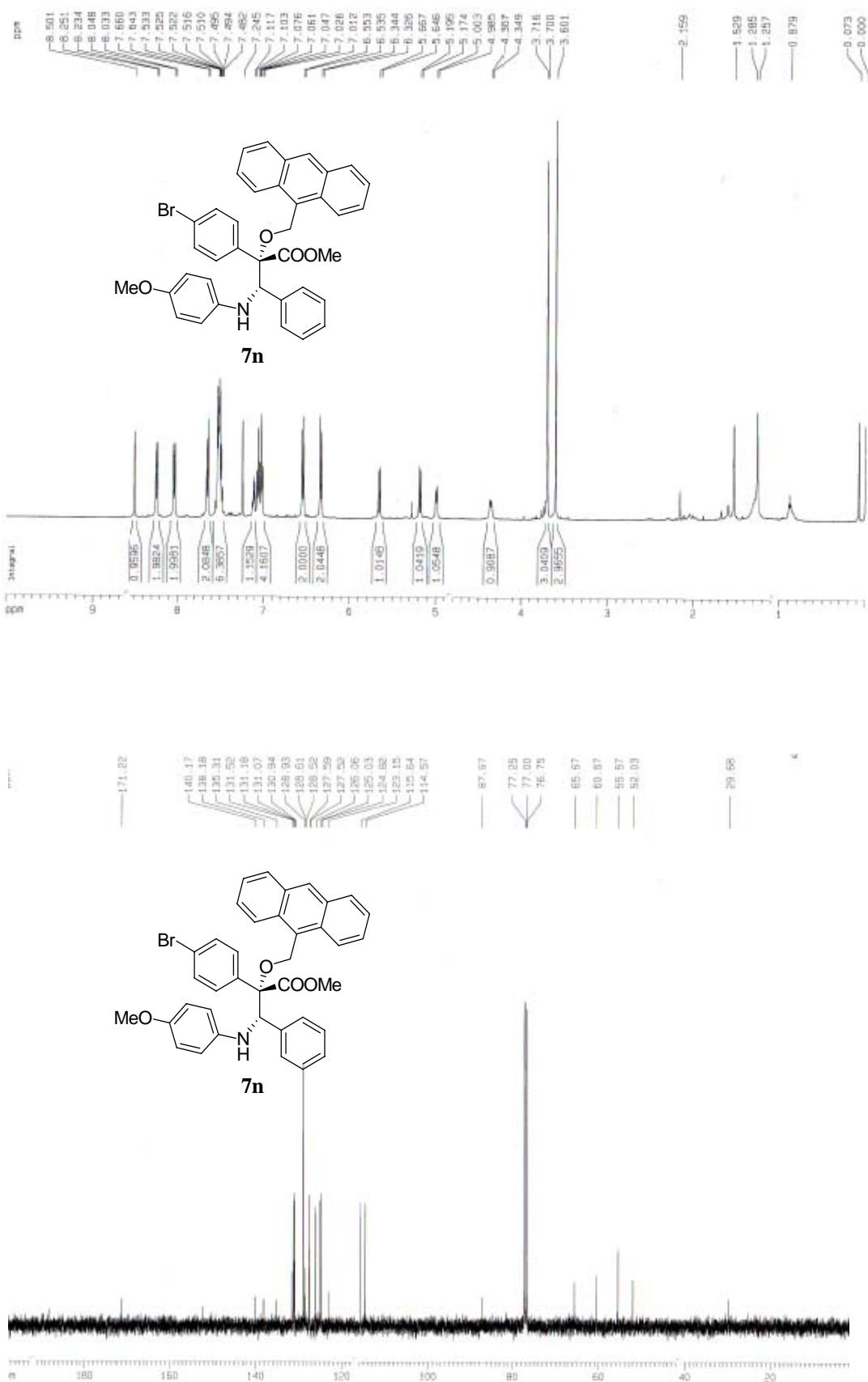


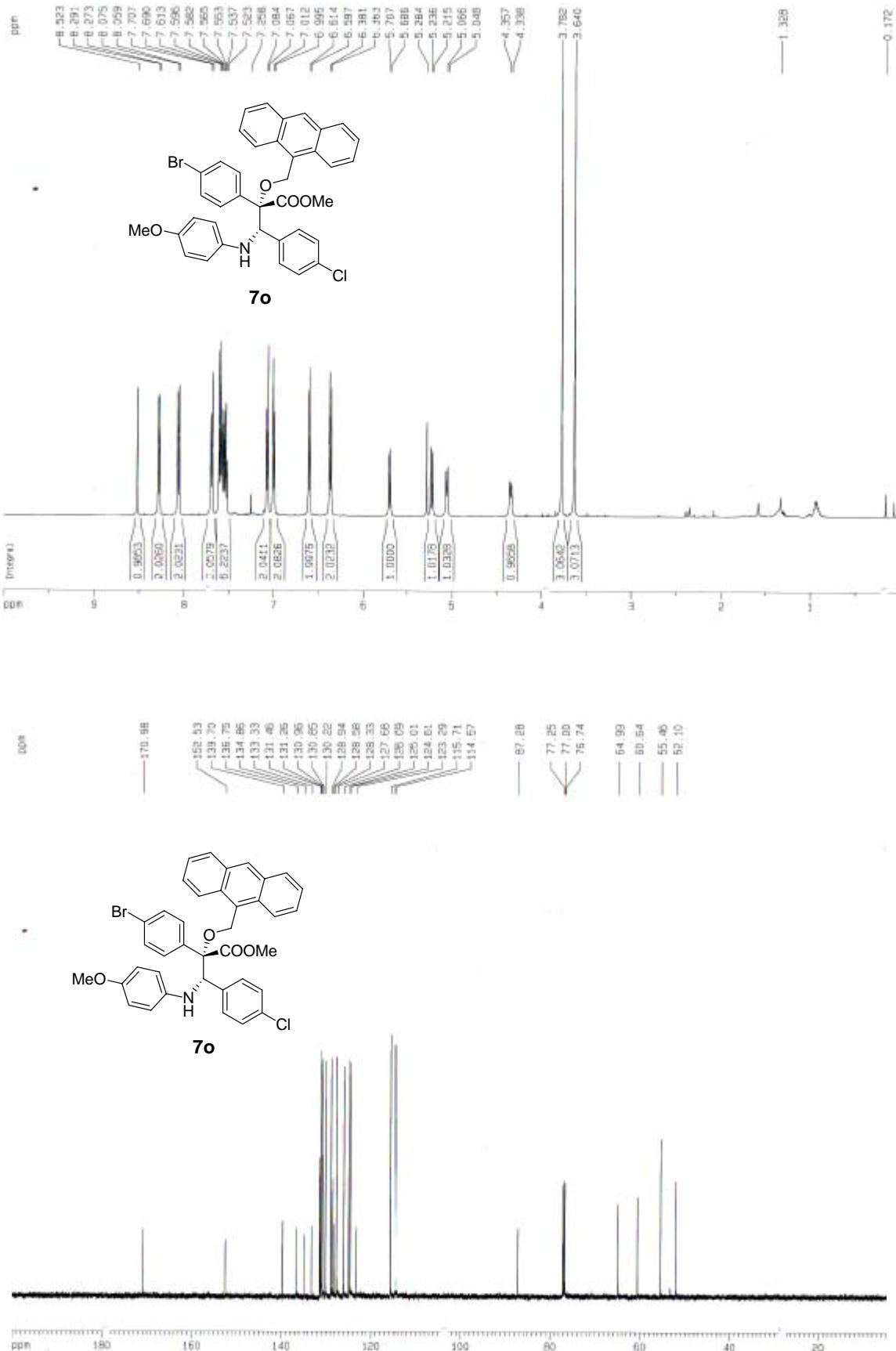


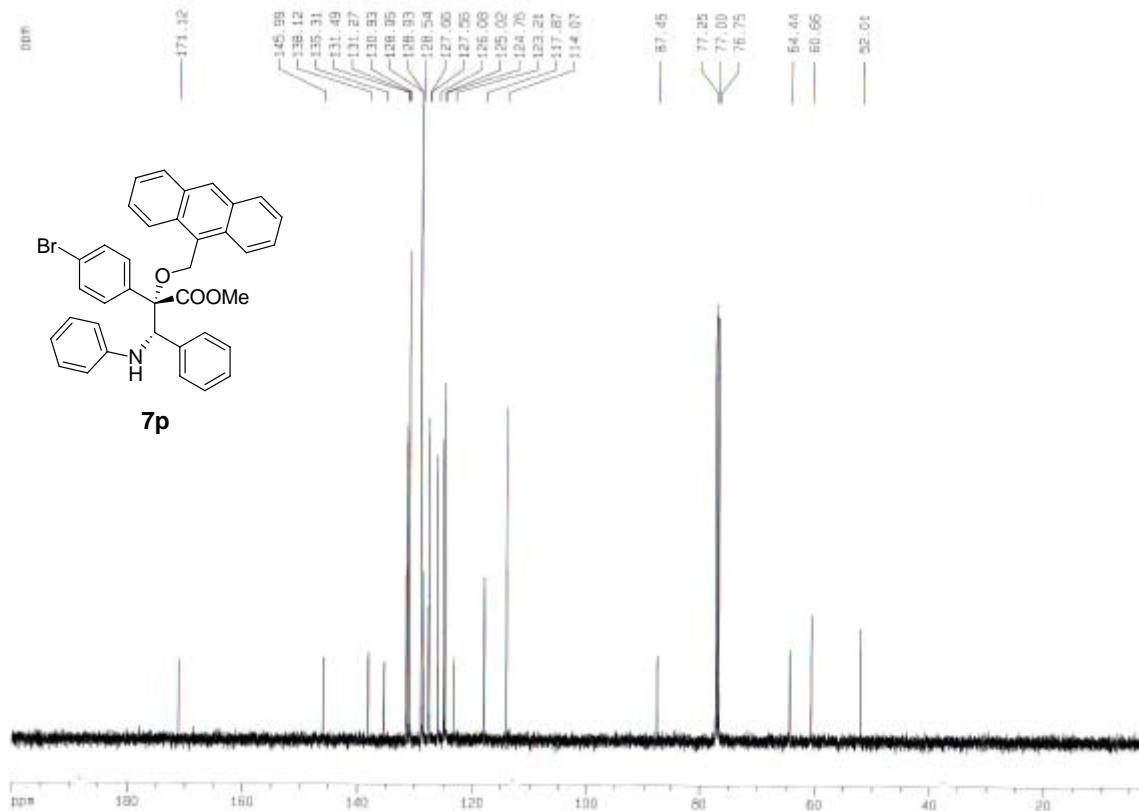
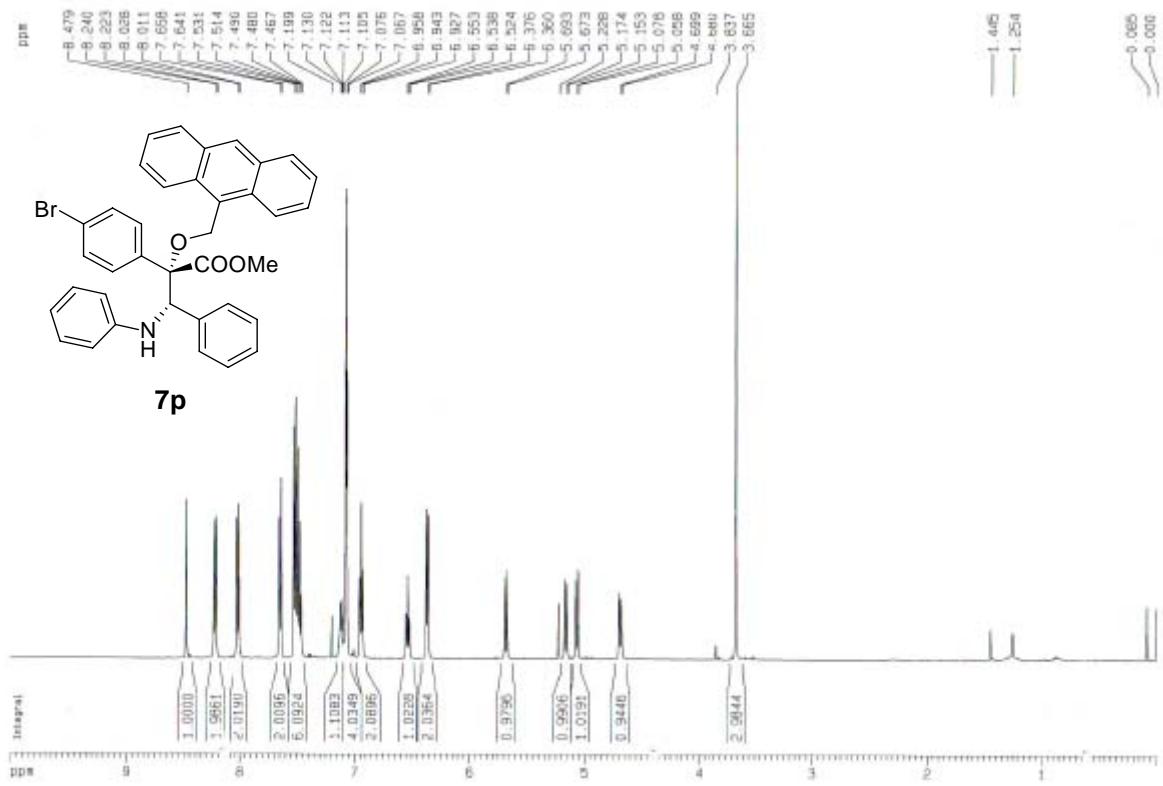


