Organocatalytic Asymmetric Friedel-Crafts Alkylation/Cascade Reactions of Naphthols and Nitroolefins

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Supplementary Information

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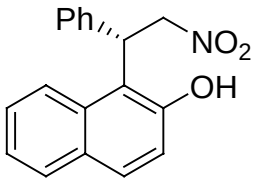
General Methods

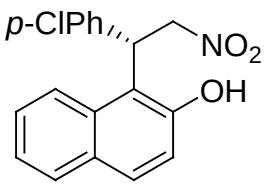
NMR spectra were recorded with tetramethylsilane as the internal standard. Column chromatography was performed using silica gel (200-300 mesh) eluting with ethyl acetate and petroleum ether. Optical rotations were measured at 589 nm at 20 °C. TLC was performed on glass-backed silica plates. Enantiomeric excess was determined by HPLC analysis on Chiralpak AS, AD and OD columns. Commercial grade solvents were dried and purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann: 1997).

General procedure for thiourea-tertiary amine catalyzed asymmetric Friedel-Crafts Reaction and domino reactions of naphthols 2 and nitroalkenes 3

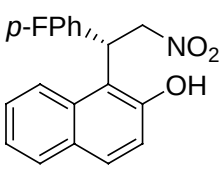
Catalyst **1a** (5.6 mg, 0.01 mmol, 10 mol %), naphthol **2** (0.10 mmol), and 4A MS (20 mg) were stirred in dry toluene (0.80 mL) and cooled to the desired temperature under argon. Then nitroalkene **3** (0.15 mmol) in dry toluene (0.2 mL) were added. After the stated reaction time, the product was purified by flash chromatography on silica gel (*previously saturated with cold petroleum ether in order to retard the unreacted starting materials*) to give the product **4** and **5**. For the F-C reaction, the reaction was generally conducted for 96 h. For the domino reactions, the reaction was extended to 144 h. The enantiomeric excess was determined by HPLC analysis on chiral column.



4aa 80% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -27.3 (c = 0.26 in $CHCl_3$); 93% *ee*, determined by HPLC analysis [Daicel Chiralcel AS, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 13.92 min, t(minor) = 16.06 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.12 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.55-7.50 (m, 1H), 7.39-7.34 (m, 3H), 7.31-7.22 (m, 3H), 6.97 (d, *J* = 8.8 Hz, 1H), 5.87 (t, *J* = 7.5 Hz, 1H), 5.47 (dd, *J* = 7.8, 13.2 Hz, 1H), 5.33 (dd, *J* = 7.1, 13.2 Hz, 1H), 5.28 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.4, 139.5, 133.0, 130.1, 129.7, 128.9, 128.8, 127.4, 127.2, 123.6, 122.4, 118.4, 117.6, 78.2, 41.0 ppm; ESI-HRMS: calcd. for $C_{18}H_{15}NO_3$+Na 316.0944, found 316.0941.

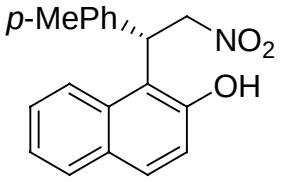


4ab 82% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -24.0 (c = 0.30 in $CHCl_3$); 94% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 23.72 min, t(major) = 30.46 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.07 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 7.9 Hz, 1H), 7.71 (d, *J* = 8.8 Hz, 1H), 7.56-7.50 (m, 1H), 7.40-7.35 (m, 1H), 7.32-7.29 (m, 2H), 7.26-7.22 (m, 2H), 6.97 (d, *J* = 8.8 Hz, 1H), 5.81 (t, *J* = 7.3 Hz, 1H), 5.46 (dd, *J* = 8.1, 13.2 Hz, 1H), 5.44 (s, 1H), 5.30 (dd, *J* = 6.7, 13.3 Hz, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.3, 138.1, 132.9, 130.3, 129.6, 129.0, 128.9, 128.8, 127.6, 123.7, 122.2, 118.3, 117.2, 78.2, 40.5 ppm; ESI-HRMS: calcd. for $C_{18}H_{14}ClNO_3$+Na 350.0554, found 350.0555.

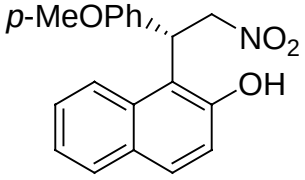


4ac 81% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = +23.9 (c = 0.28 in $CHCl_3$); 91% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 21.75 min, t(major) = 27.68 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.09 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 8.7 Hz, 1H), 7.56-7.50 (m,

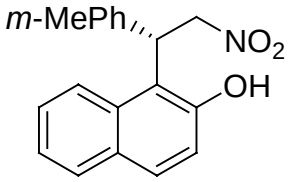
1H), 7.40-7.32 (m, 3H), 7.00-6.93 (m, 3H), 5.81 (t, *J* = 7.4 Hz, 1H), 5.46 (dd, *J* = 8.1, 13.2 Hz, 1H), 5.35-5.28 (m, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 161.8 (d, $^{1}J_{C,F}$ = 244.3 Hz), 151.2, 135.3, 132.9, 130.2, 129.7, 129.2, 129.0 (d, $^{3}J_{C,F}$ = 8.0 Hz), 127.6, 123.7, 122.3, 118.3, 117.4, 115.6 (d, $^{2}J_{C,F}$ = 21.2 Hz), 78.5, 40.6 ppm; ESI-HRMS: calcd. for $C_{18}H_{14}FNO_3$+Na 334.0850, found 334.0834.



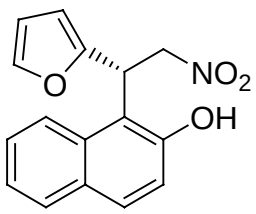
4ad 69% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -14.4 (c = 0.26 in $CHCl_3$); 85% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 15.40 min, t(major) = 21.53 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 8.7 Hz, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 8.8 Hz, 1H), 7.55-7.50 (m, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.1 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 1H), 5.84 (t, *J* = 7.5 Hz, 1H), 5.45 (dd, *J* = 8.0, 13.2 Hz, 1H), 5.31 (dd, *J* = 7.0, 13.2 Hz, 1H), 5.25 (s, 1H), 2.29 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.4, 136.9, 136.3, 133.0, 129.9, 129.5, 128.9, 127.4, 127.2, 123.6, 122.4, 118.5, 117.7, 78.2, 40.7, 21.0 ppm; ESI-HRMS: calcd. for $C_{19}H_{17}NO_3$+Na 330.1101, found 330.1095.



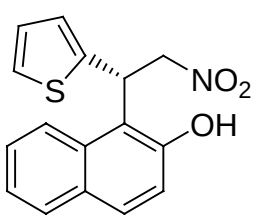
4ae 74% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -22.4 (c = 0.24 in $CHCl_3$); 85% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 25.02 min, t(major) = 30.06 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.12 (d, *J* = 8.6 Hz, 1H), 7.78 (dd, *J* = 1.1, 8.1 Hz, 1H), 7.69 (d, *J* = 8.8 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.31-7.28 (m, 2H), 6.96 (d, *J* = 8.8 Hz, 1H), 6.83-6.80 (m, 2H), 5.80 (t, *J* = 7.5 Hz, 1H), 5.45 (dd, *J* = 8.1, 13.2 Hz, 1H), 5.33-5.28 (m, 2H), 3.75 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.4, 133.0, 131.3, 130.0, 129.7, 129.1, 129.0, 128.5, 127.4, 123.6, 123.0, 122.4, 118.5, 117.7, 114.2, 78.4, 55.2, 40.5 ppm; ESI-HRMS: calcd. for $C_{19}H_{17}NO_4$+Na 346.1050, found 346.1051.



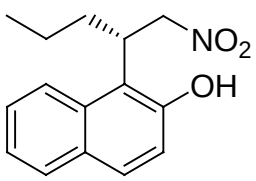
4af 72% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -14.1 (c = 0.18 in $CHCl_3$); 91% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 14.77 min, t(major) = 18.63 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.14 (d, *J* = 8.6 Hz, 1H), 7.79 (dd, *J* = 0.83, 8.1 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.58-7.51 (m, 1H), 7.40-7.35 (m, 1H), 7.20-7.18 (m, 3H), 7.06-7.05 (m, 1H), 6.94 (d, *J* = 8.8 Hz, 1H), 5.84 (t, *J* = 7.5 Hz, 1H), 5.45 (dd, *J* = 7.8, 13.2 Hz, 1H), 5.36-5.29 (m, 2H), 2.29 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.5, 139.3, 138.5, 133.0, 129.9, 129.6, 128.9, 128.7, 128.1, 128.0, 127.4, 124.3, 123.6, 122.4, 118.5, 117.5, 78.2, 40.9, 21.5 ppm; ESI-HRMS: calcd. for $C_{19}H_{17}NO_3$+Na 330.1101, found 330.1109.



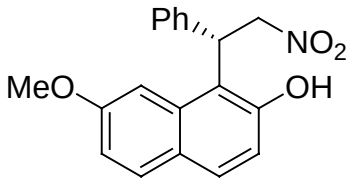
4ag 77% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -17.5 (c = 0.32 in $CHCl_3$); 90% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 14.57 min, t(major) = 25.75 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.05 (d, *J* = 8.7 Hz, 1H), 7.80 (dd, *J* = 0.76, 8.1 Hz, 1H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.59-7.50 (m, 1H), 7.41-7.34 (m, 2H), 7.00 (d, *J* = 8.8 Hz, 1H), 6.31-6.30 (m, 1H), 6.11 (d, *J* = 3.3 Hz, 1H), 5.98 (t, *J* = 7.2 Hz, 1H), 5.58 (s, 1H), 5.47 (dd, *J* = 8.8, 13.3 Hz, 1H), 5.13 (dd, *J* = 6.7, 13.3 Hz, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 152.1, 151.9, 142.0, 132.7, 130.4, 129.6, 129.0, 127.4, 123.7, 122.2, 118.5, 114.8, 110.6, 106.9, 76.2, 35.4 ppm; ESI-HRMS: .calcd. for $C_{16}H_{13}NO_4$+Na 306.0737, found 306.0730.



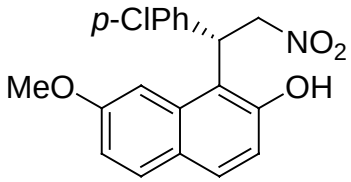
4ah 79% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = +17.5 (c = 0.35 in $CHCl_3$); 94% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 19.11 min, t(major) = 24.79 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.11 (d, *J* = 8.7 Hz, 1H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.71 (d, *J* = 8.8 Hz, 1H), 7.58-7.50 (m, 1H), 7.43-7.37 (m, 1H), 7.17 (dd, *J* = 1.2, 5.1 Hz, 1H), 7.02-6.97 (m, 2H), 6.93-6.90 (m, 1H), 6.09 (t, *J* = 7.2 Hz, 1H), 5.55 (s, 1H), 5.48 (dd, *J* = 7.8, 13.3 Hz, 1H), 5.32 (dd, *J* = 6.7, 13.3 Hz, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.6, 142.6, 132.6, 130.4, 129.6, 129.0, 127.5, 126.8, 125.0, 124.8, 123.7, 122.1, 118.3, 117.0, 78.7, 37.0 ppm; ESI-HRMS: .calcd. for $C_{16}H_{13}NO_3S$+Na 322.0508, found 322.0508.



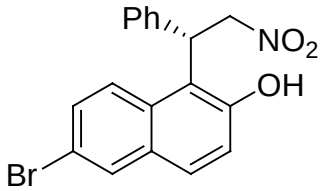
4ai 69% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -5.7 (c = 0.60 in $CHCl_3$); 94% *ee*, determined by HPLC analysis [Daicel Chiralcel AS, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 7.25 min, t(minor) = 9.27 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 8.9 Hz, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 1H), 6.93 (d, *J* = 8.7 Hz, 1H), 5.26 (s, 1H), 5.09-5.02 (m, 1H), 4.99-4.93 (m, 1H), 4.53-4.46 (m, 1H), 2.23-2.07 (m, 1H), 1.84-1.69 (m, 1H), 1.34-1.09 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 151.4, 133.8, 129.4, 128.8, 127.1, 123.4, 122.5, 118.1, 117.7, 78.6, 36.5, 33.0, 20.9, 14.1 ppm; ESI-HRMS: .calcd. for $C_{15}H_{17}NO_3$+Na 282.1101, found 282.1090.



4ba 81% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 13:1); $[\alpha]_D^{20}$ = -47.9 (c = 0.24 in $CHCl_3$); 91% *ee*, determined by HPLC analysis [Daicel Chiralcel AS, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 16.48 min, t(major) = 18.21 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.66 (d, *J* = 8.9 Hz, 1H), 7.61 (d, *J* = 8.7 Hz, 1H), 7.38-7.24 (m, 6H), 7.01 (dd, *J* = 2.4, 8.9 Hz, 1H), 6.82 (d, *J* = 8.7 Hz, 1H), 5.76 (t, *J* = 7.3 Hz, 1H), 5.46 (dd, *J* = 7.9, 13.2 Hz, 1H), 5.30 (dd, *J* = 6.9, 13.2 Hz, 1H), 5.23 (br.s, 1H), H), 3.87 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 158.9, 152.0, 139.7, 134.4, 130.4, 129.8, 128.8, 127.4, 127.2, 125.0, 116.7, 115.8, 115.7, 102.0, 78.3, 55.2, 41.3 ppm; ESI-HRMS: .calcd. for $C_{19}H_{17}NO_4$+Na 346.1050, found 346.1055.