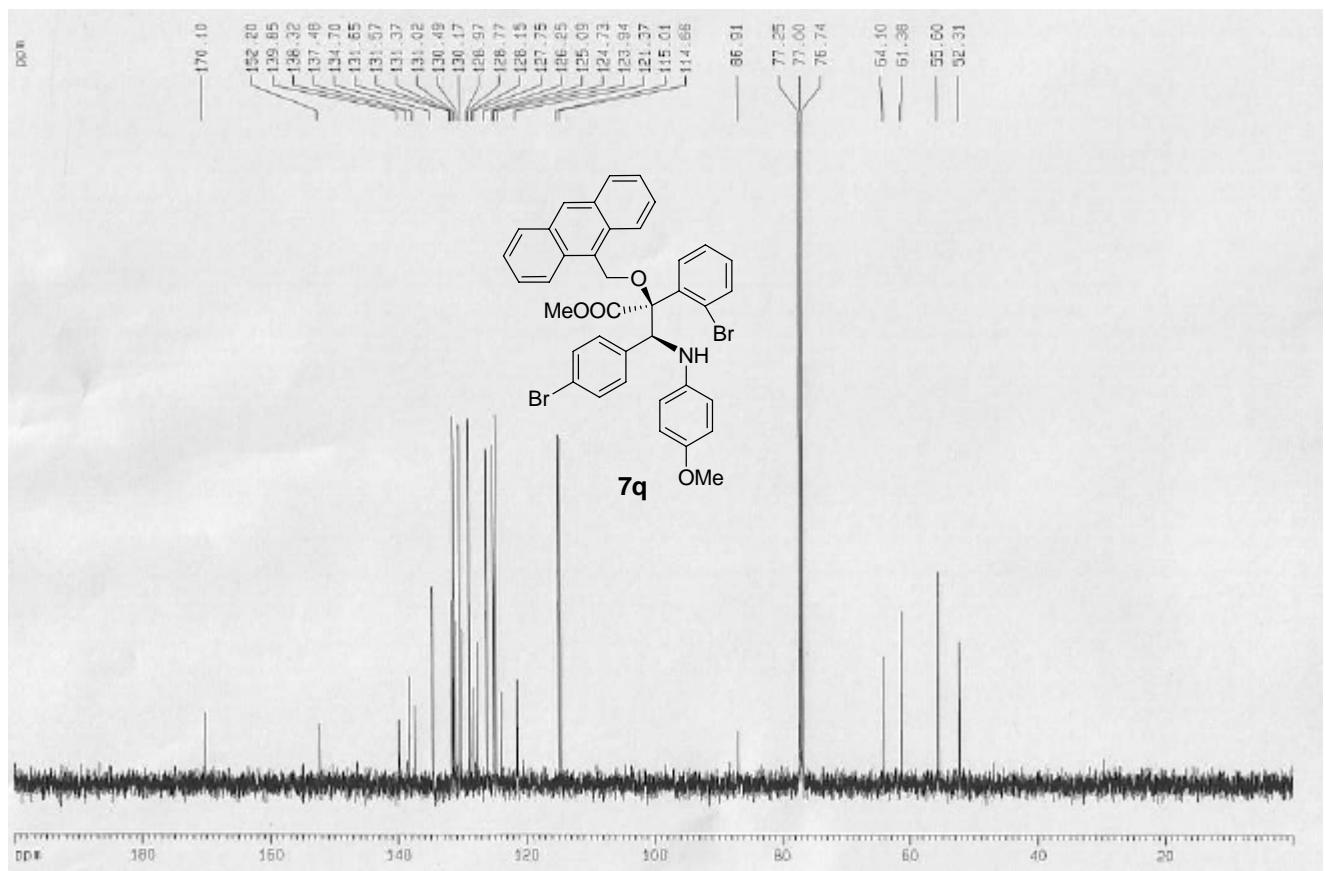
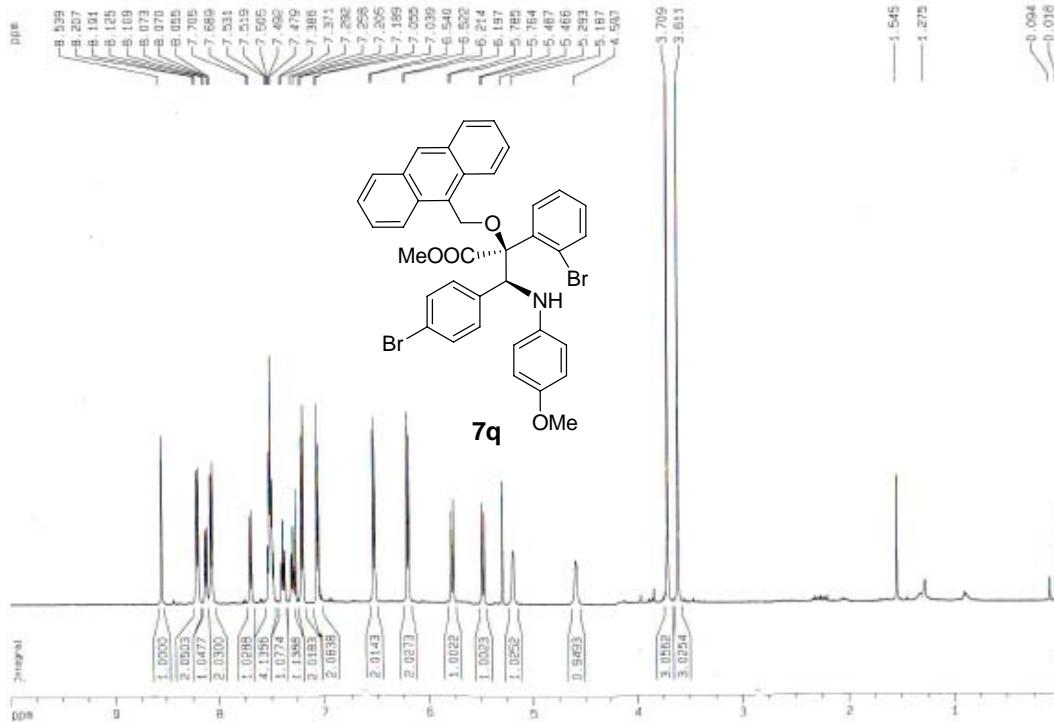


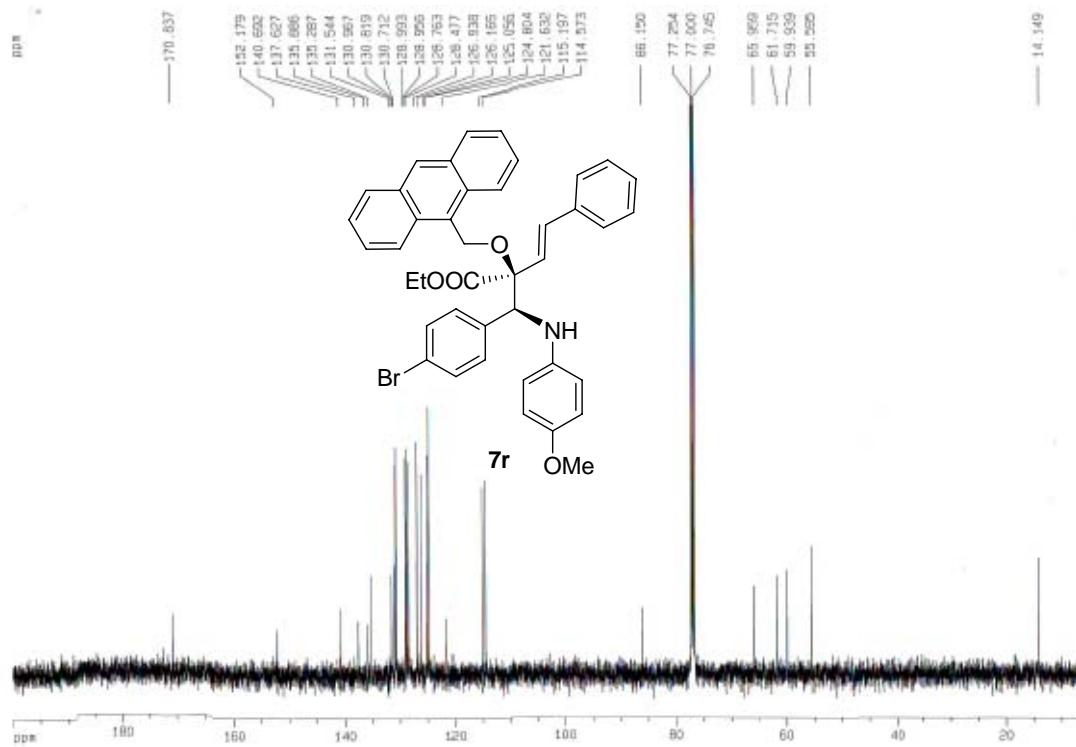
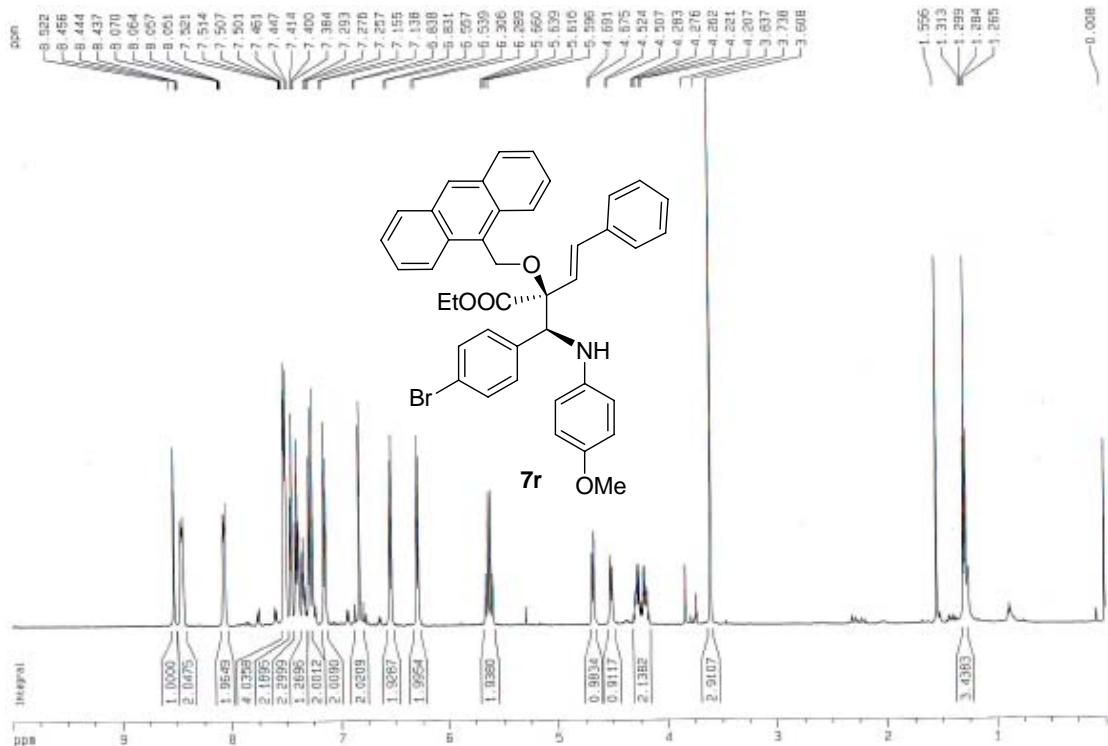


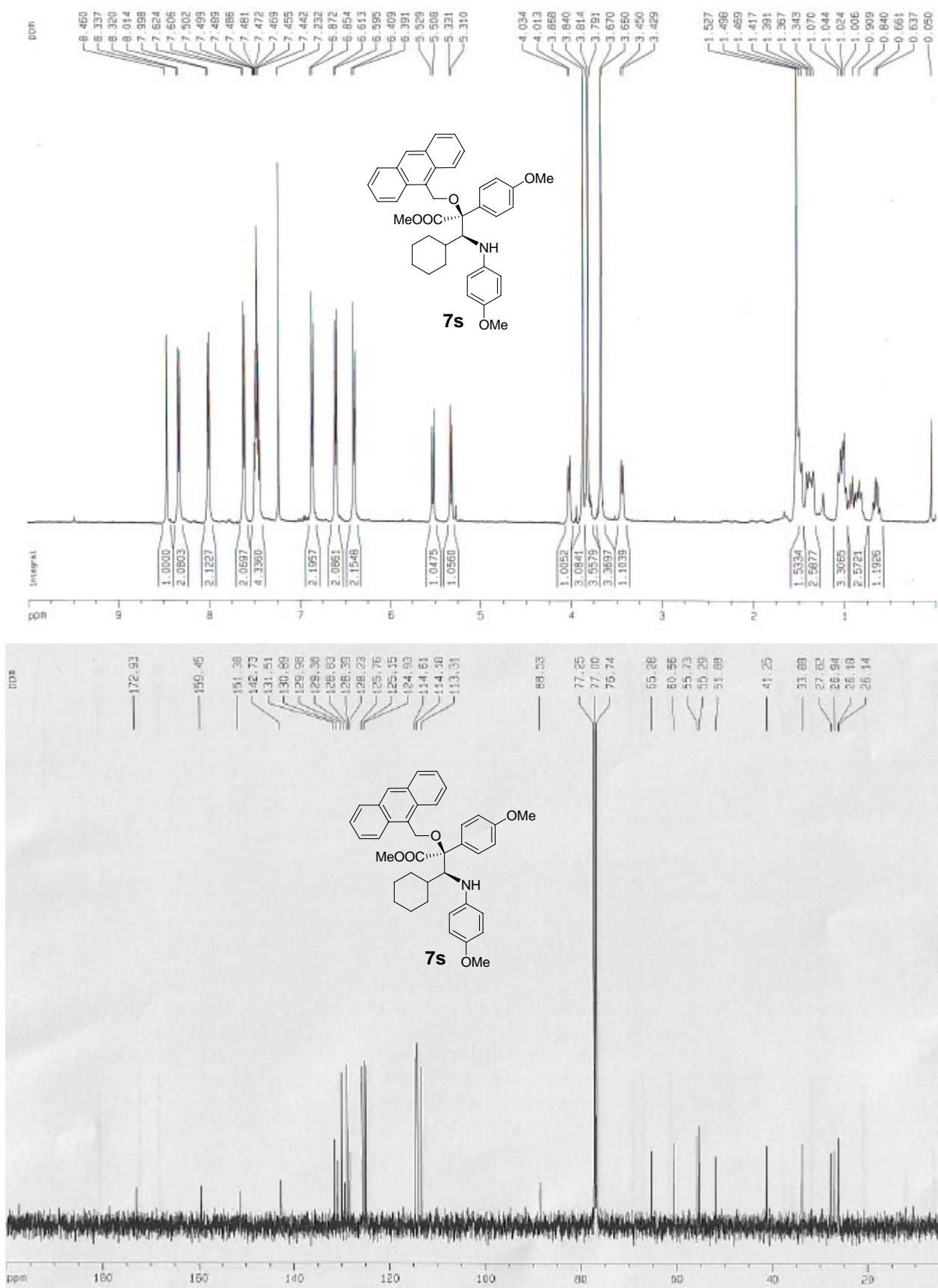


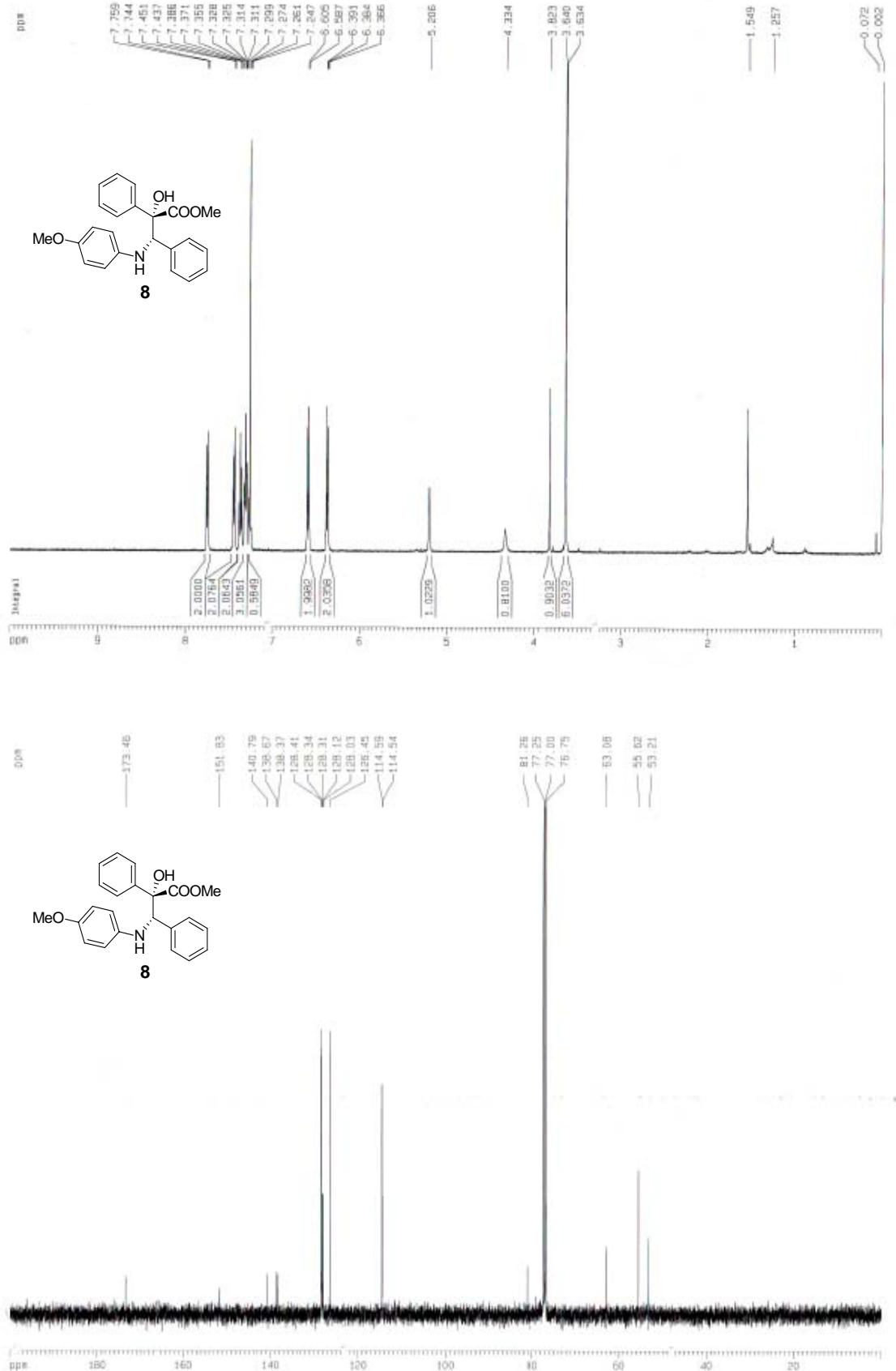












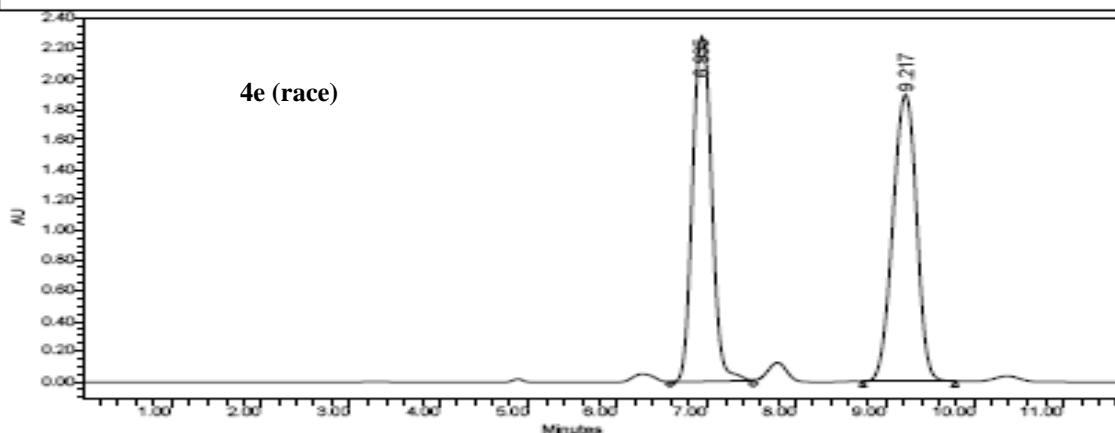
USTC

Project Name: xx0
Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xx0-en-meso-11011	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/11/2007 3:27:43 PM
Vial:	1	Acq. Method:	xx0109
Injection #:	4	Date Processed:	11/2/2007 5:59:03 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



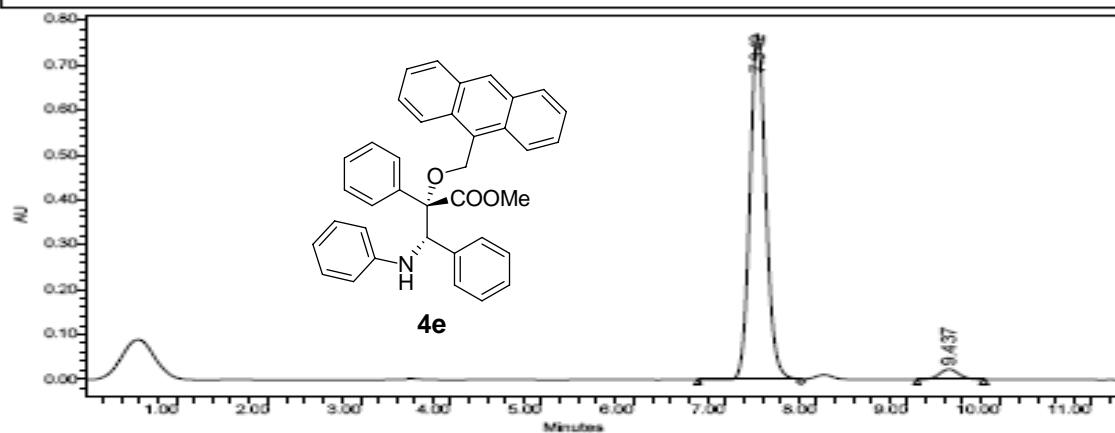
USTC

Project Name: xx0
Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xx0-en-cat2--20-11141	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/14/2007 8:43:32 PM
Vial:	1	Acq. Method:	xx0109
Injection #:	2	Date Processed:	11/29/2007 11:40:38 AM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



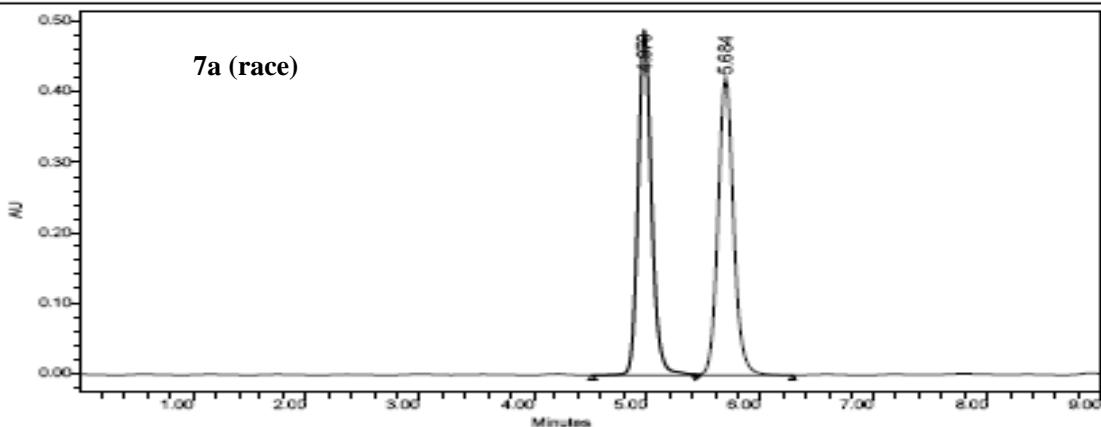
USTC

Project Name: xxr
Reported by User: System

✓Breeze

SAMPLE INFORMATION

Sample Name:	xxr-6-meso-1119	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/19/2007 5:12:30 PM
Vial:	1	Acq. Method:	XXF20%
Injection #:	1	Date Processed:	11/19/2007 5:24:36 PM
Injection Volume:	20.00 μ l	Channel Name:	2487/Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



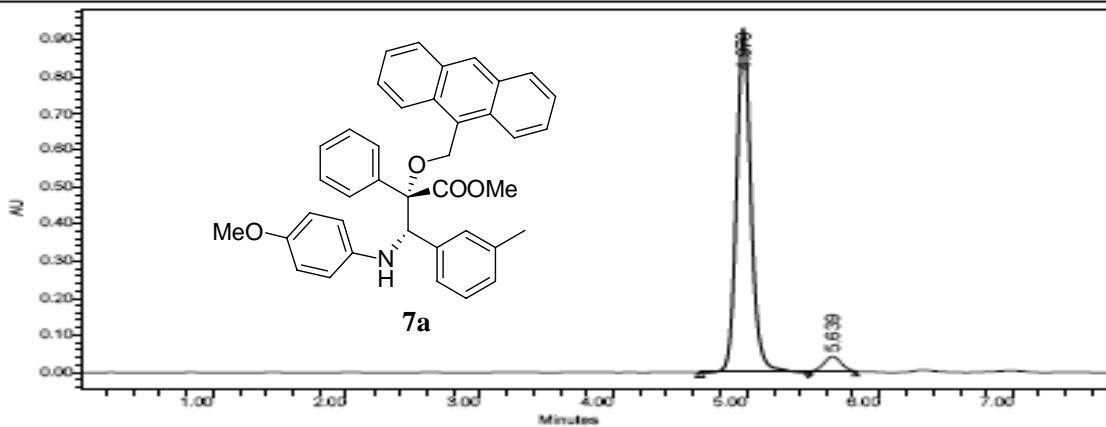
USTC

Project Name: xxr
Reported by User: System

✓Breeze

SAMPLE INFORMATION

Sample Name:	xxr-6-cat-1119	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/19/2007 5:23:39 PM
Vial:	1	Acq. Method:	XXF20%
Injection #:	2	Date Processed:	11/19/2007 5:33:52 PM
Injection Volume:	20.00 μ l	Channel Name:	2487/Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