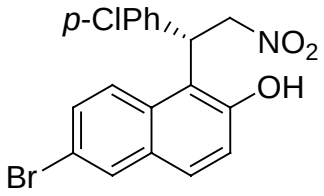


4bb 83% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 12:1); $[\alpha]_D^{20}$ = -51.0 (c = 0.20 in $CHCl_3$); 95% *ee*, determined by HPLC analysis [Daicel Chiralcel AS, *n*-hexane/*i*-PrOH = 92/8, 1.0 mL/min, λ= 254 nm, t(minor) = 20.71 min, t(major) = 24.11 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 8.9 Hz, 1H), 7.62 (d, *J* = 8.7 Hz, 1H), 7.31-7.23 (m, 5H), 7.03 (dd, *J* = 2.4, 8.9 Hz, 1H), 6.82 (d, *J* = 8.7 Hz, 1H), 5.69 (t, *J* = 7.3 Hz, 1H), 5.45 (dd, *J* = 8.1, 13.3 Hz, 1H), 5.30-5.23 (m, 2H), 3.89 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 159.0, 151.9, 138.3, 130.5, 130.0, 129.2, 129.1, 128.8, 125.0, 116.3, 115.7, 115.6, 101.9, 78.3, 55.3, 40.8 ppm; ESI-HRMS: .calcd. for $C_{19}H_{16}ClNO_4$+H 358.0841, found 358.0844.

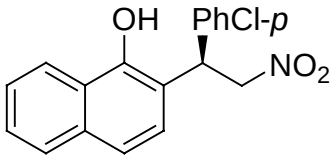


4ca 72% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -41.3 (c = 0.21 in $CHCl_3$); 90% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 11.40 min, t(major) = 13.53 min]; ^{1}H NMR (300 MHz, DMSO): δ = 10.31 (s, 1H), 8.07-8.04 (m, 2H), 7.74 (d, *J* = 8.9 Hz, 1H), 7.55 (dd, *J* = 2.1, 9.2 Hz, 1H), 7.37 (d, *J* = 7.2 Hz, 2H),

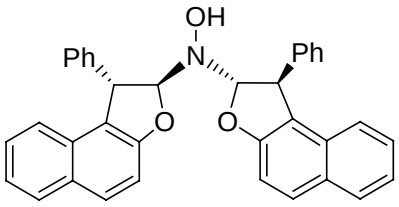
7.30-7.26 (m, 2H), 7.24-7.16 (m, 2H), 5.71-5.64 (m, 1H), 5.60-5.51 (m, 2H) ppm; ^{13}C NMR (75 MHz, DMSO): δ = 153.8, 139.9, 131.7, 130.3, 129.8, 129.5, 128.6, 128.4, 127.5, 126.7, 124.8, 120.0, 117.0, 115.4, 77.9, 59.7 ppm; ESI-HRMS: calcd. for $C_{18}H_{14}BrNO_3$-H 370.0084, found 370.0090.



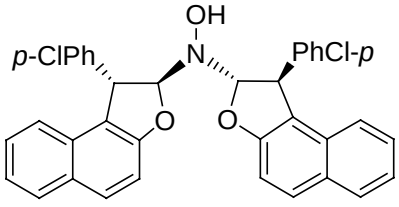
4cb 77% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -43.7 (c = 0.26 in $CHCl_3$); 90% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 11.73 min, t(major) = 14.65 min]; ^{1}H NMR (300 MHz, DMSO): δ = 10.36 (s, 1H), 8.08-8.06 (m, 2H), 7.75 (d, *J* = 8.9 Hz, 1H), 7.56 (dd, *J* = 2.0, 9.2 Hz, 1H), 7.44-7.28 (m, 4H), 7.22 (d, *J* = 8.9 Hz, 1H), 5.71-5.63 (m, 1H), 5.58-5.56 (m, 2H) ppm; ^{13}C NMR (75 MHz, DMSO): δ = 153.8, 138.9, 131.6, 130.3, 129.7, 129.6, 129.4, 128.8, 128.4, 124.8, 119.9, 116.6, 115.5, 77.5, 59.7 ppm; ESI-HRMS: calcd. for $C_{18}H_{13}BrClNO_3$-H 403.9695, found 403.9690.



4db 67% yield (catalyzed by **1b** derived from quinine); yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = +33.9 (c = 0.26 in $CHCl_3$); 80% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 70/30, 1.0 mL/min, λ= 254 nm, t(major) = 9.39 min, t(minor) = 25.47 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.94-7.91 (m, 1H), 7.82-7.79 (m, 1H), 7.55-7.49 (m, 2H), 7.46 (d, *J* = 8.2 Hz, 1H), 7.32-7.25 (m, 4H), 7.19 (d, *J* = 8.6 Hz, 1H), 5.69 (br.s, 1H), 5.43 (t, *J* = 8.1 Hz, 1H), 5.18-5.08 (m, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.3, 137.4, 133.8, 133.5, 129.2, 128.3, 126.5, 126.2, 125.3, 124.5, 121.7, 119.9, 119.7, 77.9, 42.7 ppm; ESI-HRMS: calcd. for $C_{18}H_{14}ClNO_3$+Na 350.0554, found 350.0560.

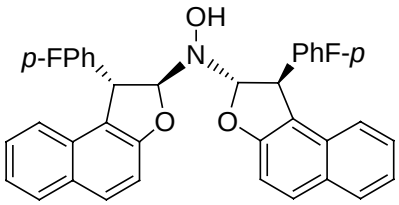


5aa 64% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +301.0 (c = 0.30 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 6.01 min, t(minor) = 10.33 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.85-7.80 (m, 4H), 7.36-7.27 (m, 18H), 5.73 (d, *J* = 4.3 Hz, 2H), 5.29 (d, *J* = 4.2 Hz, 2H), 5.13 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.9, 142.0, 130.2, 130.0, 129.8, 128.9, 128.8, 128.1, 127.2, 126.7, 123.0, 122.9, 120.3, 111.7, 104.0, 51.0 ppm; ESI-HRMS: calcd. for $C_{36}H_{27}NO_3$+Na 544.1883, found 544.1888.

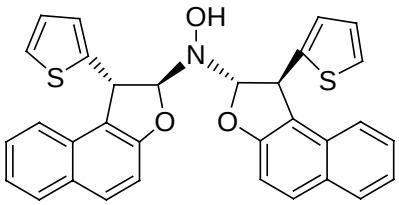


5ab 63% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +292.4 (c = 0.35 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 6.60 min, t(minor) = 11.82 min]; ^{1}H NMR (300 MHz, DMSO): δ = 8.86 (s, 1H), 7.87 (d, *J* = 8.6 Hz, 4H), 7.42-7.34 (m, 6H), 7.31-7.23 (m, 10H), 5.79 (d, *J* = 4.0 Hz, 2H), 5.25 (d, *J* = 3.7 Hz, 2H) ppm; ^{13}C NMR (75 MHz, DMSO): δ = 156.8, 141.4, 131.5, 129.9, 129.8, 129.5, 129.2, 128.9, 128.8, 126.9, 122.8, 122.2, 120.5, 111.8, 103.7, 49.0 ppm; ESI-HRMS: calcd. for $C_{36}H_{25}Cl_2NO_3$+Na 612.1104, found 612.1095.

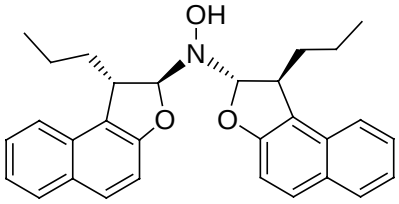
5ac 57% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +317.9 (c = 0.24 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH =



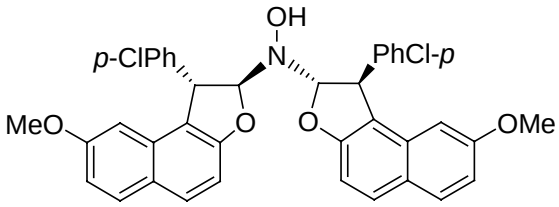
90/10, 1.0 mL/min, λ= 254 nm, t(major) = 6.32 min, t(minor) = 11.70 min]; ^{1}H NMR (300 MHz, DMSO): δ = 8.85 (s, 1H), 7.86 (d, *J* = 8.9 Hz, 4H), 7.38-7.13 (m, 16H), 5.78 (d, *J* = 4.0 Hz, 2H), 5.25 (d, *J* = 3.9 Hz, 2H) ppm; ^{13}C NMR (75 MHz, DMSO): δ = 161.2 (d, $^1J_{C,F}$ = 241.5 Hz), 156.7, 138.6, 130.0, 129.8 (d, $^3J_{C,F}$ = 5.2 Hz), 129.6, 129.2, 128.9, 126.8, 122.8, 122.3, 120.8, 115.6 (d, $^2J_{C,F}$ = 21.2 Hz), 111.8, 103.8, 49.0 ppm; ESI-HRMS: calcd. for $C_{36}H_{25}F_2NO_3$+Na 580.1695, found 580.1621.



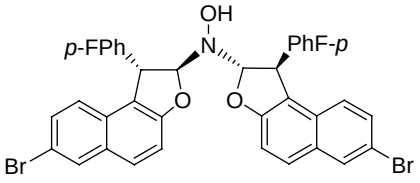
5ah 61% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +283.4 (c = 0.34 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 6.19 min, t(minor) = 8.40 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.82-7.78 (m, 4H), 7.48 (d, *J* = 7.9 Hz, 2H), 7.36-7.25 (m, 6H), 7.20 (t, *J* = 3.1 Hz, 2H), 6.95 (d, *J* = 3.4 Hz, 4H), 5.75 (d, *J* = 4.2 Hz, 2H), 5.59 (d, *J* = 4.2 Hz, 2H), 5.05 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.6, 145.2, 130.4, 130.2, 129.8, 128.8, 127.1, 126.8, 125.5, 124.9, 123.2, 122.9, 119.7, 111.7, 103.9, 45.9 ppm; ESI-HRMS: calcd. for $C_{32}H_{23}NO_3S_2$+Na 556.1012, found 556.1004.



5ai 52% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +285.5 (c = 0.40 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 4.85 min, t(minor) = 6.21 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.81 (d, *J* = 8.1 Hz, 2H), 7.76-7.68 (m, 4H), 7.48-7.43 (m, 2H), 7.32-7.27 (m, 2H), 7.17 (d, *J* = 8.8 Hz, 2H), 5.61 (d, *J* = 2.9 Hz, 2H), 4.87 (s, 1H), 4.11-3.94 (m, 2H), 2.10-1.96 (m, 2H), 1.82-1.75 (m, 2H), 1.55-1.40 (m, 4H), 0.96 (t, *J* = 7.4 Hz, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.3, 130.2, 129.7, 129.2, 129.0, 126.6, 122.8, 122.4, 121.1, 111.8, 102.7, 100.0, 44.8, 35.6, 19.6, 14.1 ppm; ESI-HRMS: calcd. for $C_{30}H_{31}NO_3$+Na 476.2196, found 476.2201.

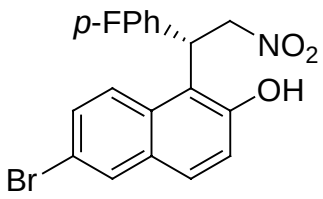


5bb 67% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +63.2 (c = 0.22 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(major) = 9.09 min, t(minor) = 19.00 min]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.72-7.68 (m, 4H), 7.29 (d, *J* = 8.6 Hz, 4H), 7.21 (d, *J* = 8.5 Hz, 4H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.92 (dd, *J* = 2.4 Hz, 8.9 Hz, 2H), 6.51 (d, *J* = 2.4 Hz, 2H), 5.63 (d, *J* = 4.4 Hz, 2H), 5.19 (d, *J* = 4.4 Hz, 2H), 5.05 (br.s, 1H), 3.65 (s, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 158.4, 157.3, 140.2, 133.1, 131.3, 130.4, 130.0, 129.6, 129.1, 125.2, 119.0, 115.6, 109.0, 103.8, 101.5, 55.0, 50.4 ppm; ESI-HRMS: calcd. for $C_{38}H_{29}Cl_2NO_5$+Na 672.1315, found 672.1325.