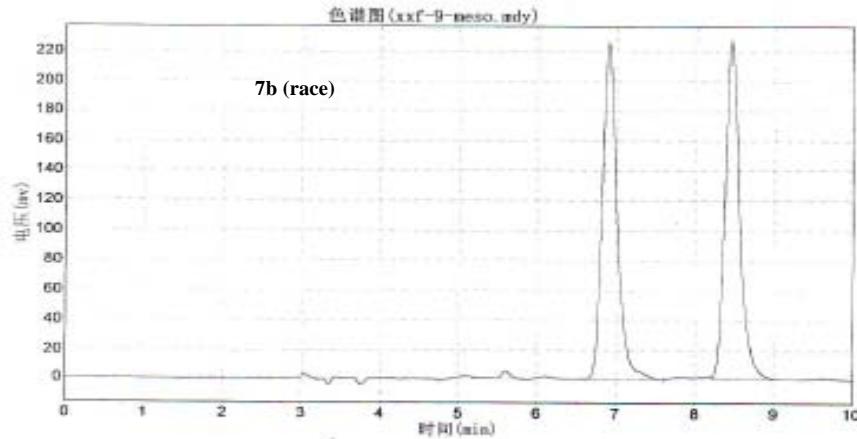
实验内容简介：

TB

254nm

n-Hex/i-PrOH/EtOH/TPA=450:25:25:1

1.0ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		6.900	224327.063	2961788.750	50.2167
2		8.453	225108.875	2936227.500	49.7833
总计			449435.938	5898016.250	100.0000

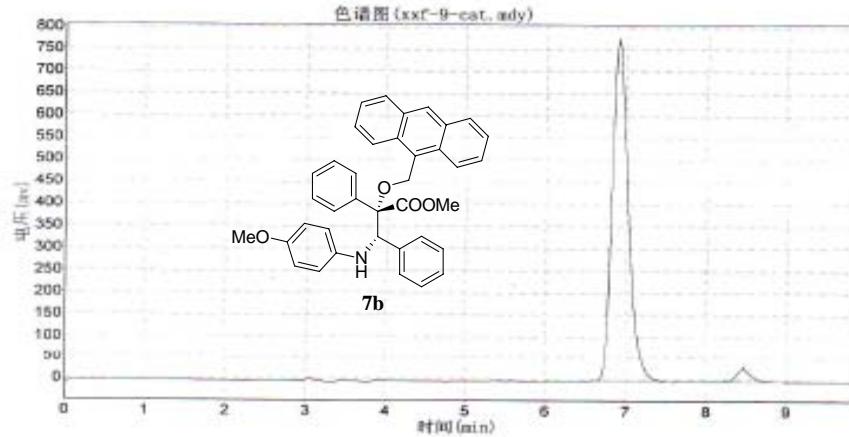
实验内容简介：

TB

254nm

n-Hex/i-PrOH/EtOH/TPA=450:25:25:1

1.0ml/min



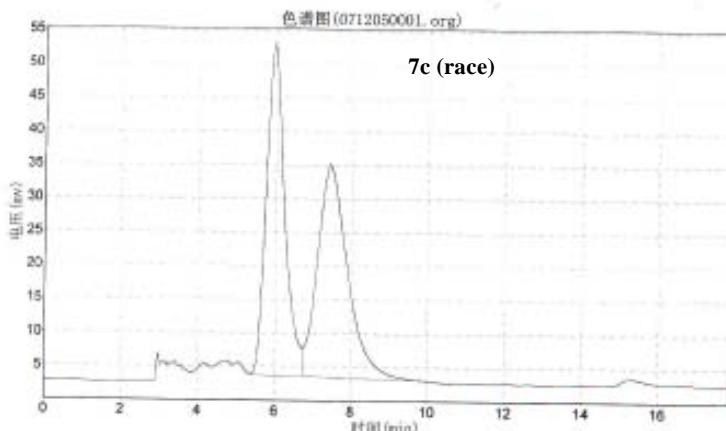
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		6.905	772720.313	10922602.000	96.7674
2		8.462	28461.967	364884.250	3.2326
总计			801182.279	11287486.250	100.0000

AD-H
n-hex/EtOH/DEA=90:10:0.1
254nm
1.0ml/min

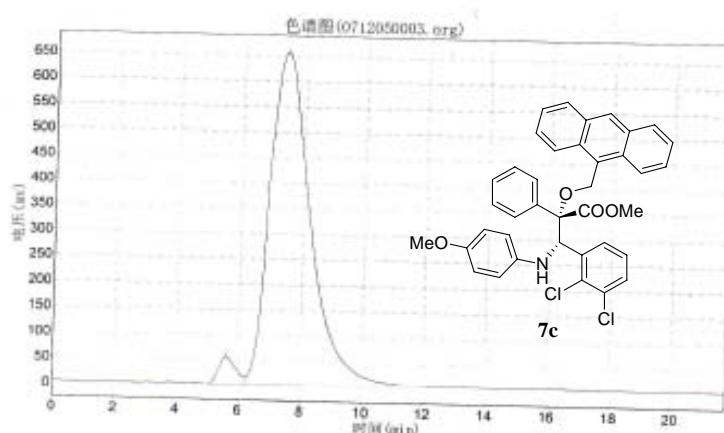
实验时间: 2007-12-05, 上午 10:13:55
报告文件:C:\浙大智达\N2000\浙大\0712050001.org

实验者: hujuan
报告时间: 2007-12-05, 上午 10:52:02
积分方法: 面积归一法



实验时间: 2007-12-05, 下午 03:45:35
报告文件:C:\浙大智达\N2000\浙大\0712050003.org

实验者: hujuan
报告时间: 2007-12-05, 下午 04:10:24
积分方法: 面积归一法



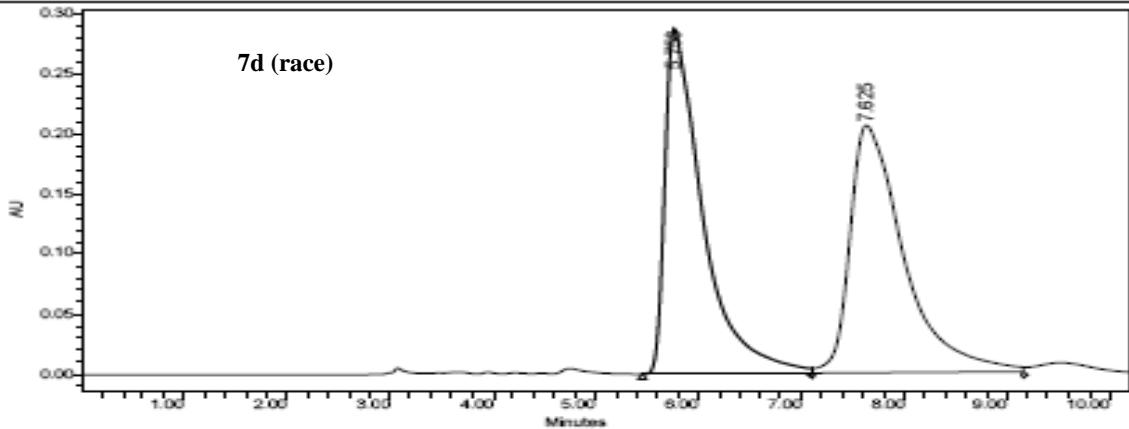
USTC

Project Name: xxf
Reported by User: System

/breeze

S A M P L E I N F O R M A T I O N

Sample Name:	xxf-30-meso-1125	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/25/2007 8:37:37 PM
Vial:	1	Acq. Method:	xxf5%
Injection #:	1	Date Processed:	11/25/2007 8:51:42 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



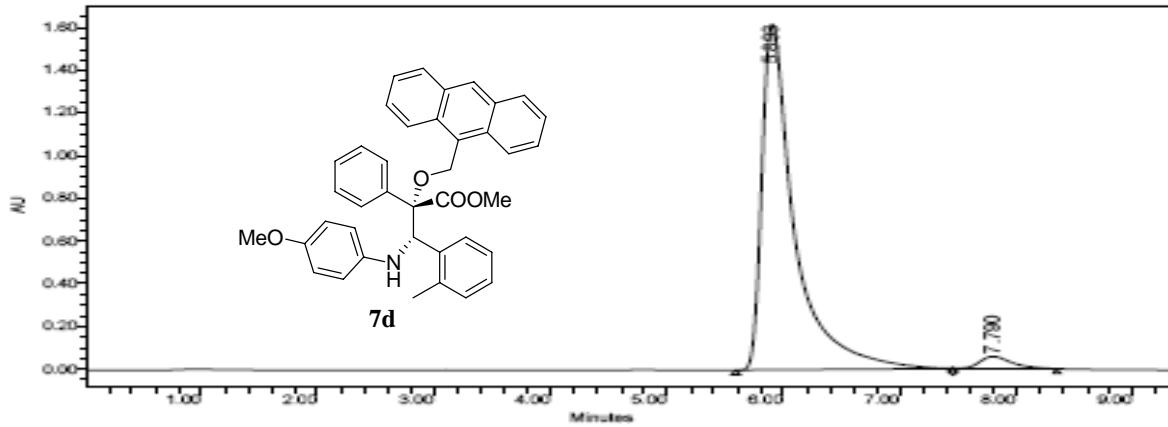
USTC

Project Name: xxf
Reported by User: System

/breeze

S A M P L E I N F O R M A T I O N

Sample Name:	xxf-30-cat-1125	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/25/2007 9:00:11 PM
Vial:	1	Acq. Method:	xxf5%
Injection #:	3	Date Processed:	11/25/2007 9:16:31 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area ($V^{\prime}sec$)	% Area	Height (V)	% Height
1	5.693	31741322	96.39	1621370	96.49
2	7.790	1169308	3.61	59052	3.51

使用仪器类型:气相色谱

检测器:FID

进样器:分流

柱温:程序升温

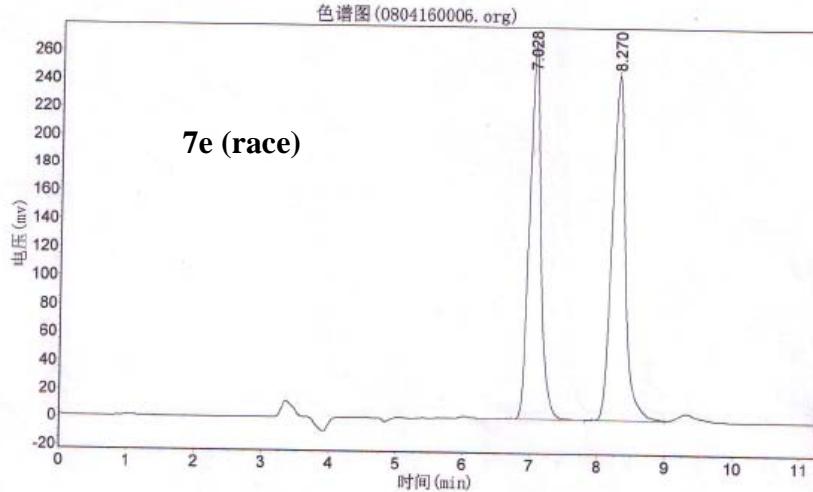
实验内容简介:

column: IA

M. P:n-Hex/i-prOH//EtOH/TFA=425:25:25:1

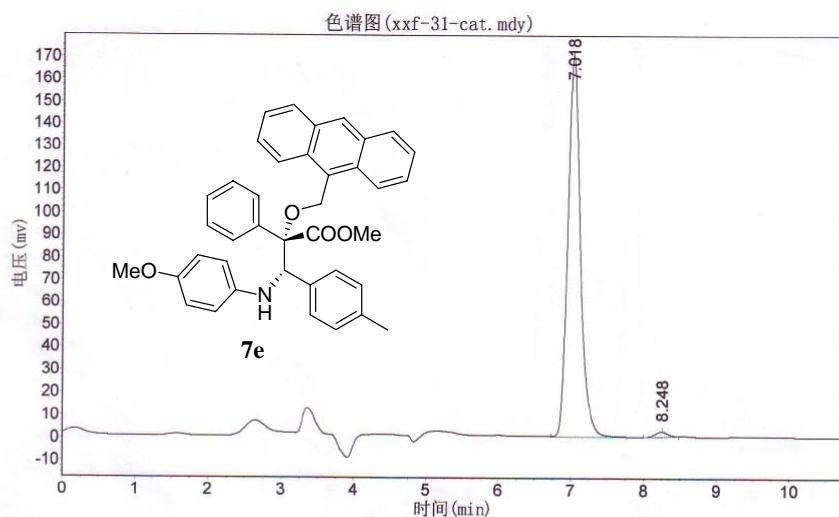
UV:254nm

0.9ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		7.028	264165.906	3132674.250	48.0808
2		8.270	244702.313	3382768.000	51.9192
总计			508868.219	6515442.250	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		7.018	169626.078	2001834.000	98.8137
2		8.248	2177.186	24032.199	1.1863
总计			171803.264	2025866.199	100.0000

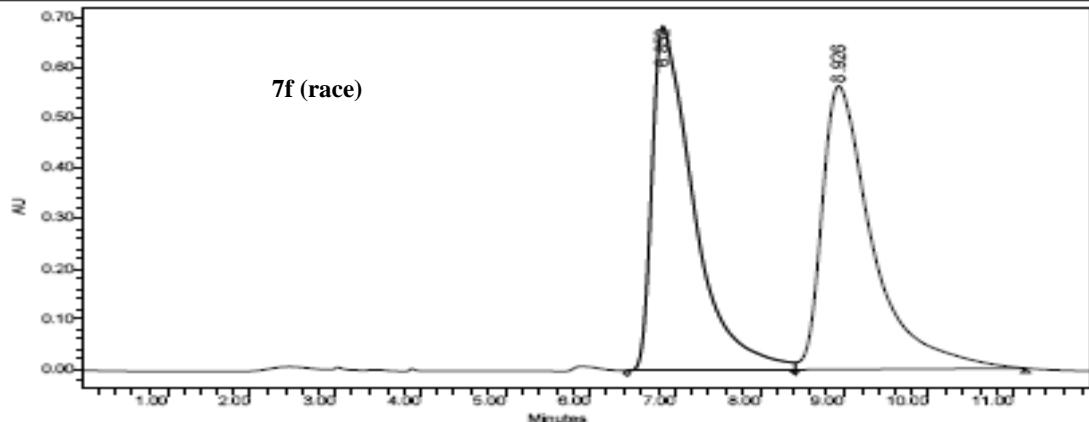
USTC

Project Name: xxr
Reported by User: System

/Breeze

SAMPLE INFORMATION

Sample Name:	xxr-33-meso-1127	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/27/2007 2:49:10 PM
Vial:	1	Aq. Method:	xxr5%
Injection #:	5	Date Processed:	11/27/2007 3:02:23 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (V ^{sec})	% Area	Height (V)	% Height
1	6.852	23305413	49.96	683603	54.82
2	8.926	23325563	50.02	563389	45.18

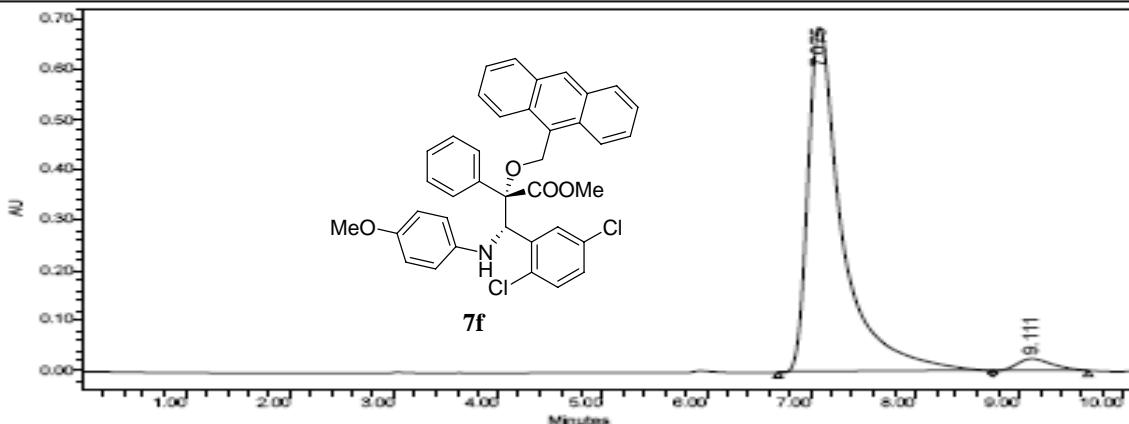
USTC

Project Name: xxr
Reported by User: System

/Breeze

SAMPLE INFORMATION

Sample Name:	xxr-33-cat-1127	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/27/2007 2:38:08 PM
Vial:	1	Aq. Method:	xxr5%
Injection #:	4	Date Processed:	11/27/2007 2:48:36 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (V ^{sec})	% Area	Height (V)	% Height
1	7.075	15876562	96.40	689212	96.58
2	9.111	592678	3.60	24397	3.42

USTC

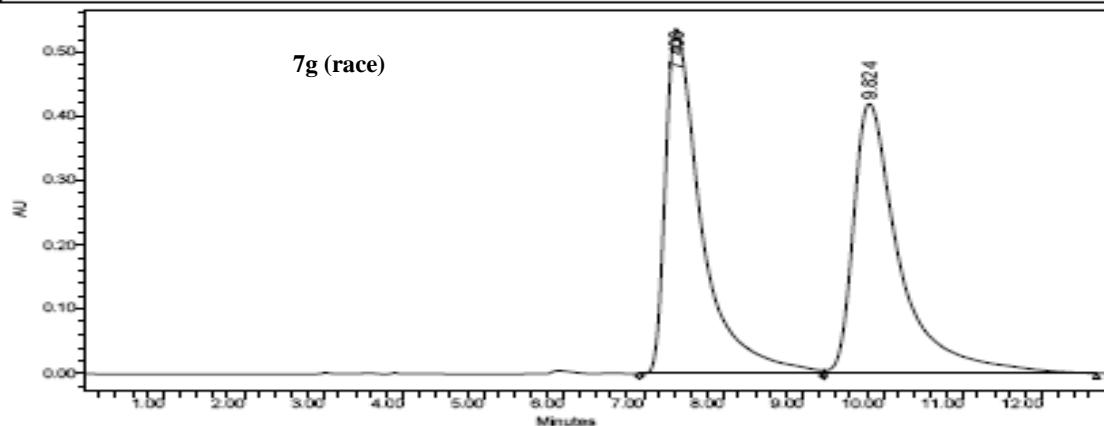
Project Name: xxf

Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xxf-34-meso-1128	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/28/2007 11:00:46 AM
Vial:	1	Acq. Method:	xxf5%
Injection #:	2	Date Processed:	11/28/2007 11:13:44 AM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



USTC

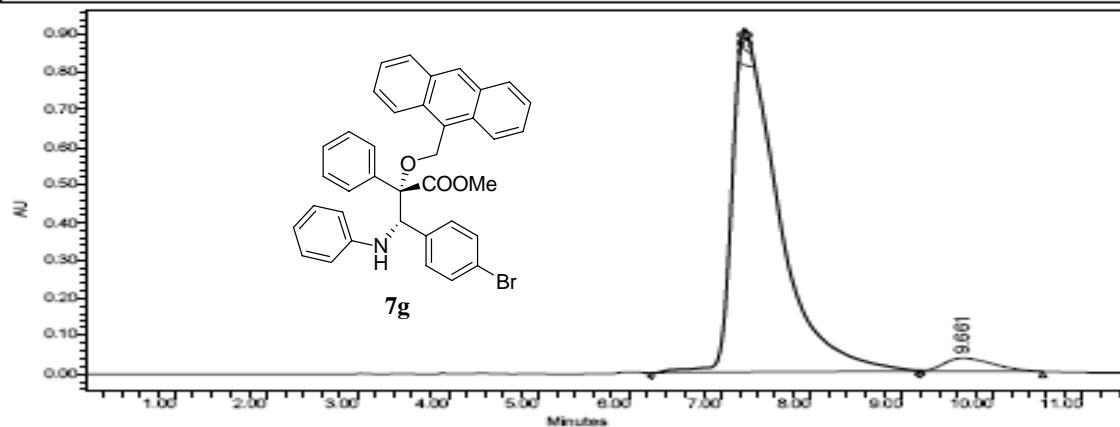
Project Name: xxf

Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xxf-34-cat-1128	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/28/2007 10:47:03 AM
Vial:	1	Acq. Method:	xxf5%
Injection #:	1	Date Processed:	11/28/2007 10:59:08 AM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



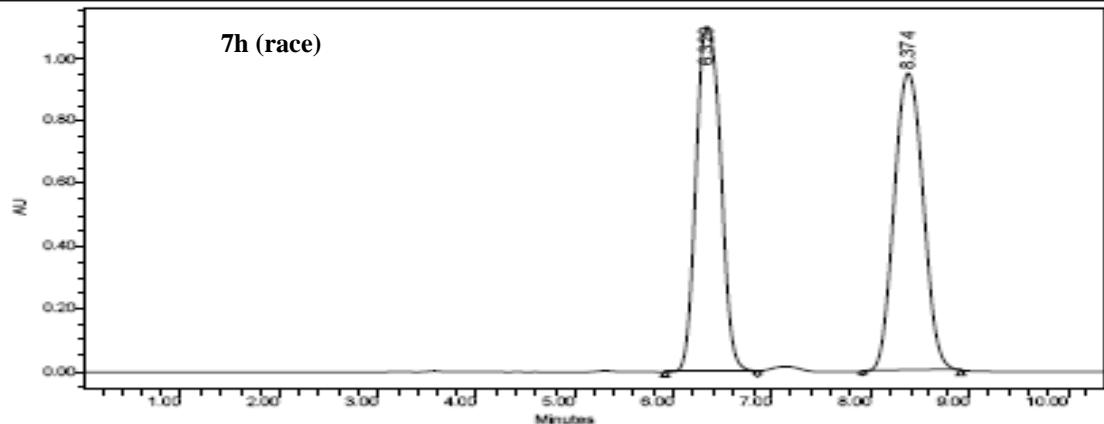
USTC

Project Name: xdf
Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xd-22-meso-1122	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/22/2007 2:48:47 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	1	Date Processed:	11/22/2007 3:00:43 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



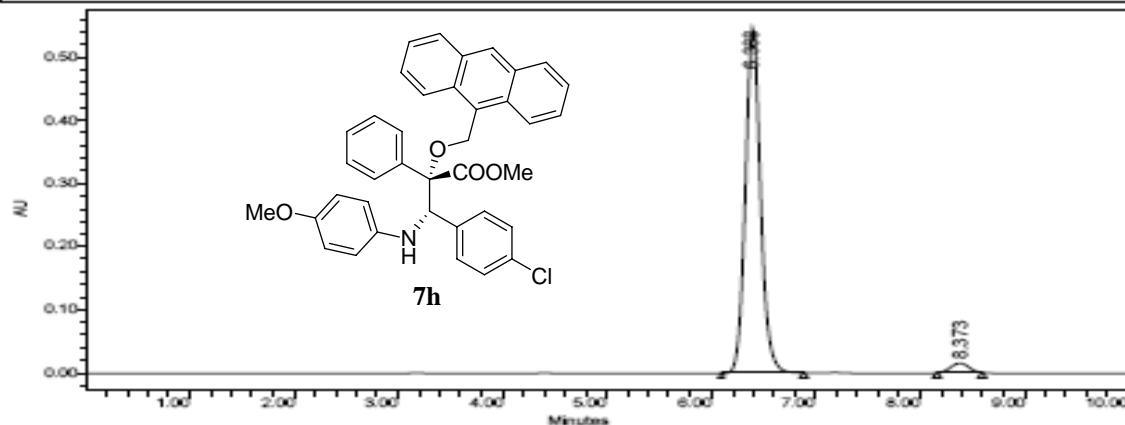
USTC

Project Name: xdf
Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xd-22-cat-1122	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/22/2007 6:19:27 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	1	Date Processed:	11/22/2007 6:43:51 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



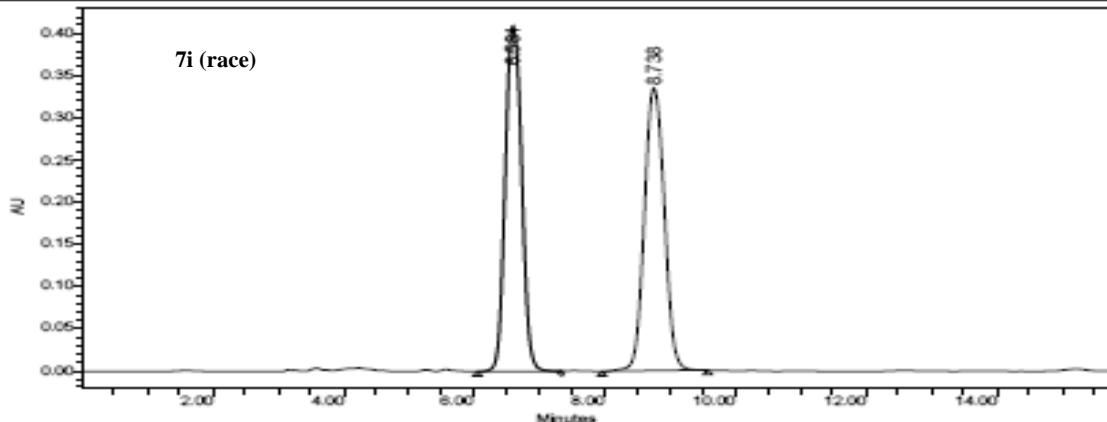
USTC

Project Name: xdf
Reported by User: System

/breeze

SAMPLE INFORMATION

Sample Name:	xdf-23-meso-1122	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/22/2007 2:59:48 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	2	Date Processed:	11/22/2007 3:16:01 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