5cc 64% yield; white solid; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{20}$ = +234.8 (c = 0.23 in $CHCl_3$); >99.5% *ee*, determined by HPLC analysis [Daicel Chiralcel AD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 18.50 min, t(major) = 24.79 min]; ^{1}H NMR (300 MHz, $CDCl_3$):

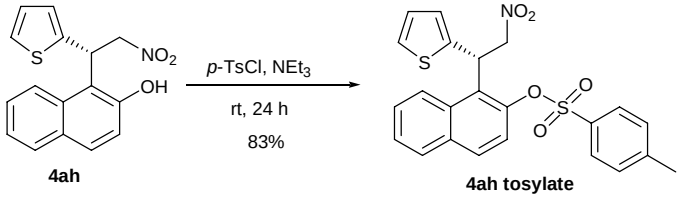
δ = 7.96 (d, *J* = 1.7 Hz, 2H), 7.69 (d, *J* = 8.9 Hz, 2H), 7.34 (dd, *J* = 1.9, 8.9 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 7.21-7.13 (m, 6H), 6.99 (t, *J* = 8.6 Hz, 4H), 5.61 (d, *J* = 4.3 Hz, 2H), 5.22 (d, *J* = 4.2 Hz, 2H), 5.14 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 162.1 (d, $^{1}J_{C,F}$ = 244.8 Hz), 157.0, 137.2, 131.0, 130.8, 130.2, 129.6 (d, $^{3}J_{C,F}$ = 8.1 Hz), 129.4, 128.5, 124.5, 120.4, 116.8, 115.9 (d, $^{2}J_{C,F}$ = 21.3 Hz), 112.7, 104.0, 50.0 ppm; ESI-HRMS: calcd. for $C_{36}H_{23}Br_2F_2NO_3$+Na 735.9905, found 735.9910.



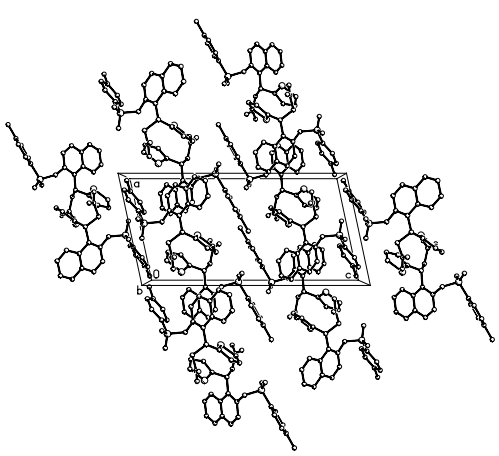
4cc was obtained as a minor product in 23% yield; yellow oil; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{20}$ = -44.0 (c = 0.26 in $CHCl_3$); 91% *ee*, determined by HPLC analysis [Daicel Chiralcel OD, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ= 254 nm, t(minor) = 11.95 min, t(major) = 12.91 min]; ^{1}H NMR (300 MHz, DMSO): δ = 10.36 (s, 1H), 8.10-8.07 (m, 2H), 7.74 (d, *J* = 8.9 Hz, 1H), 7.56 (dd, *J* = 2.1, 9.2 Hz, 1H), 7.44-7.39 (m, 2H), 7.22 (d, *J* = 8.9 Hz, 1H), 7.11 (t, *J* = 8.9 Hz, 2H), 5.70-5.63 (m, 1H), 5.59-5.49 (m, 2H) ppm; ^{13}C NMR (75 MHz, DMSO): δ = 161.0 (d, $^{1}J_{C,F}$ = 241.5 Hz), 153.7, 136.0, 135.9, 131.6, 130.3, 129.7, 129.5 (d, $^{3}J_{C,F}$ = 8.0 Hz), 128.7, 124.8, 120.0, 116.9, 115.5, 115.2 (d, $^{2}J_{C,F}$ = 21.2 Hz), 77.9, 59.7 ppm; ESI-HRMS: calcd. for $C_{18}H_{13}BrFNO_3$-H 387.9990, found 387.9987.

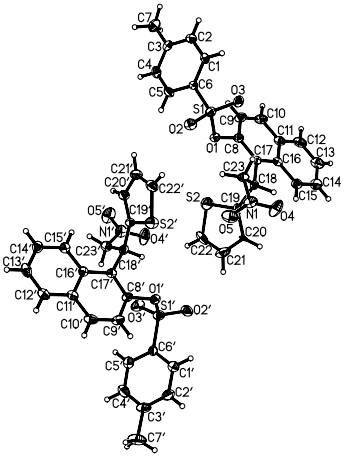
Crystal data and absolute configuration of enantiopure *p*-Tosylate of 4ah

In order to determine the absolute configuration of the F-C product catalysed by bifunctional **1a**, the oily chiral adduct **4ah** was converted to the corresponding *p*-tosylate. Then enantiopure crystals suitable for X-ray analysis were obtained from a mixture of EtOAc and *n*-hexane. Its absolute configuration was determined to be (*S*), therefore the absolute configuration of the tertiary carbon center of **4ah** was (*S*) (**thermal ellipsoids are shown at 30% probability**).



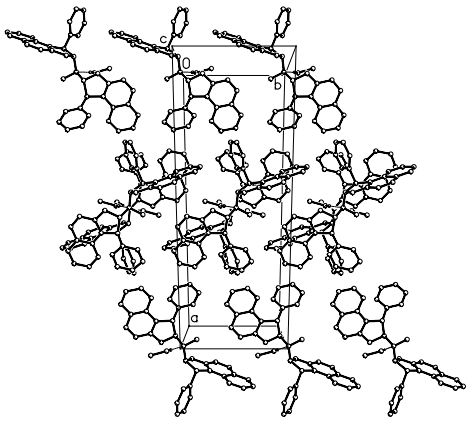
4ah **4ah tosylate**

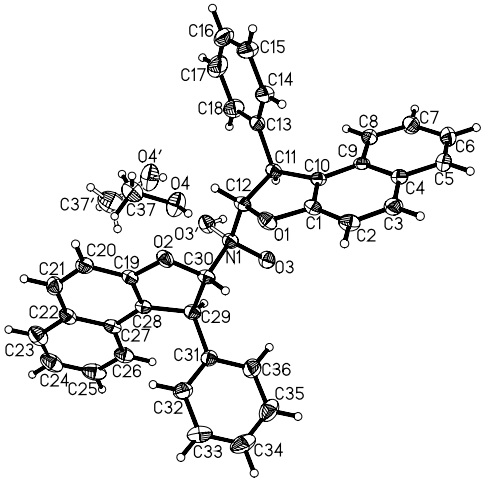




Identification code	**4ah tosylate**
Empirical formula	C23 H19 N O5 S2
Formula weight	453.51
Temperature	290(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 10.090(2) A alpha = 90 deg. b = 10.883(2) A beta = 101.95(2) deg. c = 20.027(4) A gamma = 90 deg.
Volume	2151.5(7) A^3
Z, Calculated density	4, 1.400 Mg/m^3
Absorption coefficient	0.283 mm^-1
F(000)	944
Crystal size	0.48 x 0.40 x 0.26 mm
Theta range for data collection	1.04 to 25.50 deg.
Limiting indices	-12<=h<=12, -13<=k<=13, -24<=l<=24
Reflections collected / unique	9211 / 7999 [R(int) = 0.0192]
Completeness to theta = 25.50	100.0 %
Absorption correction	Empirical
Max. and min. transmission	0.9837 and 0.9296
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7999 / 1 / 562
Goodness-of-fit on F^2	0.886
Final R indices [I>2sigma(I)]	R1 = 0.0439, wR2 = 0.0786
R indices (all data)	R1 = 0.0856, wR2 = 0.0868
Absolute structure parameter	-0.02(6)
Extinction coefficient	0.0058(4)
Largest diff. peak and hole	0.230 and -0.346 e.A^-3

Crystal data and structure refinement for racemic 5aa

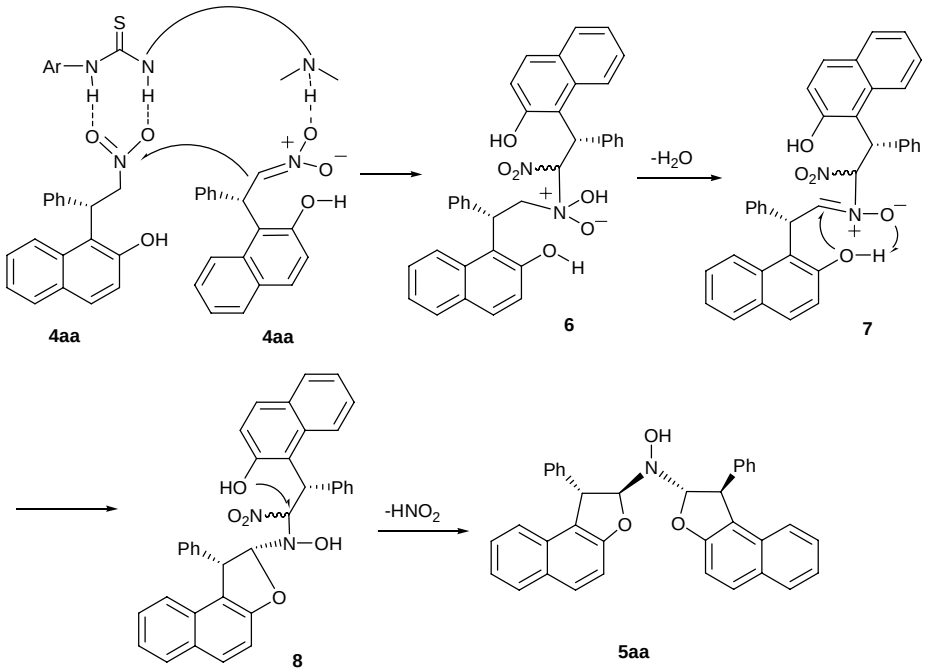




Identification code	**5aa**
Empirical formula	C37 H31.50 N O4
Formula weight	554.13
Temperature	289(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 30.038(4) A alpha = 90 deg. b = 9.530(1) A beta = 124.57(1) deg. c = 24.662(4) A gamma = 90 deg.
Volume	5813.3(14) A^3
Z, Calculated density	8, 1.266 Mg/m^3
Absorption coefficient	0.082 mm^-1
F(000)	2340
Crystal size	0.56 x 0.34 x 0.28 mm
Theta range for data collection	1.65 to 25.50 deg.
Limiting indices	0<=h<=36, 0<=k<=11, -29<=l<=24
Reflections collected / unique	6867 / 5409 [R(int) = 0.0175]
Completeness to theta = 25.50	100.0 %
Absorption correction	None

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5409 / 2 / 413
Goodness-of-fit on F^2	0.864
Final R indices [I>2sigma(I)]	R1 = 0.0439, wR2 = 0.0515
R indices (all data)	R1 = 0.1273, wR2 = 0.0599
Extinction coefficient	0.00166(4)
Largest diff. peak and hole	0.179 and -0.158 e.A^-3

Plausible mechanism for thiourea-tertiary amine catalysed cascade reactions of Friedel-Craft product.

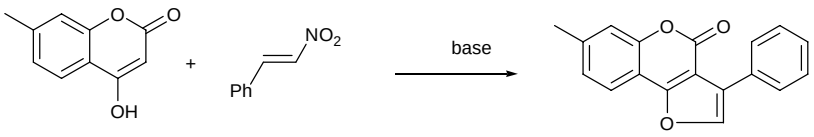


We have proposed a plausible mechanism for the novel domino reactions. As illustrated in above scheme, two molecular **4aa** might be concertedly activated by the bifunctional thiourea-tertiary amine catalyst, and serve as electrophile and nucleophile, respectively, to give intermediate **6**, which would produce **7** after removing a H_2O. The observed kinetic resolution might be attained in the presence of a chiral catalyst. Then one hydroxyl group would attack the adjacent nitrone functionality to generate the desired dihydrofuranyl-2-hydroxylamine structure. Finally, the nitro group of **8** might be substituted by the hydroxyl group to form the other dihydrofuranyl composition through the elimination of a nitrous acid (HNO_2).[1] In fact, the pH value of the solution of domino reactions is found to be much lower than that of the starting materials, which shows that the acidic compound is generated in the domino reactions. Moreover, a simple base (such as the arrangement of K_2CO_3 and 18-crown-6 or TMG) could smoothly promoted the same

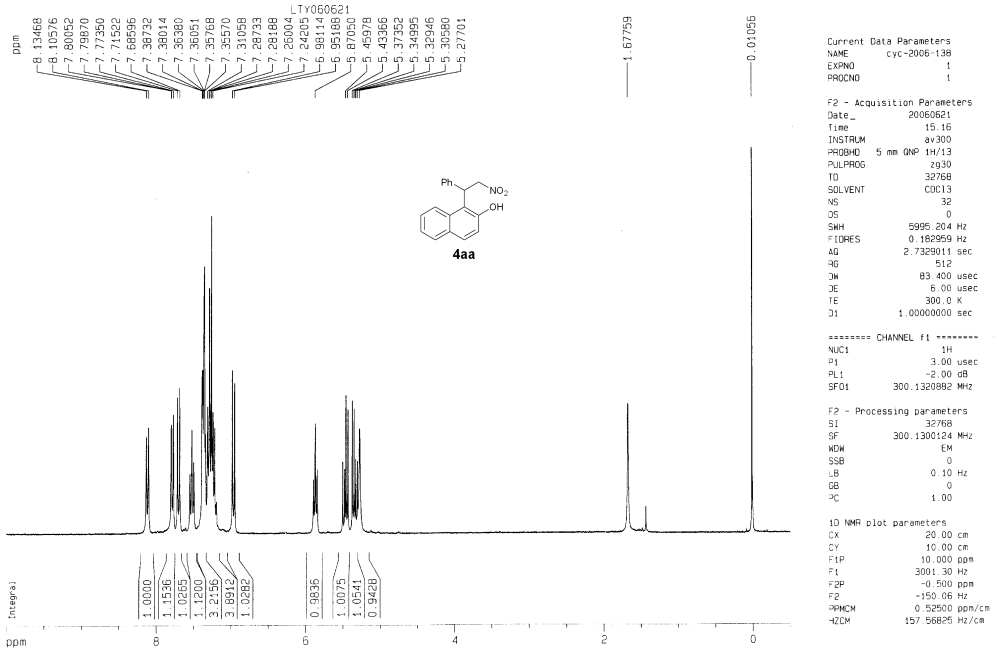
domino reactions, indicating that one **4aa** (nitroalkane) would be deprotonated and react with the electrophilic nitro group of other **4aa**, and then the domino reactions would be facilitated. Nevertheless, the real reaction mechanism still remains to be explored.