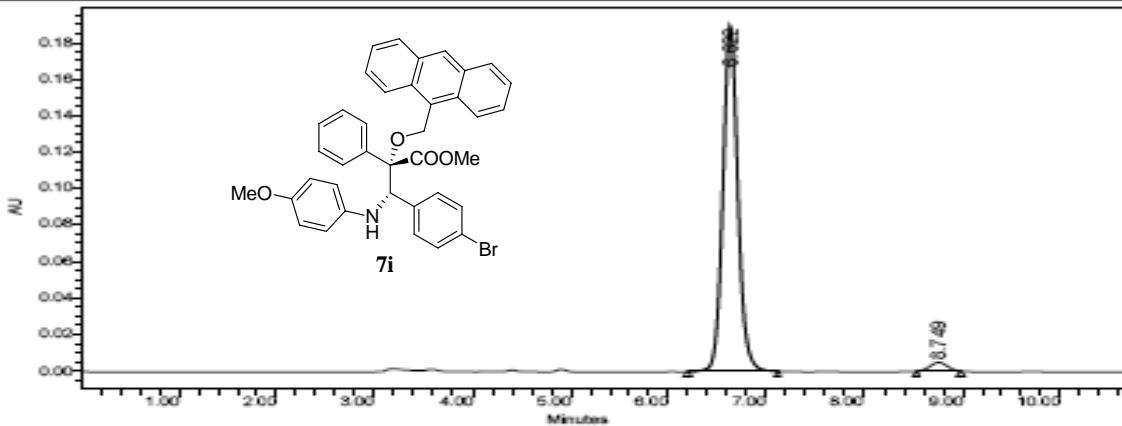
USTC

Project Name: xdf
Reported by User: System

/breeze

SAMPLE INFORMATION

Sample Name:	xdf-23-cat-1122	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/22/2007 6:31:09 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	2	Date Processed:	11/22/2007 6:42:22 PM
Injection Volume:	20.00 μ l	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.622	2015516	97.11	189790	97.52
2	8.749	60049	2.89	4836	2.48

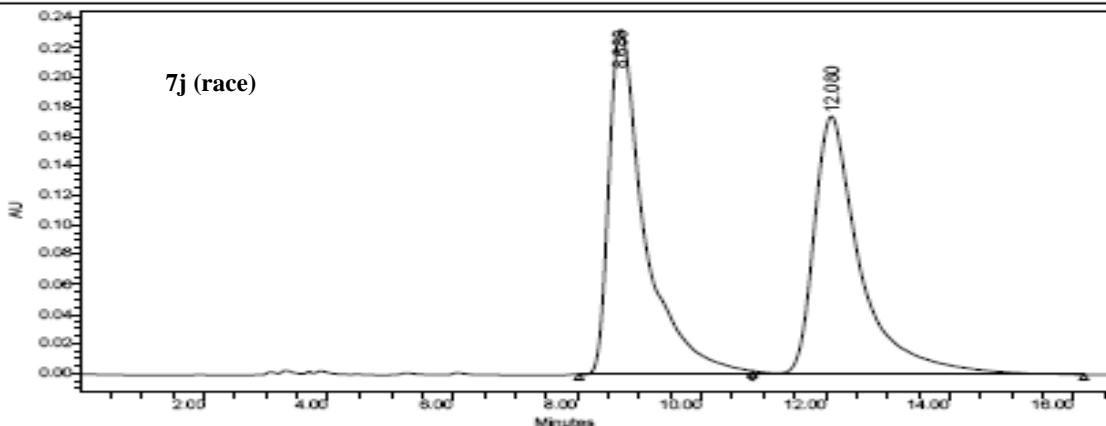
USTC

Project Name: xdf
Reported by User: System

breeze

SAMPLE INFORMATION

Sample Name:	xdf-26-meso-11241	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/24/2007 7:27:37 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	2	Date Processed:	11/24/2007 7:46:45 PM
Injection Volume:	20.00 μ l	Channel Name:	2487/Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



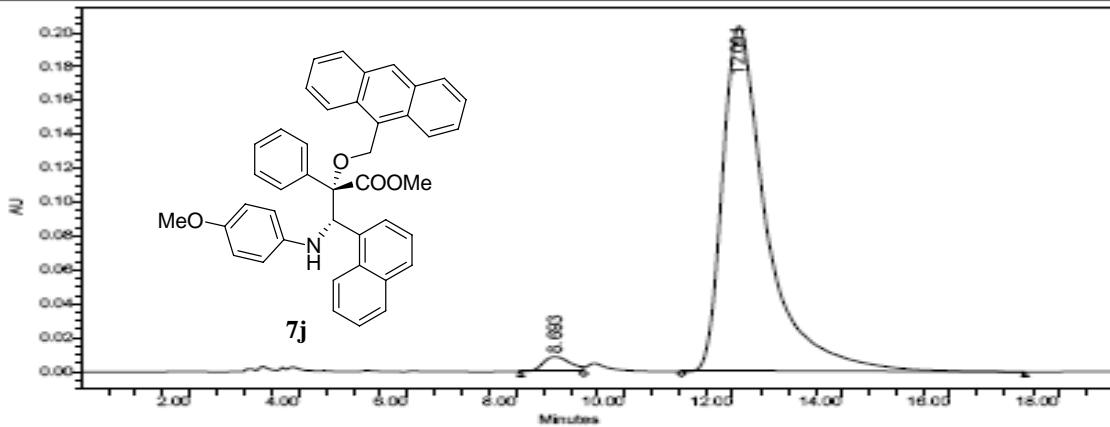
USTC

Project Name: xdf
Reported by User: System

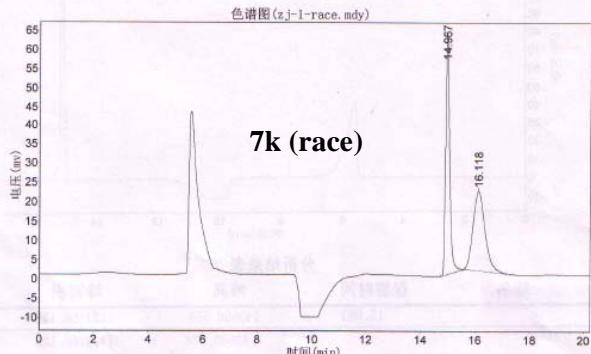
breeze

SAMPLE INFORMATION

Sample Name:	xdf-26-cat-11241	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/24/2007 8:35:21 PM
Vial:	1	Acq. Method:	XXF10%
Injection #:	5	Date Processed:	11/24/2007 8:56:13 PM
Injection Volume:	20.00 μ l	Channel Name:	2487/Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	

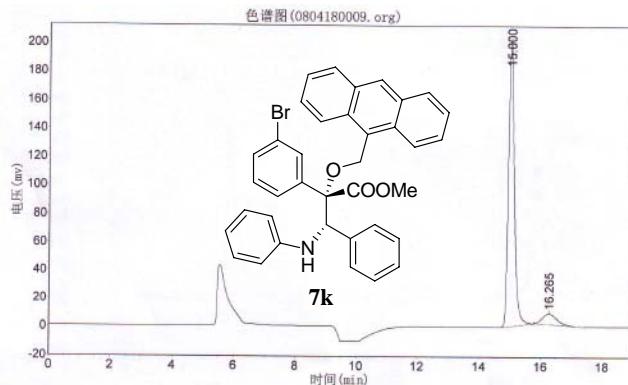


使用仪器类型:气相色谱 检测器:FID 进样器:分流
 柱温:程序升温
 载气:内标混合气
 column:OD-H
 M.P:n-Hex/i-prOH/TFA=500:5:5:1
 UV:254nm
 0.8ml/min



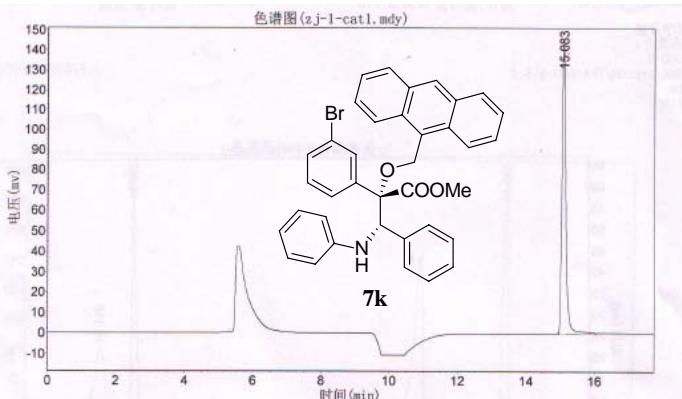
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.967	62269.543	641802.938	50.3781
2		16.118	20656.410	632169.188	49.6219
总计			82925.953	1273972.125	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.000	200917.000	2669268.250	91.9287
2		16.265	7479.230	234360.500	8.0713
总计			208396.230	2903628.750	100.0000

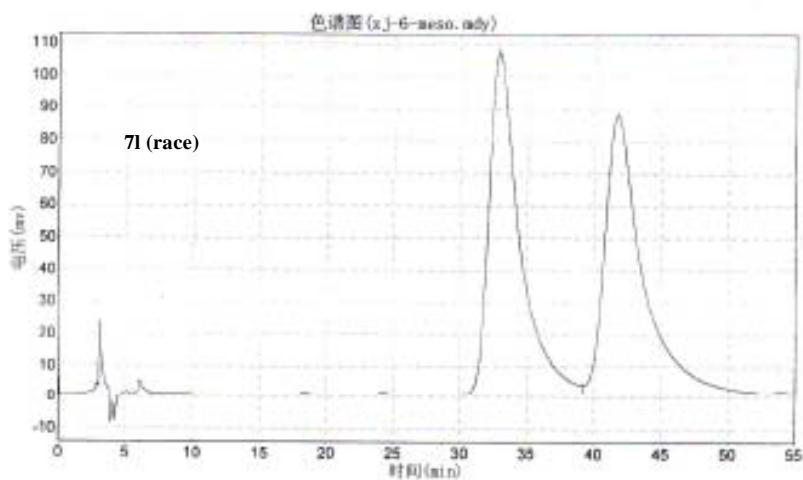


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.083	143680.578	1133168.125	100.0000
总计			143680.578	1133168.125	100.0000

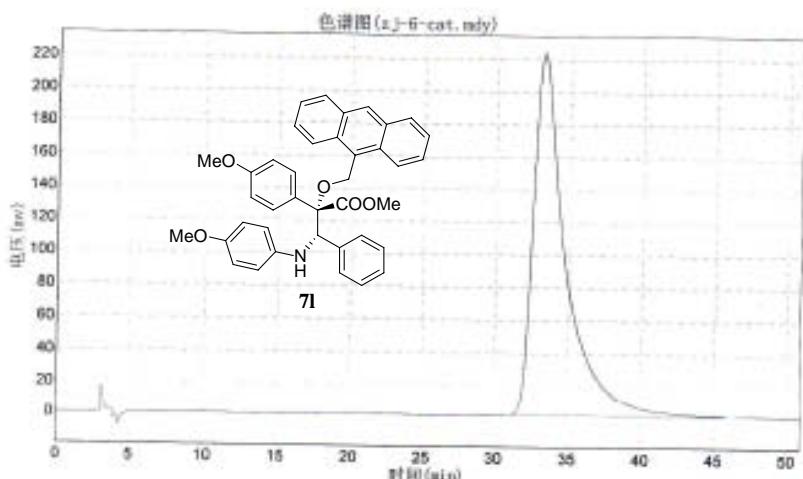
实验内容简介：

AB-H
n-Hex/i-ProH/EtOH/TFA=490:10:10:1
254nm
1ml/min



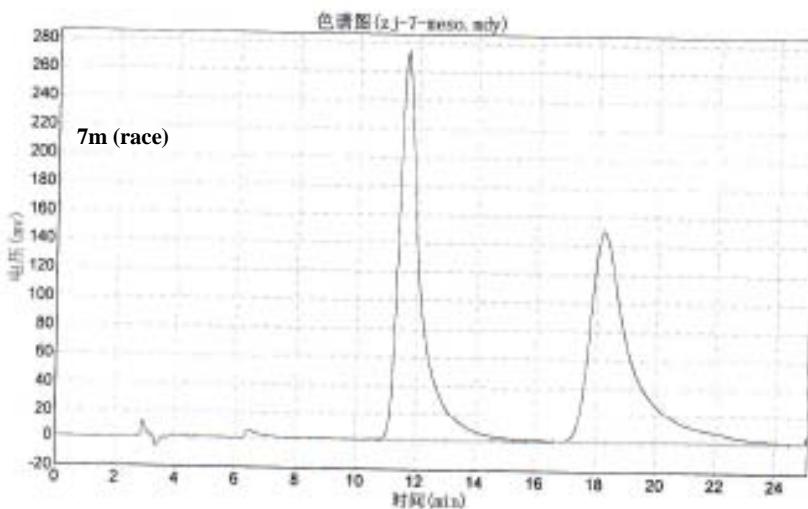
实验内容简介：

AB-H
n-Hex/i-ProH/EtOH/TFA=490:10:10:1
254nm
1ml/min



实验内容简介：

AB-H
n-Hex/i-ProH/EtOH/TFA=450:25:25:1
254nm
1ml/min

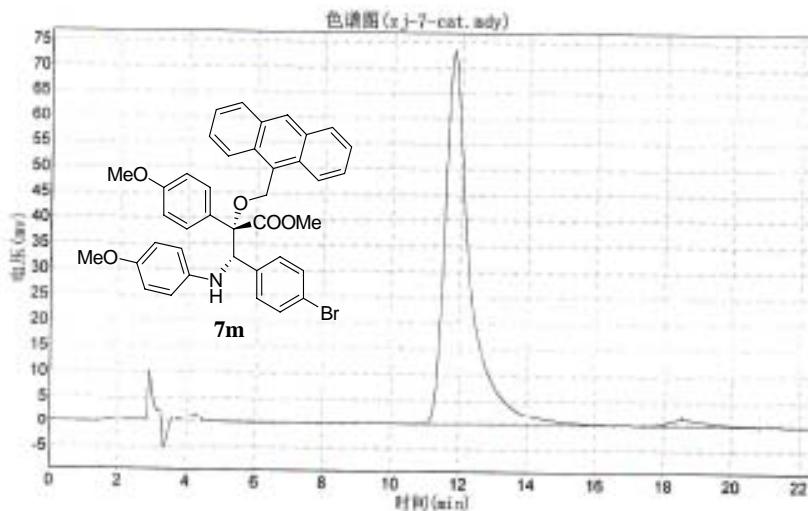


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.613	271781.031	14036522.000	49.8618
2		18.223	148205.156	14114332.000	50.1382
总计			419986.188	28150854.000	100.0000

实验内容简介：

AB-H
n-Hex/i-ProH/EtOH/TFA=450:25:25:1
254nm
1ml/min

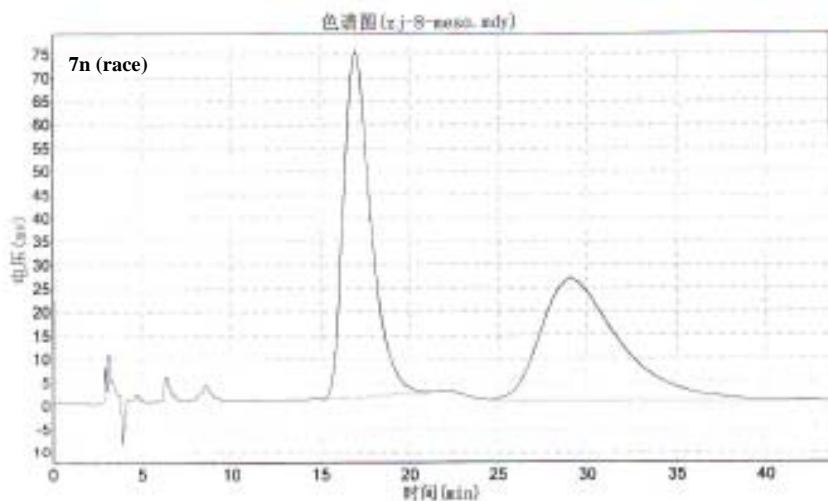


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.795	73113.727	3815851.000	97.5472
2		18.567	1206.418	95948.898	2.4528
总计			74322.145	3911799.898	100.0000

实验内容简介:

AD-H
254nm
n-Hex/i-PrOH/EtOH/TFA=490:10:10:1
1.0ml/min

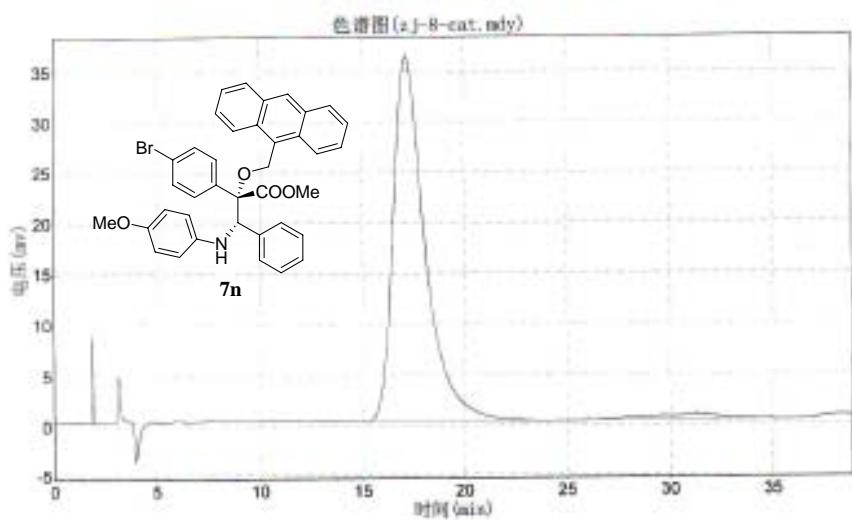


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.918	73326.914	7920455.000	50.2020
2		29.070	25583.830	7856708.500	49.7980
总计			98910.744	15777163.500	100.0000

实验内容简介:

AD-H
254nm
n-Hex/i-PrOH/EtOH/TFA=490:10:10:1
1.0ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.177	36080.602	3905776.000	97.0589
2		31.282	477.031	118436.289	2.9431
总计			36557.632	4024212.289	100.0000

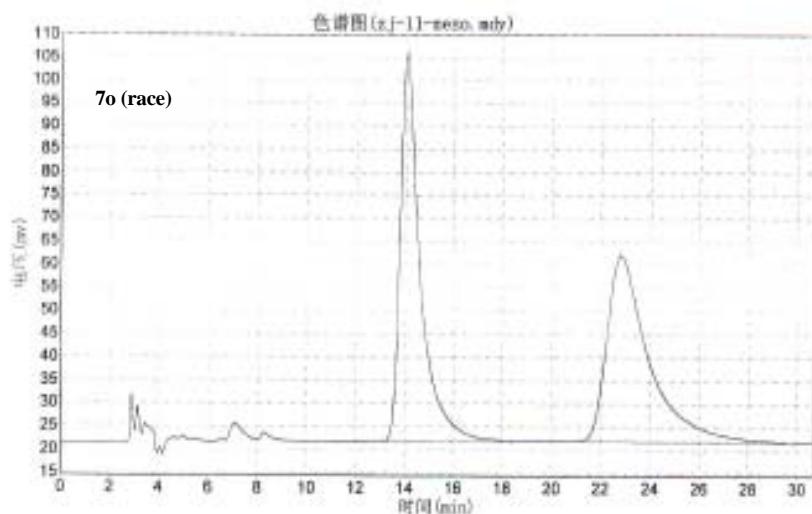
实验内容简介:

AD-H

254nm

n-Hex/1-PrOH/EtOH/TFA=490:10:10:1

1ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.078	83911.391	4781710.500	50.2077
2		22.808	40064.859	4742141.000	49.7923
总计			123976.250	9523851.500	100.0000

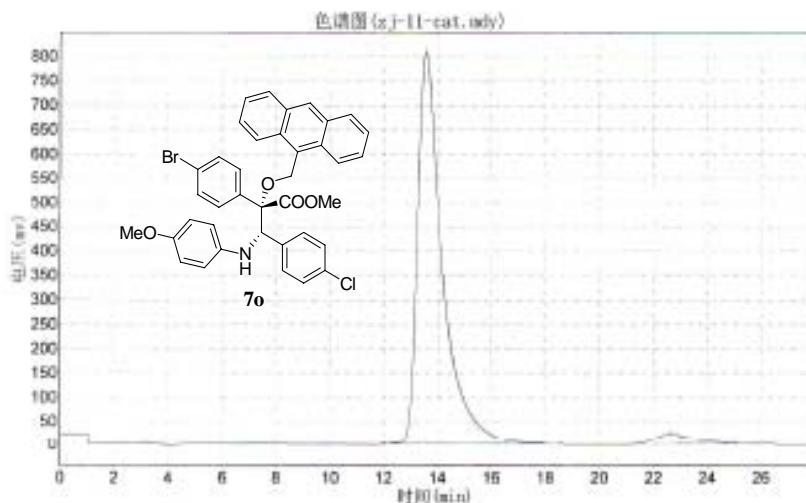
实验内容简介:

AD-H

254nm

n-Hex/1-PrOH/EtOH/TFA=490:10:10:1

1ml/min

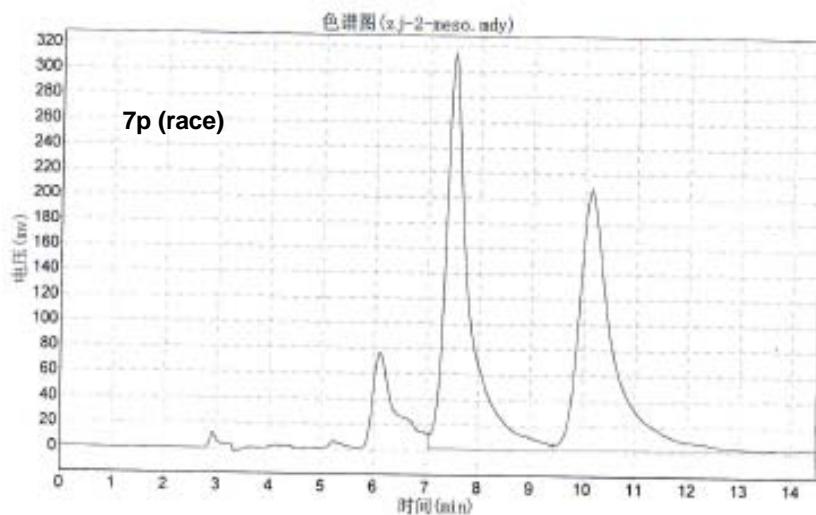


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.578	807862.625	52617212.000	95.8022
2		22.627	22079.746	2305647.500	4.1978
总计			829942.371	54922759.500	100.0000

实验内容简介：

4D-H
n-Hex/i-ProH/EtOH/TFA=450:25:25:1
254nm
1ml/min

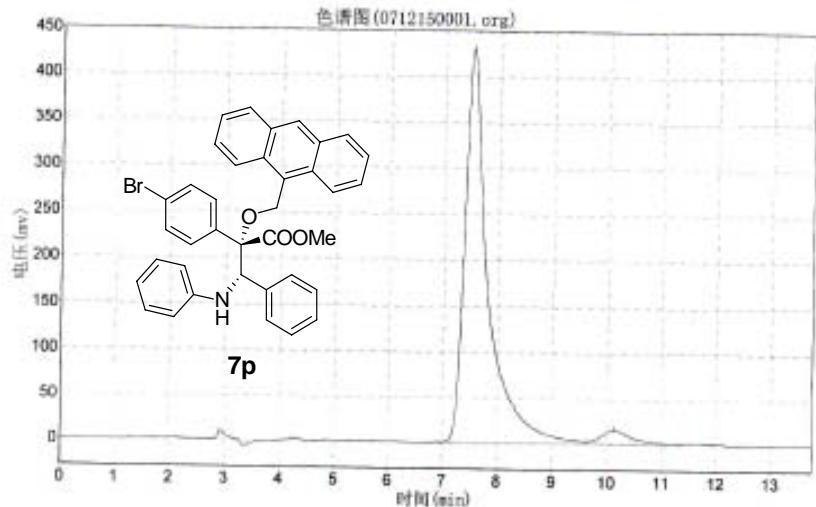


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		7.497	311201.656	9757809.000	51.5474
2		10.135	204544.000	9171963.000	48.4526
总计			515745.656	18929772.000	100.0000

实验内容简介：

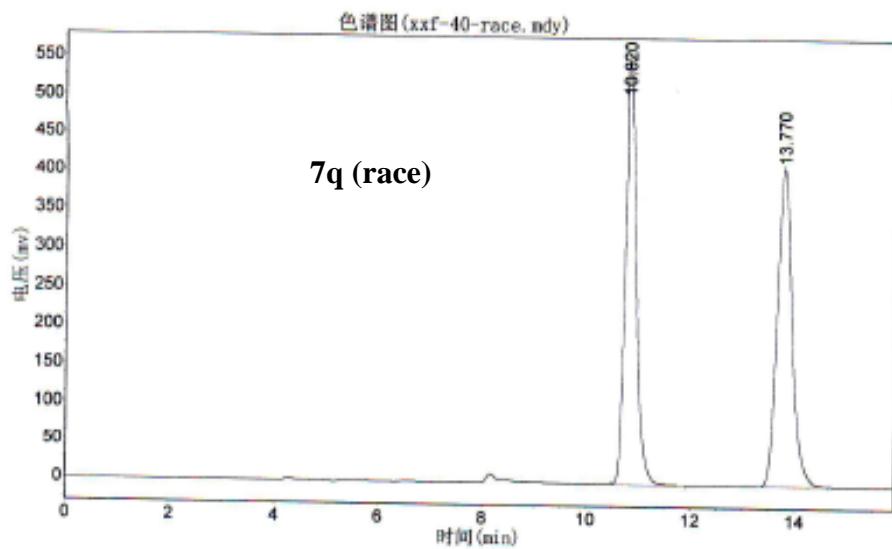
4D-H
n-Hex/i-ProH/EtOH/TFA=450:25:25:1
254nm
1ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		7.505	427785.625	12808444.000	96.1440
2		10.138	13961.692	513700.094	3.8560
总计			441747.317	13322144.094	100.0000