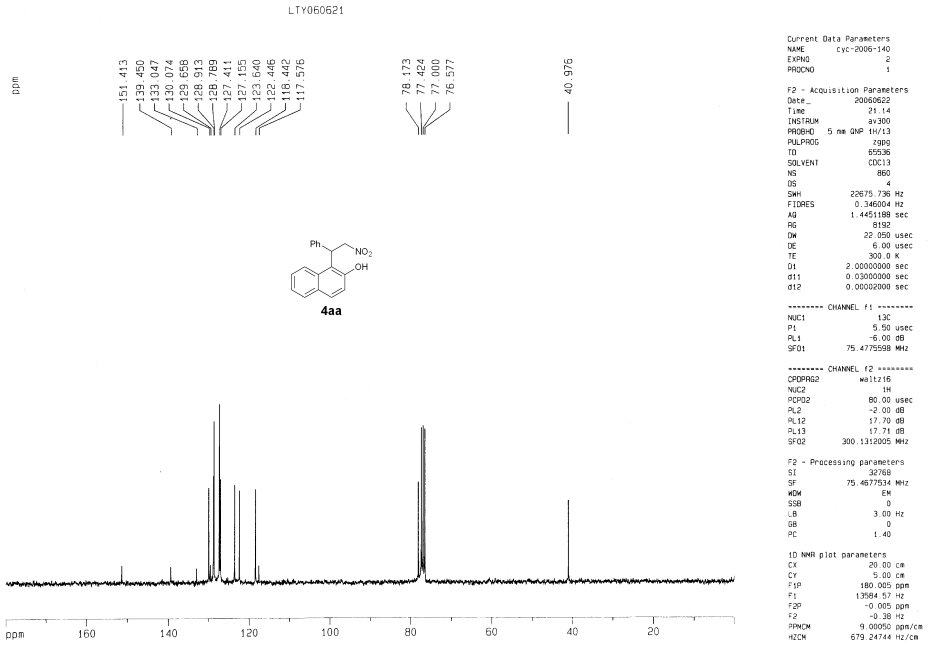
Reference

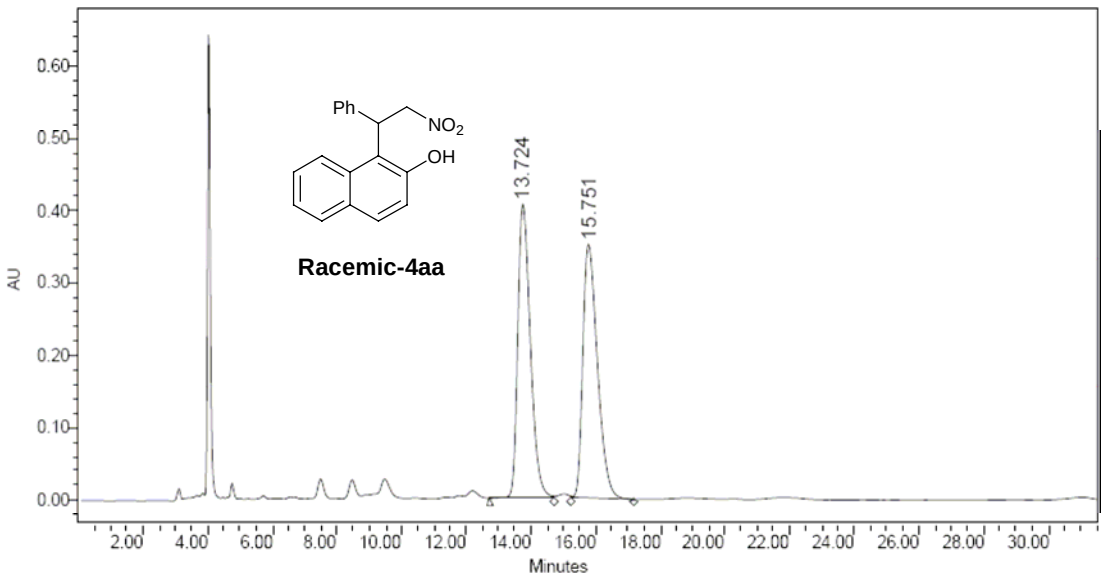
1. In a related reaction, 7-methyl-3-phenyl-4H-furo[3,2-b]-chromen-4-one rather than the dimerisation product observed in our reaction was obtained from 7-Methyl 4-hydroxycoumarin by condensing with nitrostyrene, see: F. H. Havaldar and S. S. Bhise, *Indian J. Heterocyclic Chem.* **2003**, *13*, 15.



NMR spectra and HPLC chromatograms







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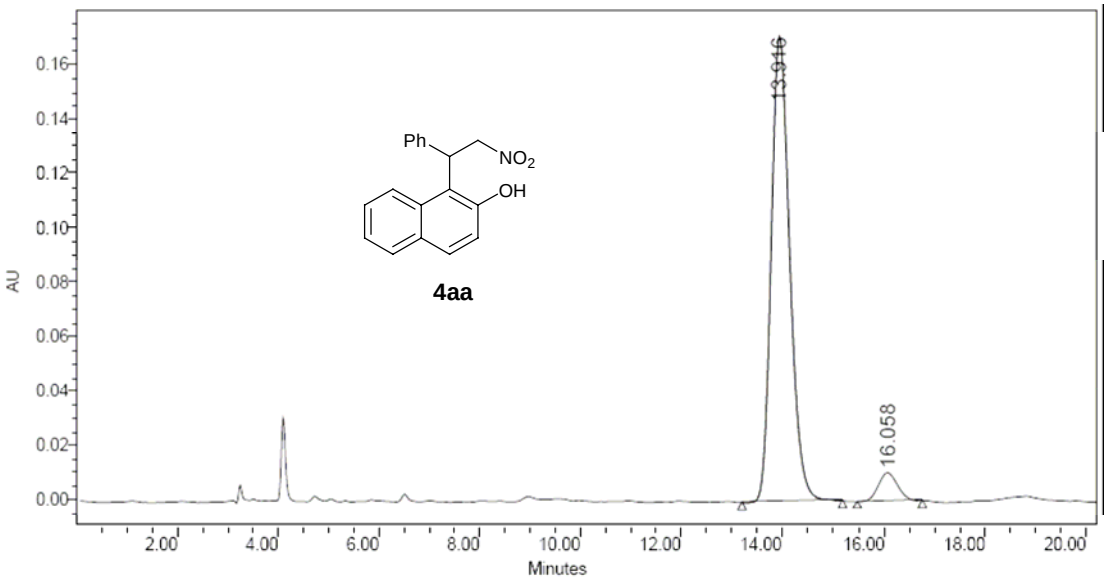
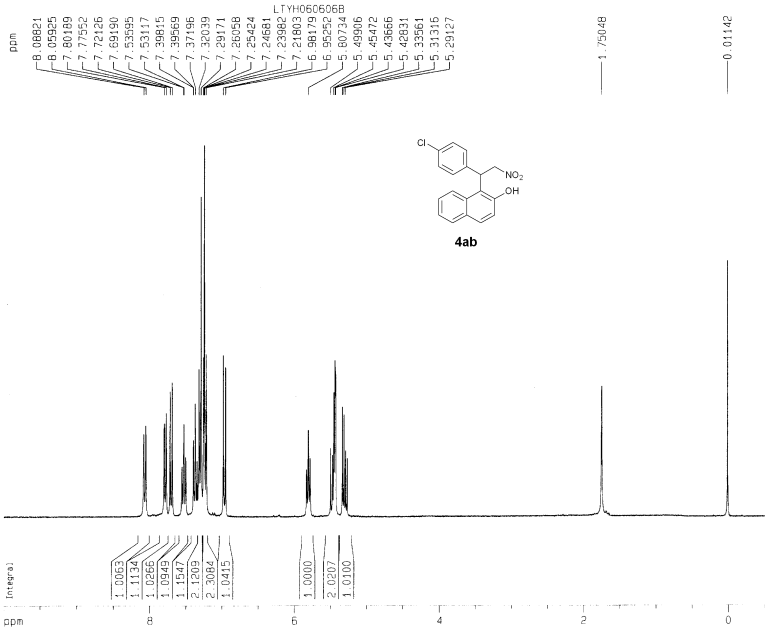
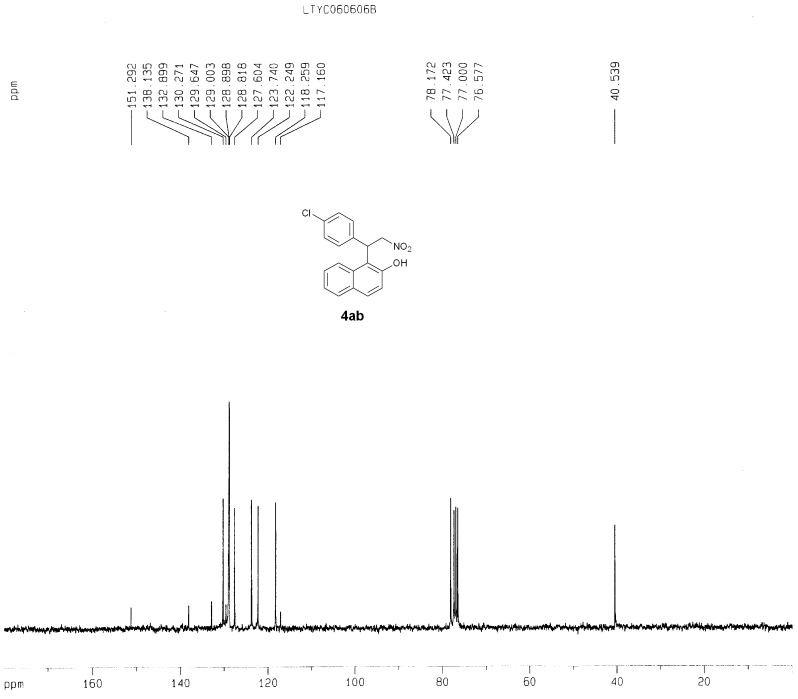
2	15.751	11119104	50.07	353295	46.40



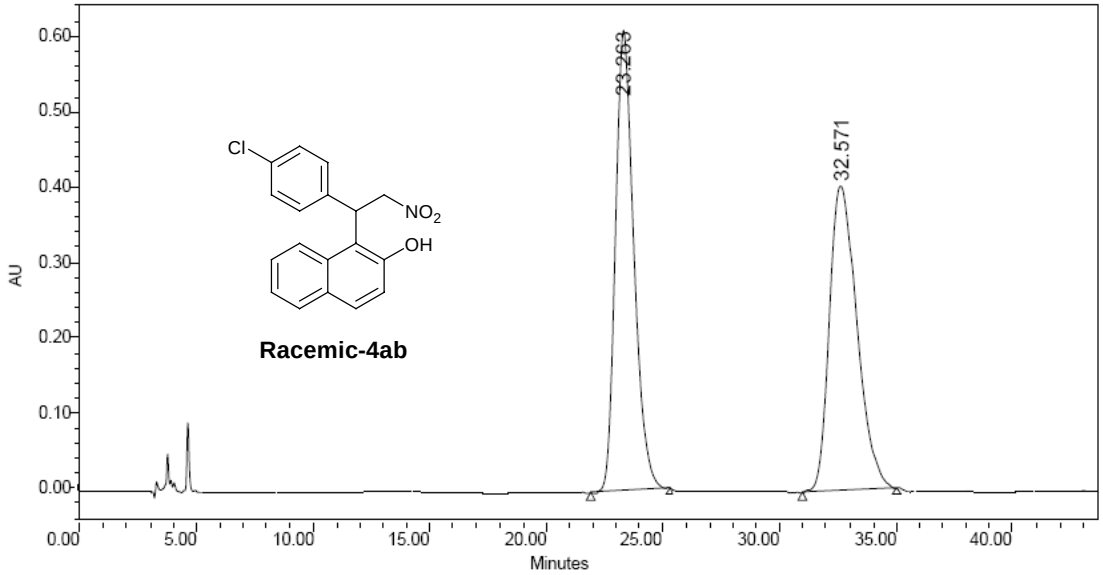
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	13.916	4307156	96.34	170485	95.52
2	16.058	163789	3.66	7993	4.48



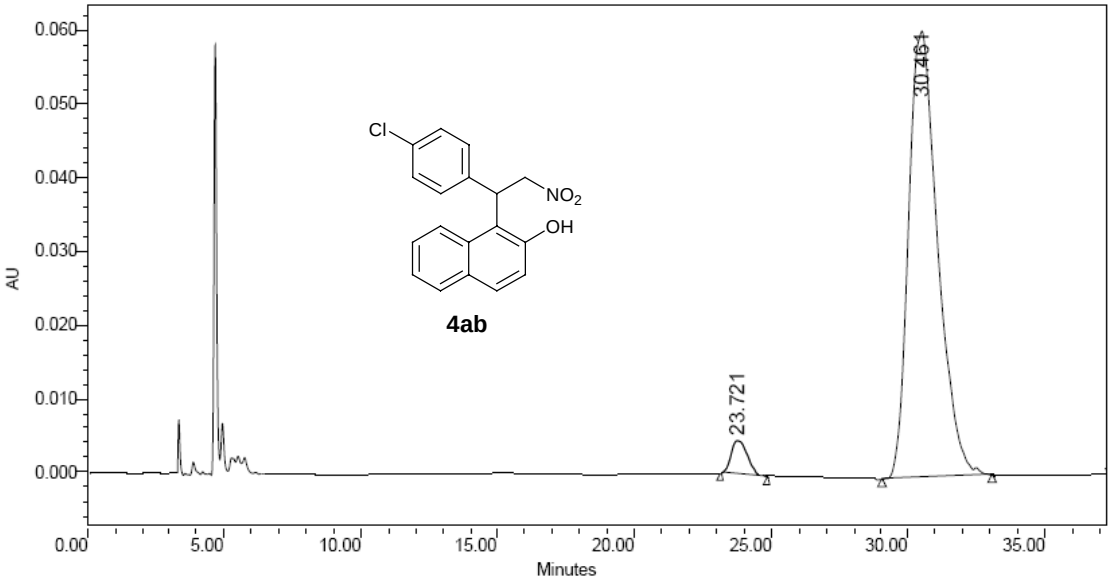

```
Current Data Parameters
NAME          cyc-2006-115
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060606
Time                  16.31
INSTRUM               av300
PROBHD  5 mm QNP 1H/13
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH                5995.204 Hz
FIDRES            0.182959 Hz
AQ                2.7329011 sec
RG                     512
DW                  83.400 usec
DE                   6.00 usec
TE                  300.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   3.00 usec
PL1                 -2.00 dB
SF01           300.1320882 MHz

F2 - Processing parameters
SI                   32768
SF              300.1300124 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                 10.000 ppm
F1                3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM             0.52500 ppm/cm
HZCM            157.56825 Hz/cm
```



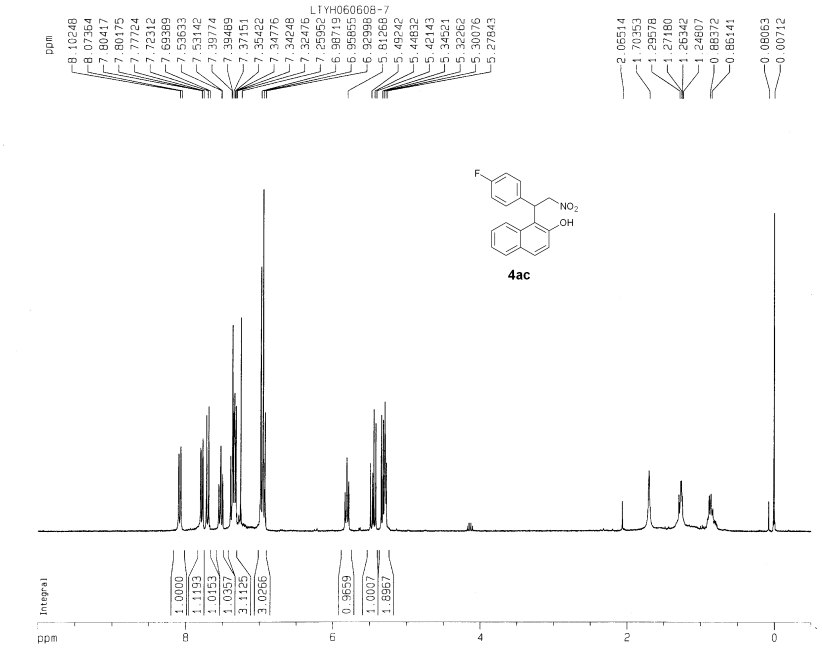

```
Current Data Parameters
NAME          cyc-2006-115
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060606
Time                  17.19
INSTRUM               av300
PROBHD  5 mm QNP 1H/13
PULPROG               zgpg
TD                   65536
SOLVENT              CDCl3
NS                     480
DS                       4
SWH               22675.736 Hz
FIDRES            0.346004 Hz
AQ                1.4451188 sec
RG                     8192
DW                  22.050 usec
DE                   6.00 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   5.50 usec
PL1                 -6.00 dB
SF01           75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                 -2.00 dB
PL12                17.70 dB
PL13                17.71 dB
SF02           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677530 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    6.00 cm
F1P                180.005 ppm
F1               13584.57 Hz
F2P                 -0.005 ppm
F2                  -0.38 Hz
PPMCM             9.00050 ppm/cm
HZCM            679.24744 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	23.263	33604200	50.23	611912	60.20
2	32.571	33296121	49.77	404472	39.80



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	23.721	146233	3.15	3995	6.18
2	30.461	4503287	96.85	60647	93.82



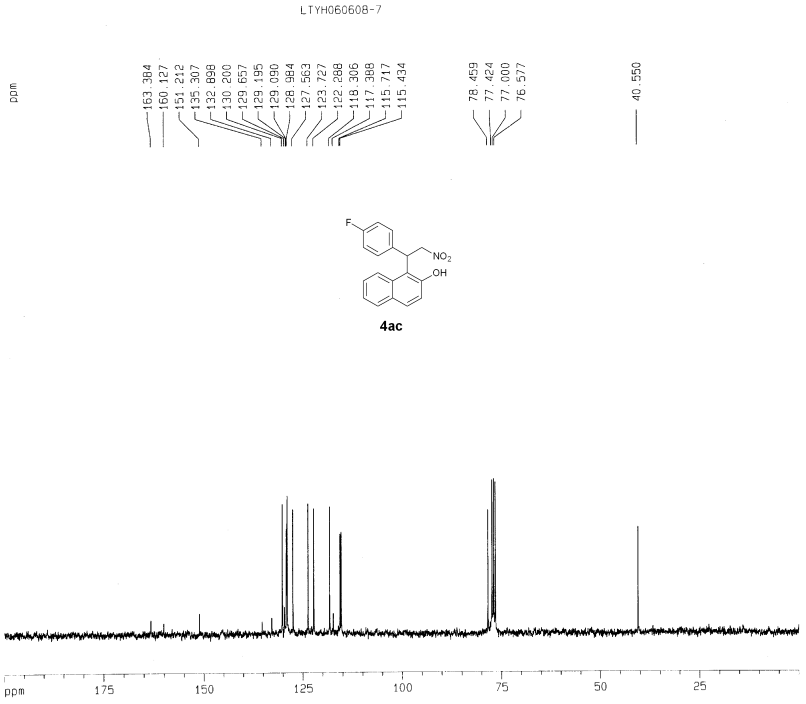
Current Data Parameters
NAME cyc-2006-122
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060609
Time 9.18
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-122
EXPNO 2
PROCNO 1

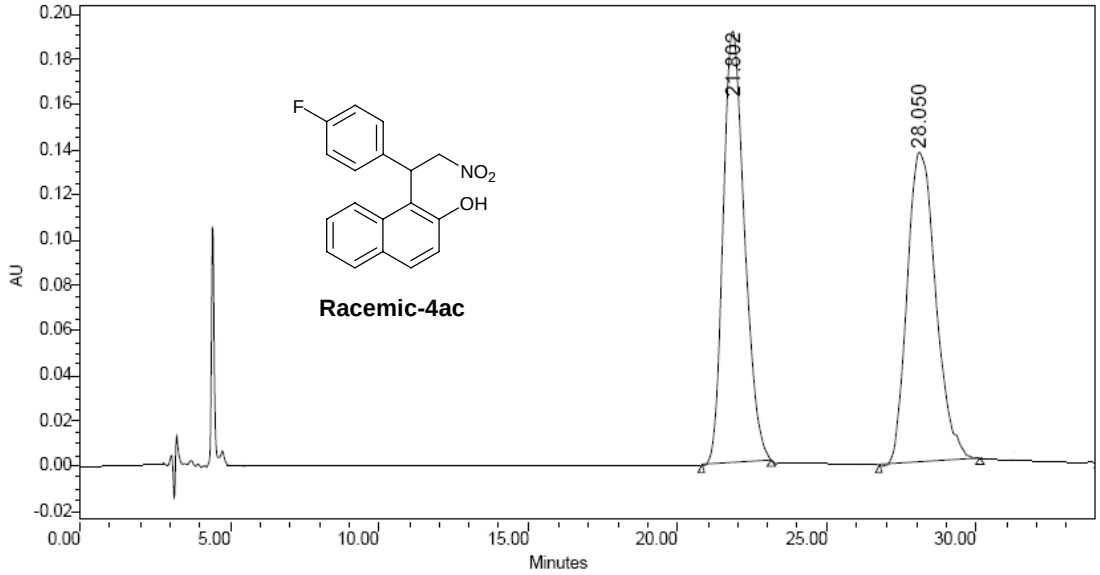
F2 - Acquisition Parameters
Date_ 20060612
Time 8.50
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 548
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

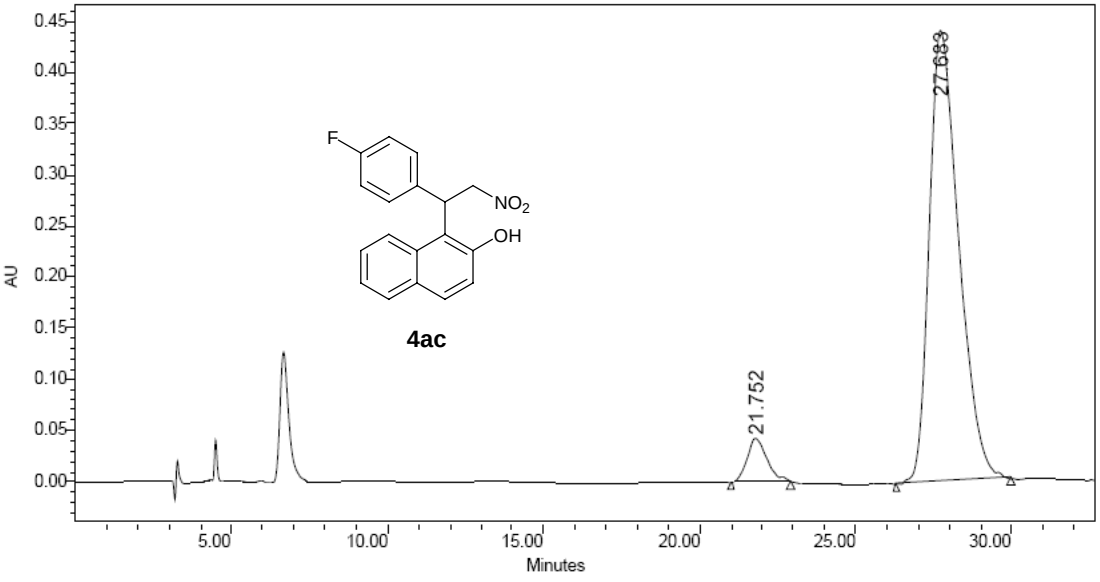
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677533 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

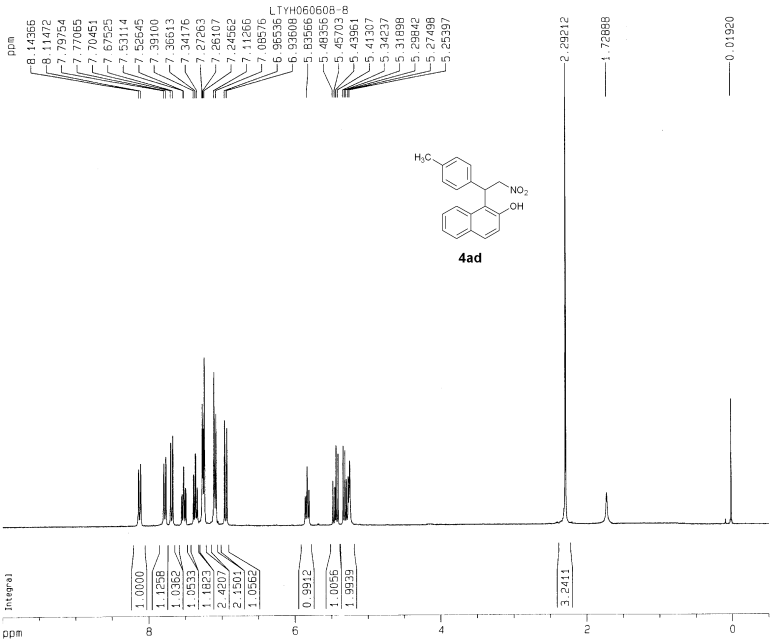
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69641 Hz/cm



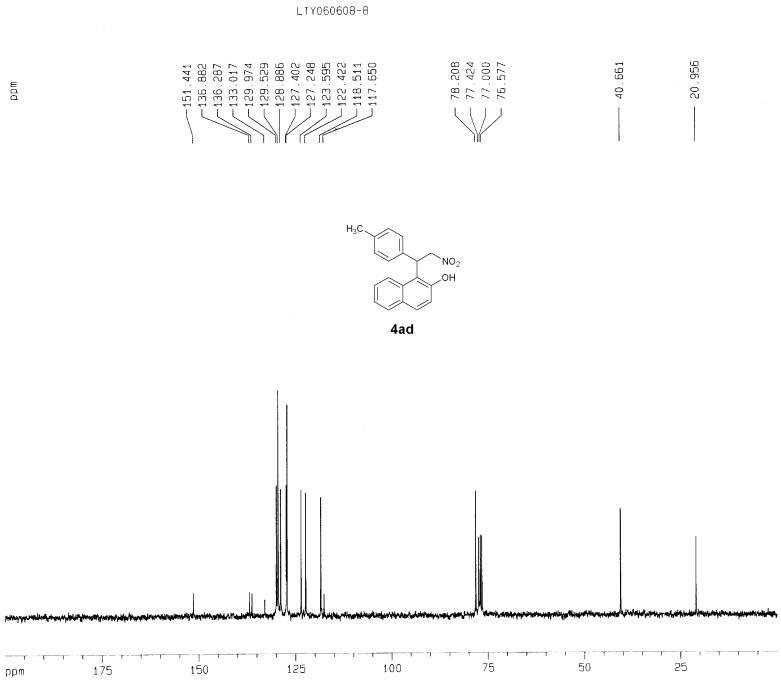
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	21.802	9487346	50.85	190663	58.16
2	28.050	9170828	49.15	137165	41.84



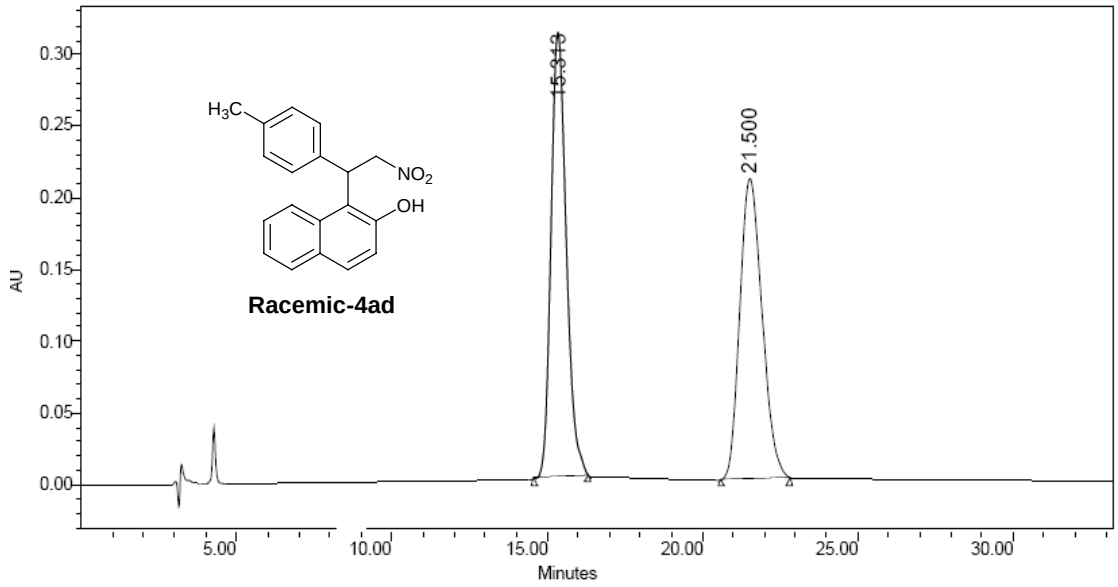
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	21.752	1347151	4.43	36608	7.66
2	27.683	29077625	95.57	441054	92.34



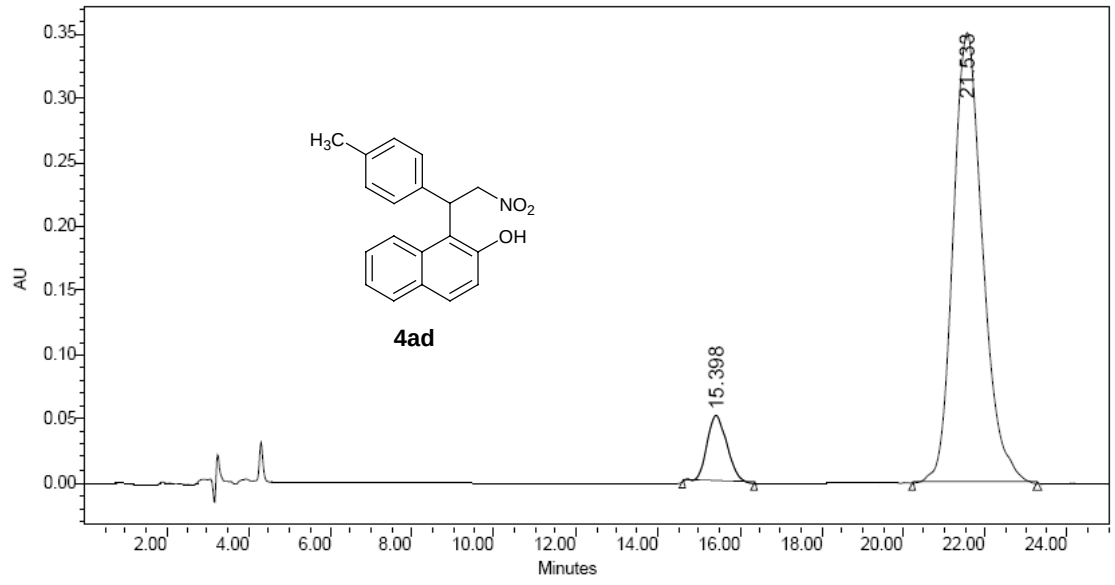