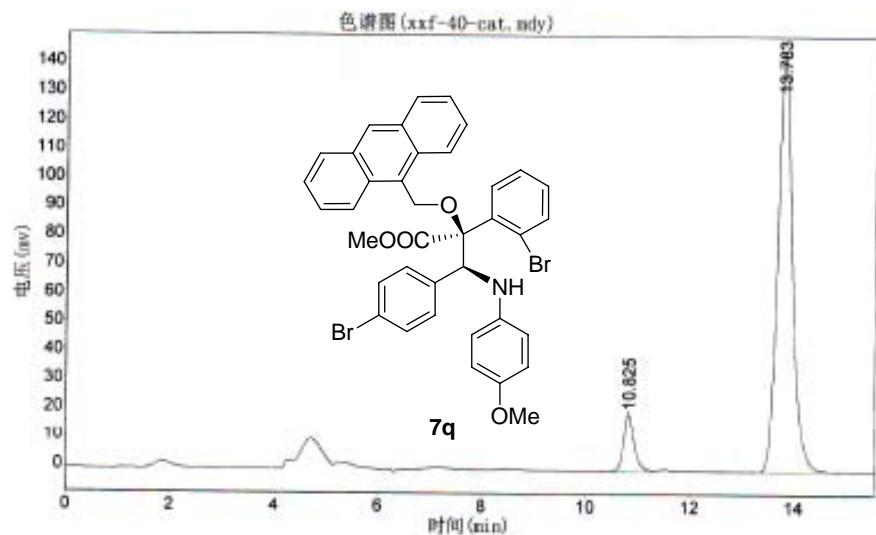
实验内容简介：
 column: IA
 M. P.: n-Hex/i-PrOH/EtOH/TFA=450:25:25:1
 UV: 254nm
 0.7ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.820	551842.875	8013048.000	49.1614
2		13.770	411011.250	8286430.000	50.8386
总计			962854.125	16299478.000	100.0000

实验内容简介：
 column: IA
 M. P.: n-Hex/i-PrOH/EtOH/TFA=450:25:25:1
 UV: 254nm
 0.7ml/min

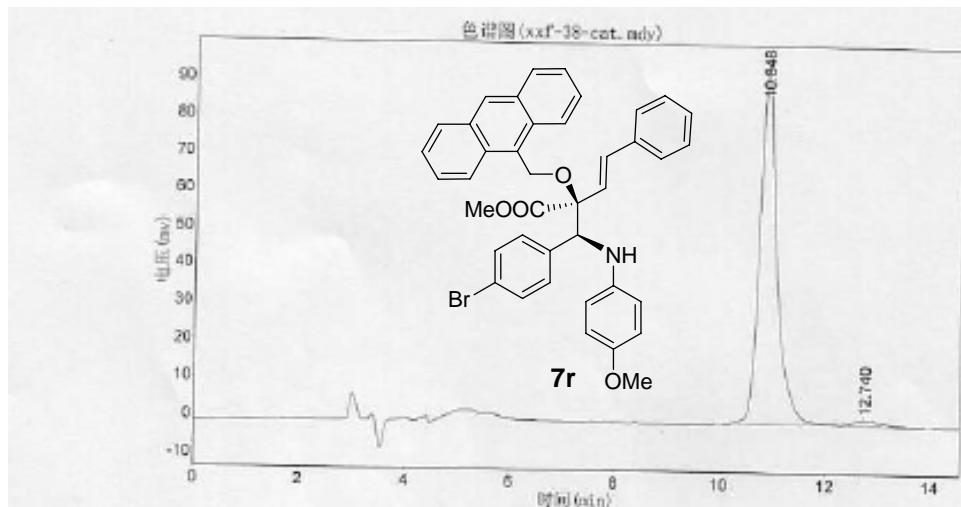
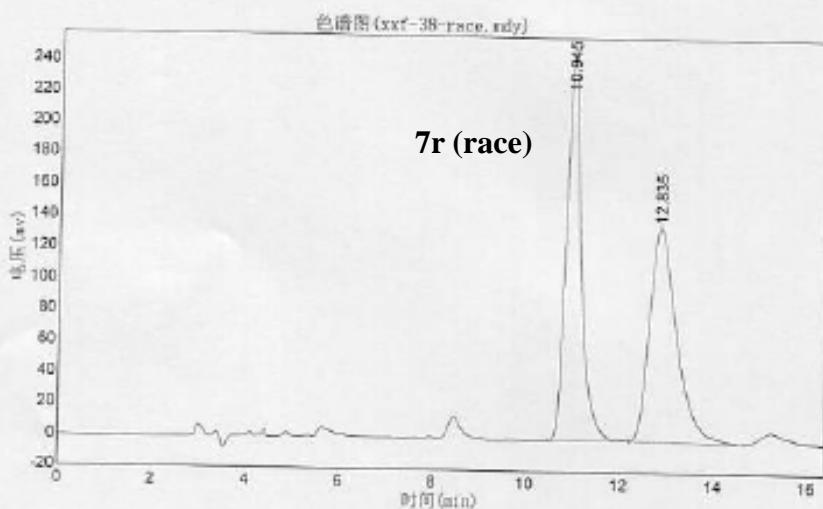


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.825	19400.287	264002.344	8.4888
2		13.783	143638.234	2846023.500	91.5112
总计			163038.521	3110025.844	100.0000

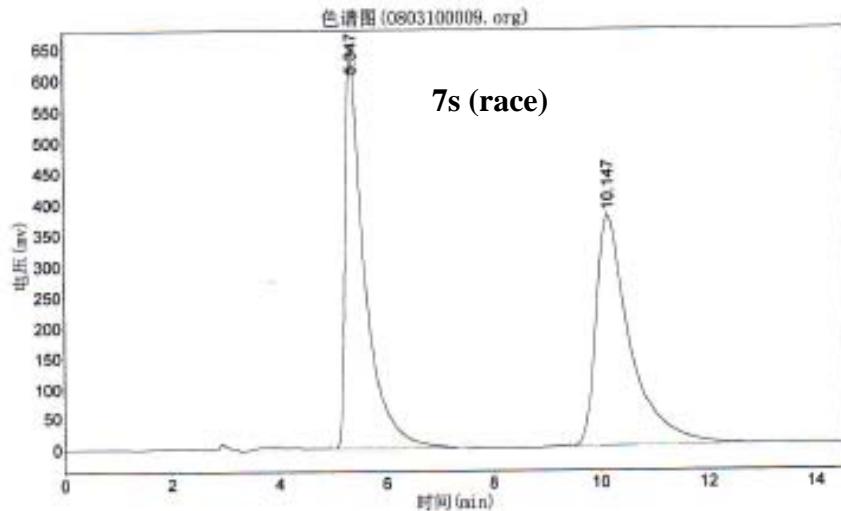
实验内容简介：

column: A
M.P.: n-Hex/i-prOH/EtOH/TPA=450:25:25:1
1.0ml/min



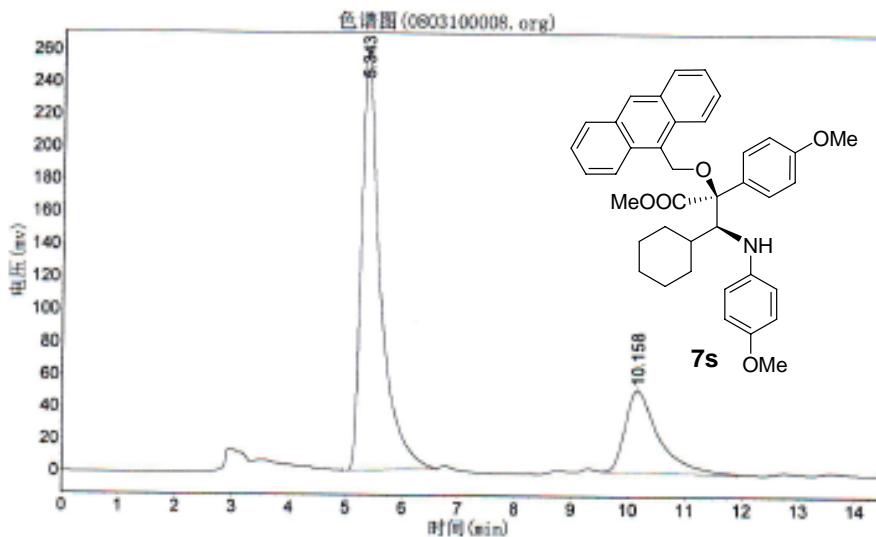
实验内容简介：

AD-H
n-Hex/EtOH/i-prOH/TFA=450:25:25:1
254nm
1ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		5.347	645175.625	16290088.000	49.8705
2		10.147	370720.030	16374709.000	50.1295
总计			1019885.656	32664797.000	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		5.343	257186.078	6044993.500	74.5550
2		10.158	50828.055	2063106.625	25.4450
总计			308014.133	8108100.125	100.0000

使用仪器类型:气相色谱

检测器:FID

进样器:分流

柱温:程序升温

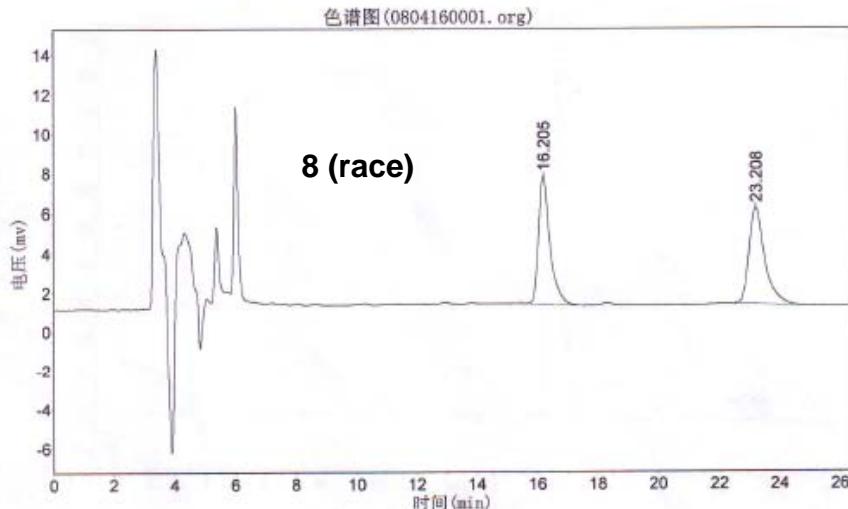
实验内容简介:

column: IA

M. P:n-Hex/i-prOH//EtOH/TFA=425:25:25:1

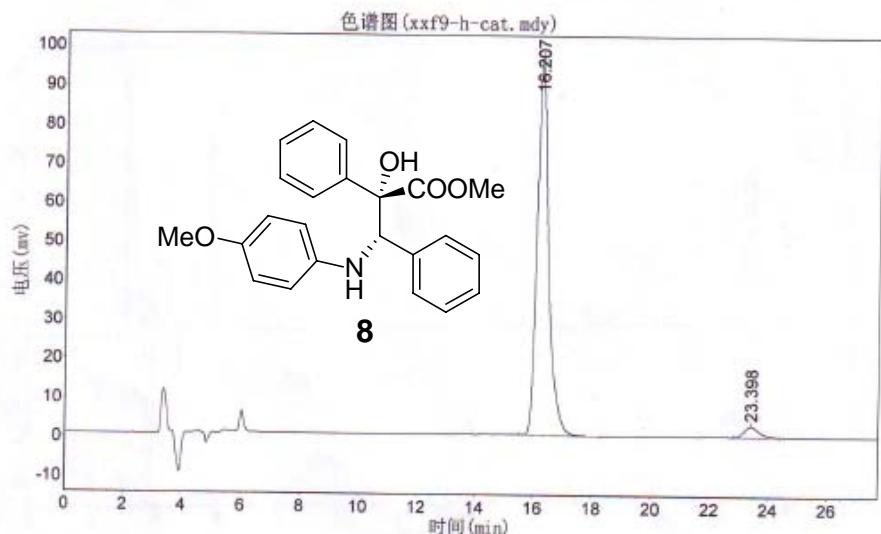
UV:254nm

0.9ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.205	6380.821	169131.500	49.2834
2		23.208	4801.113	174050.094	50.7166
总计			11181.934	343181.594	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.207	96759.195	2625662.750	96.5726
2		23.398	2668.200	93184.508	3.4274
总计			99427.395	2718847.258	100.0000