```
Current Data Parameters
NAME          cyc-2006-123
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060609
Time                   9.22
INSTRUM                av300
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      256
DW                   83.400 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    3.00 usec
PL1                  -2.00 dB
SFO1           300.1320882 MHz

F2 - Processing parameters
SI                    32768
SF             300.1300124 MHz
WDW                      EM
SSB                       0
LB                     0.10 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    13.00 cm
F1P                 10.000 ppm
F1                3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM              0.52500 ppm/cm
HZCM             157.56825 Hz/cm
```



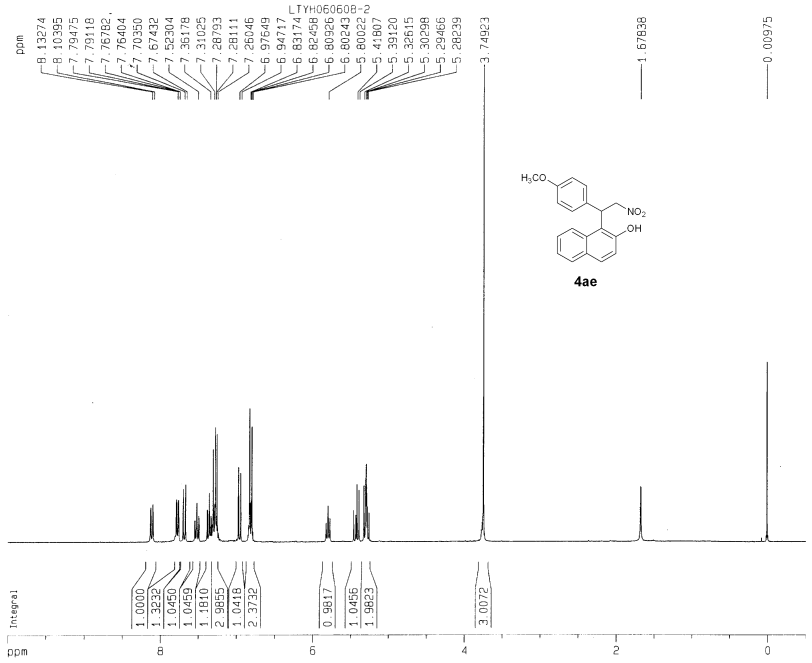

```
Current Data Parameters
NAME          cyc-2006-123
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060612
Time                  14.21
INSTRUM                av300
PROBHD   5 mm QNP 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      352
DS                        4
SWH               22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                     8192
DW                   22.050 usec
DE                    6.00 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    5.50 usec
PL1                  -6.00 dB
SFO1           75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 17.70 dB
PL13                 17.71 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF             75.4677549 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.00 cm
F1P                 200.000 ppm
F1                15093.55 Hz
F2P                 -0.005 ppm
F2                  -0.38 Hz
PPMCM              10.00025 ppm/cm
HZCM             754.69647 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	15.313	10579181	50.24	309826	59.63
2	21.500	10478271	49.76	209725	40.37



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	15.398	1424866	7.43	47114	11.81
2	21.533	17744299	92.57	351912	88.19



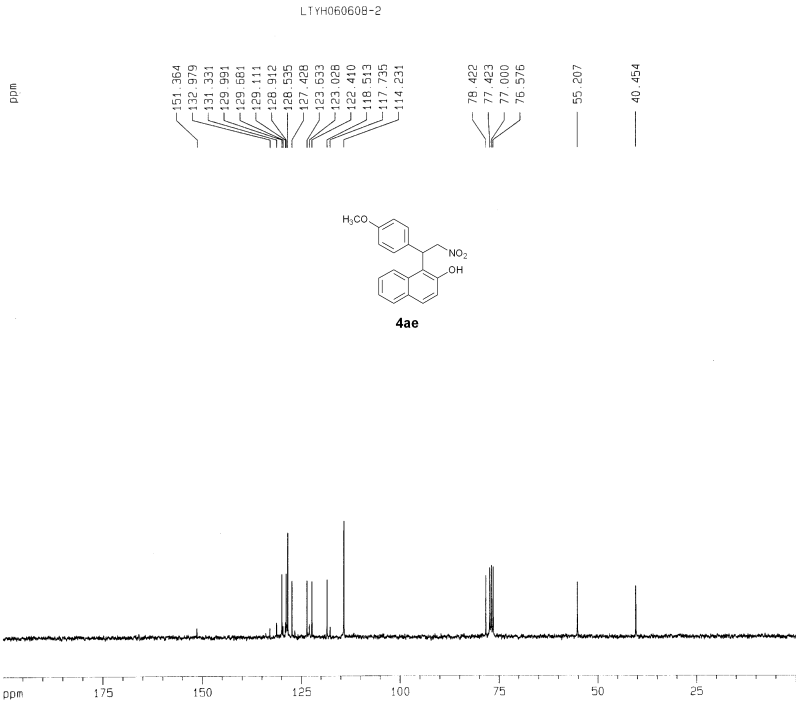
Current Data Parameters
NAME cyc-2006-118
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060609
Time 9.00
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-118
EXPNO 2
PROCNO 1

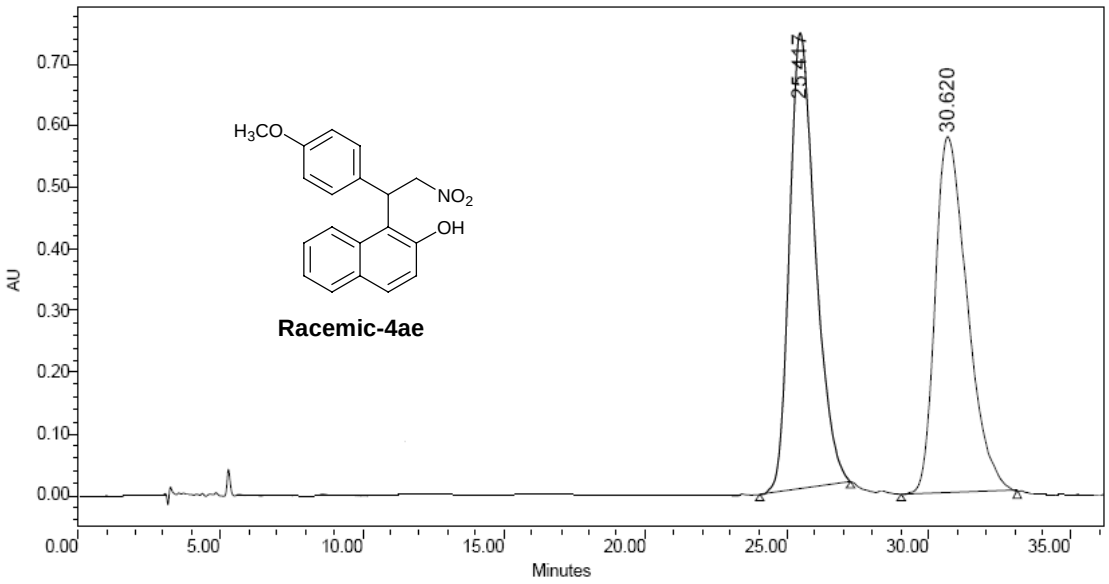
F2 - Acquisition Parameters
Date_ 20060610
Time 9.48
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 416
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451088 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

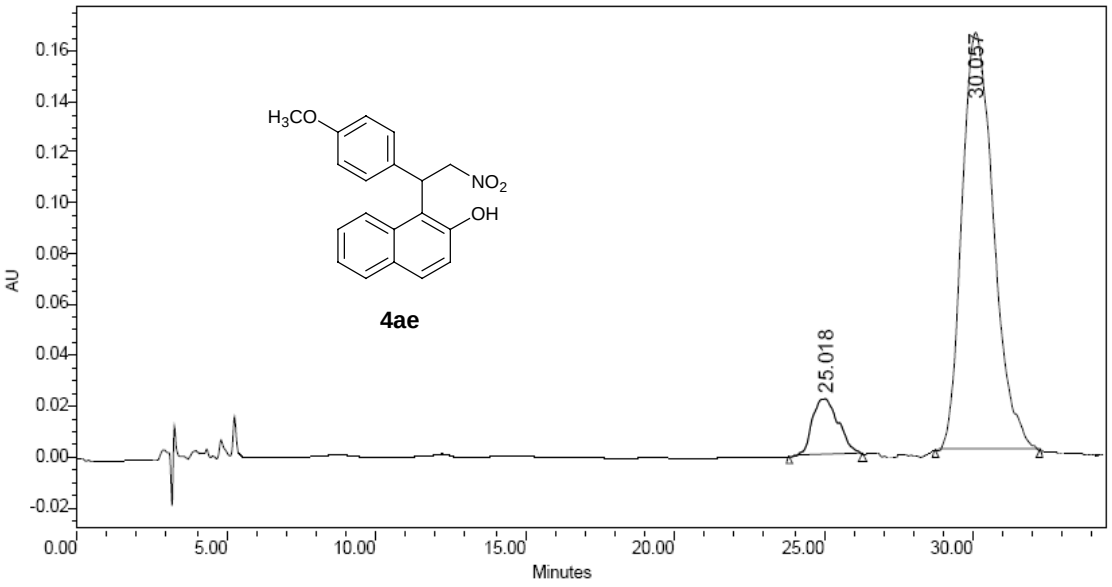
F2 - Processing parameters
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 3.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69641 Hz/cm



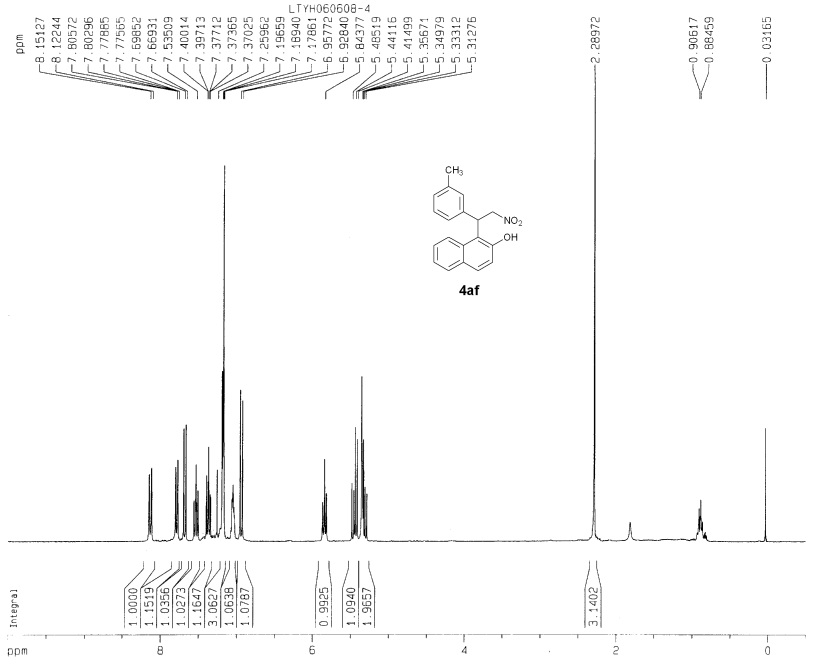
Racemic-4ae

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	25.417	47111830	50.72	740810	56.12
2	30.620	45778239	49.28	579268	43.88

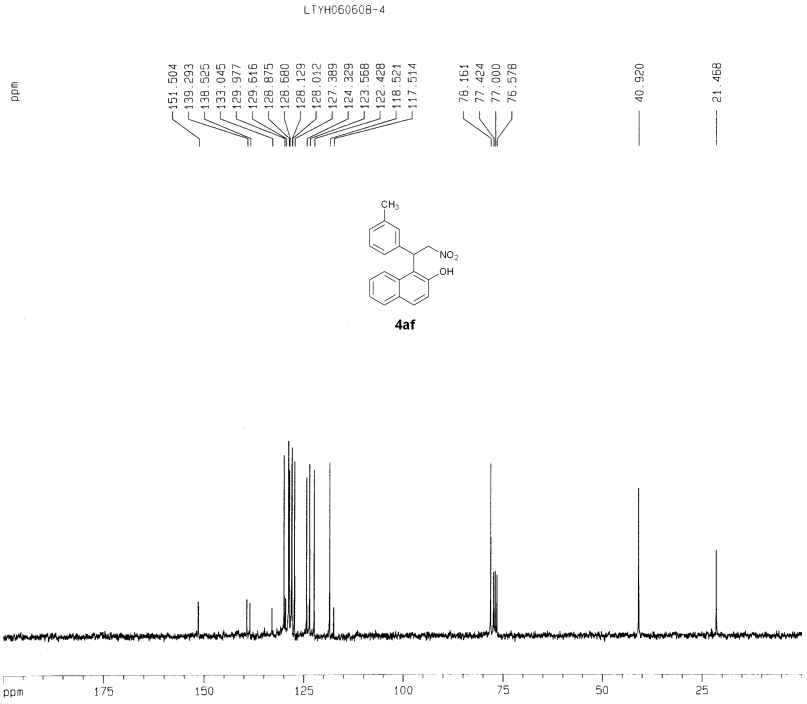


4ae

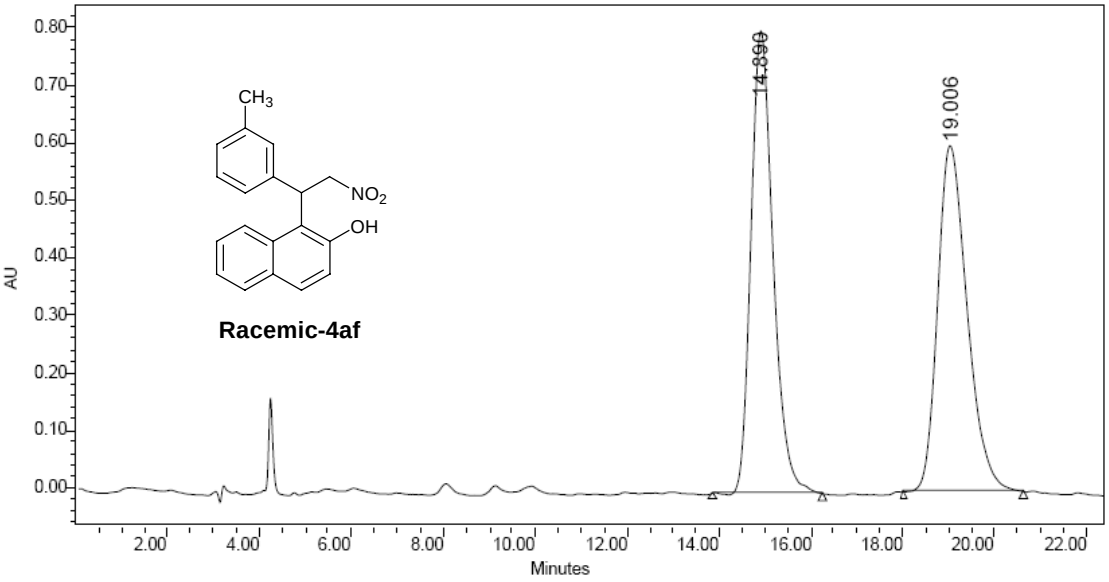
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	25.018	999025	7.56	18943	10.31
2	30.057	12207378	92.44	164817	89.69



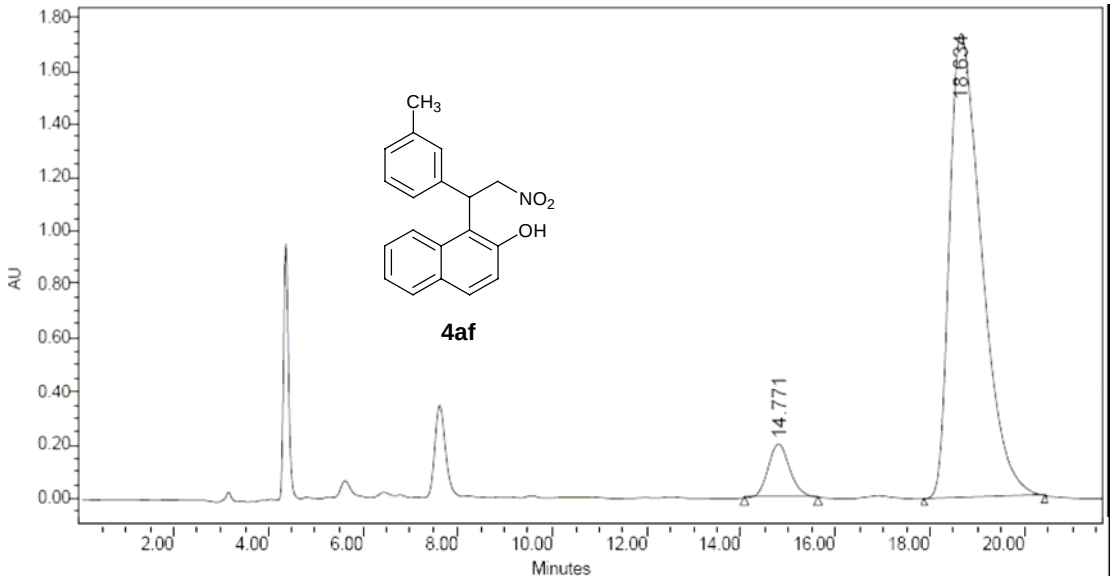

```
Current Data Parameters
NAME        cyc-2006-120
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060609
Time                9.08
INSTRUM           av300
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            5995.204 Hz
FIDRES        0.182959 Hz
AQ            2.7329011 sec
RG                  256
DW             83.400 usec
DE              6.00 usec
TE              300.0 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               3.00 usec
PL1             -2.00 dB
SF01       300.1320882 MHz

F2 - Processing parameters
SI                32768
SF         300.1300124 MHz
WDW                  EM
SSB                   0
LB               0.10 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               20.00 cm
CY               18.00 cm
F1P            10.000 ppm
F1           3001.30 Hz
F2P             -0.500 ppm
F2           -150.06 Hz
PPMCM         0.52500 ppm/cm
HZCM        157.56825 Hz/cm
```



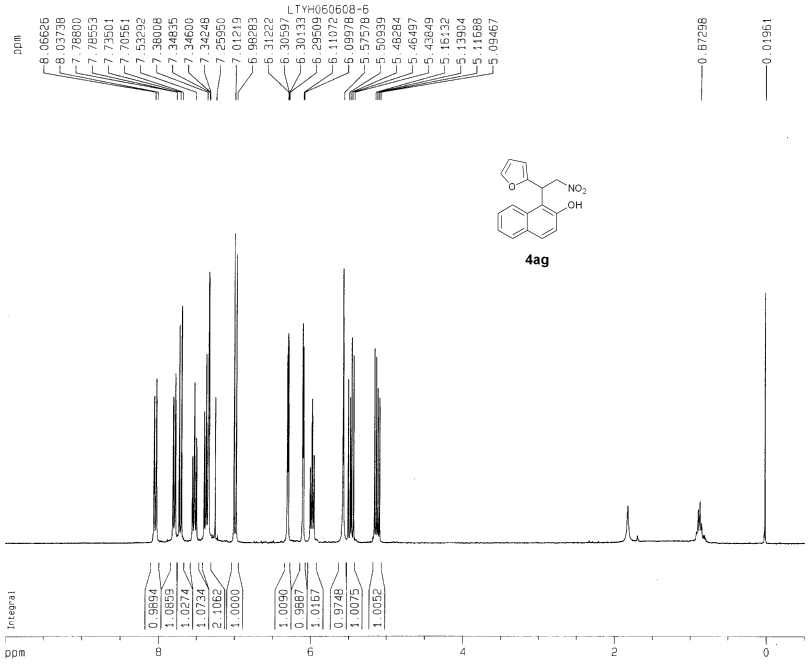

```
Current Data Parameters
NAME        cyc-2006-120
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060611
Time               19.27
INSTRUM           av300
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg
TD                65536
SOLVENT           CDCl3
NS                  188
DS                    4
SWH           22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW              22.050 usec
DE               6.00 usec
TE              300.0 K
D1          2.00000000 sec
d11         0.03000000 sec
d12         0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               5.50 usec
PL1             -6.00 dB
SF01       75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           80.00 usec
PL2             -2.00 dB
PL12            17.70 dB
PL13            17.71 dB
SF02       300.1312005 MHz

F2 - Processing parameters
SI                32768
SF         75.4677565 MHz
WDW                  EM
SSB                   0
LB               3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX               20.00 cm
CY                5.00 cm
F1P            200.000 ppm
F1           15093.55 Hz
F2P             -0.005 ppm
F2              -0.38 Hz
PPMCM        10.00025 ppm/cm
HZCM        754.69647 Hz/cm
```



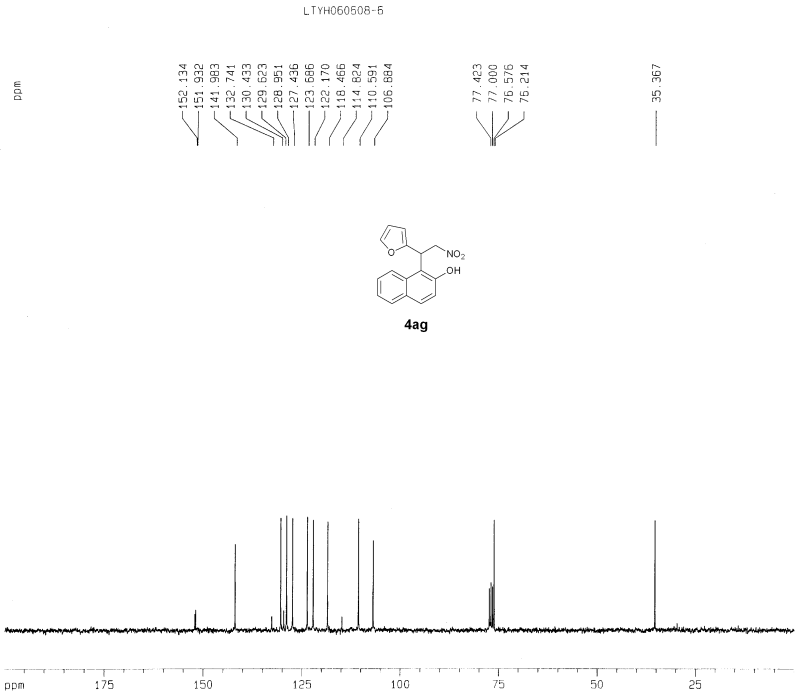
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.890	26893021	50.01	803520	57.19
2	19.006	26883528	49.99	601438	42.81



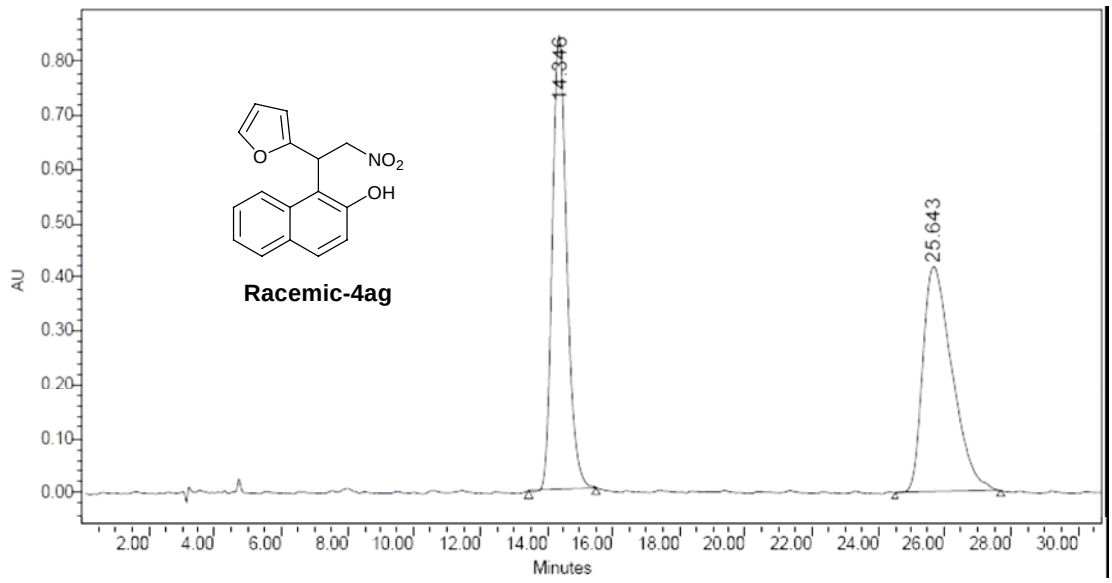
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.771	4014069	4.66	159103	8.40
2	18.634	82208598	95.34	1735804	91.60



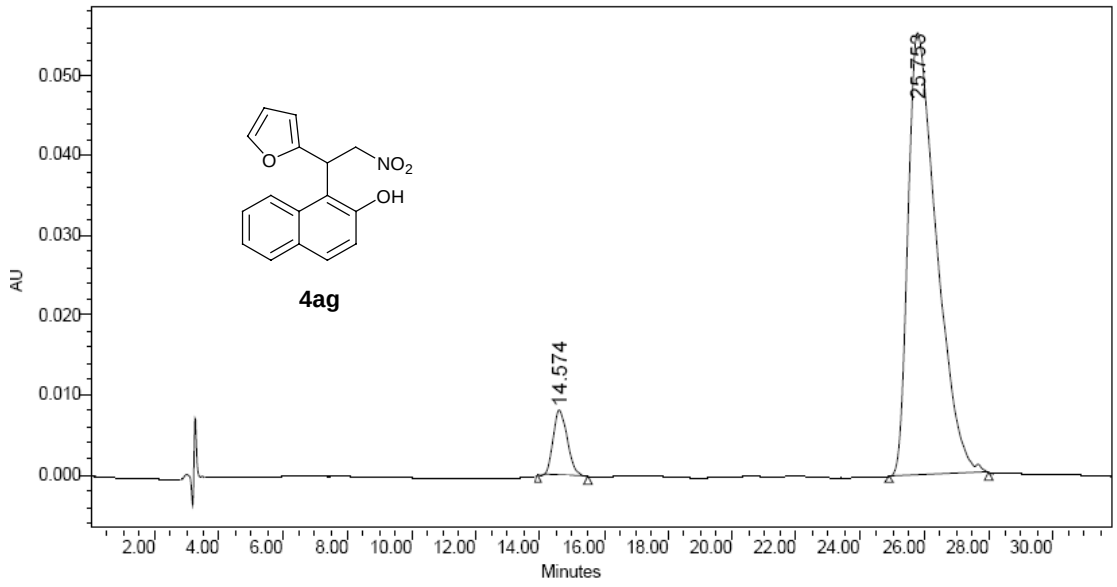

```
Current Data Parameters
NAME          cyc-2006-117
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_          20060608
Time              18.37
INSTRUM             av300
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             5995.204 Hz
FIDRES          0.182959 Hz
AQ              2.7329011 sec
RG                    256
DW                 83.400 usec
DE                  6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  3.00 usec
PL1                -2.00 dB
SFO1         300.1320882 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300124 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                  8.00 cm
F1P               10.000 ppm
F1              3001.30 Hz
F2P               -0.500 ppm
F2              -150.06 Hz
PPMCM           0.52500 ppm/cm
HZCM          157.56825 Hz/cm
```



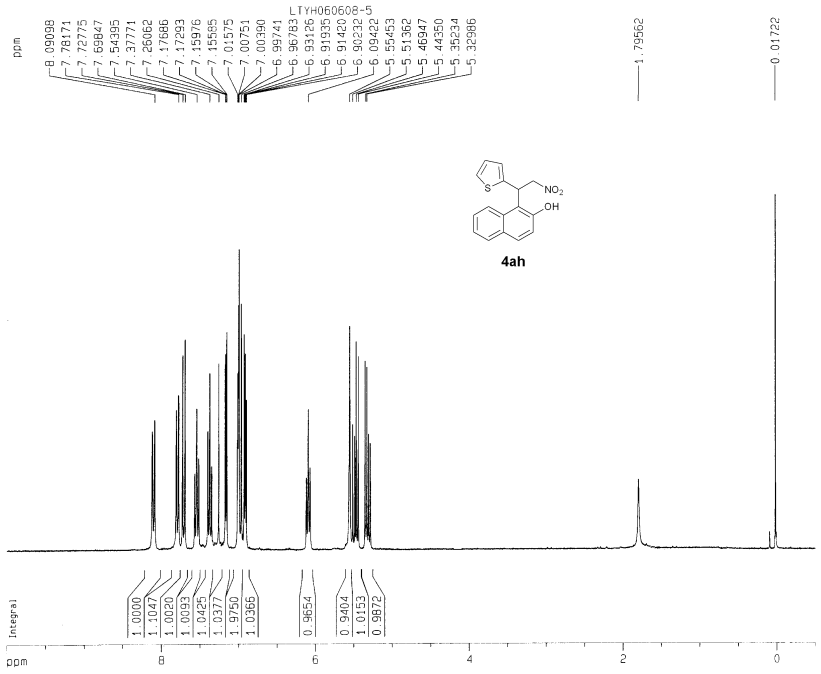

```
Current Data Parameters
NAME          cyc-2006-117
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_          20060610
Time               9.34
INSTRUM             av300
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg
TD                 65536
SOLVENT             CDCl3
NS                   192
DS                     4
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ              1.4451188 sec
RG                  8192
DW                 22.050 usec
DE                  6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

-------- CHANNEL f1 --------
NUC1                   13C
P1                  5.50 usec
PL1                -6.00 dB
SFO1         75.4775598 MHz

-------- CHANNEL f2 --------
CPDPRG2             waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               17.70 dB
PL13               17.71 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677547 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  3.00 cm
F1P              200.000 ppm
F1             15093.55 Hz
F2P               -0.005 ppm
F2              -0.38 Hz
PPMCM          10.00025 ppm/cm
HZCM          754.69647 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.346	25431237	49.67	841907	66.81
2	25.643	25766342	50.33	418304	33.19



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.574	184217	5.07	7107	11.35
2	25.753	3448731	94.93	55518	88.65



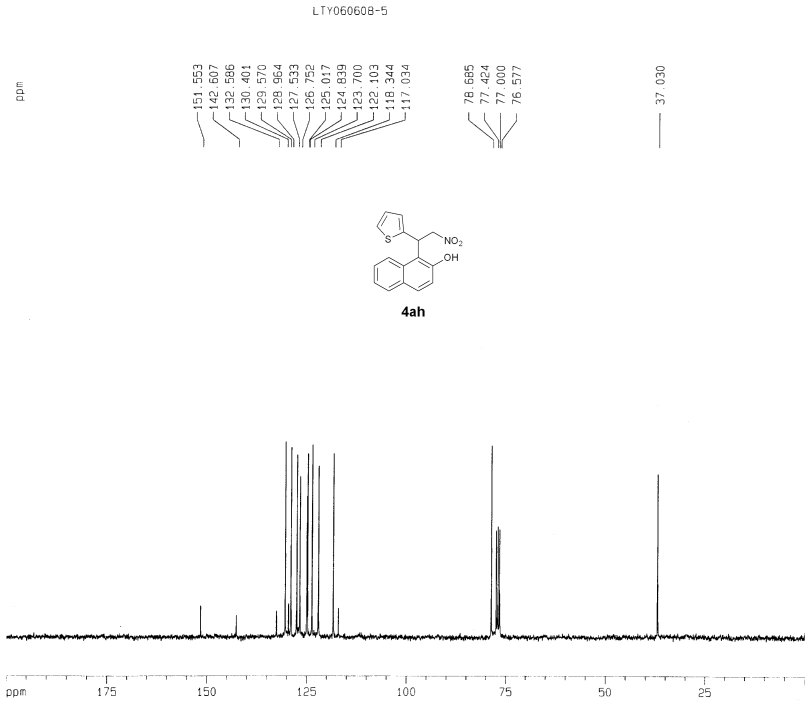
Current Data Parameters
NAME cyc-2006-121
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060609
Time 9.13
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 9.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-121
EXPNO 2
PROCNO 1

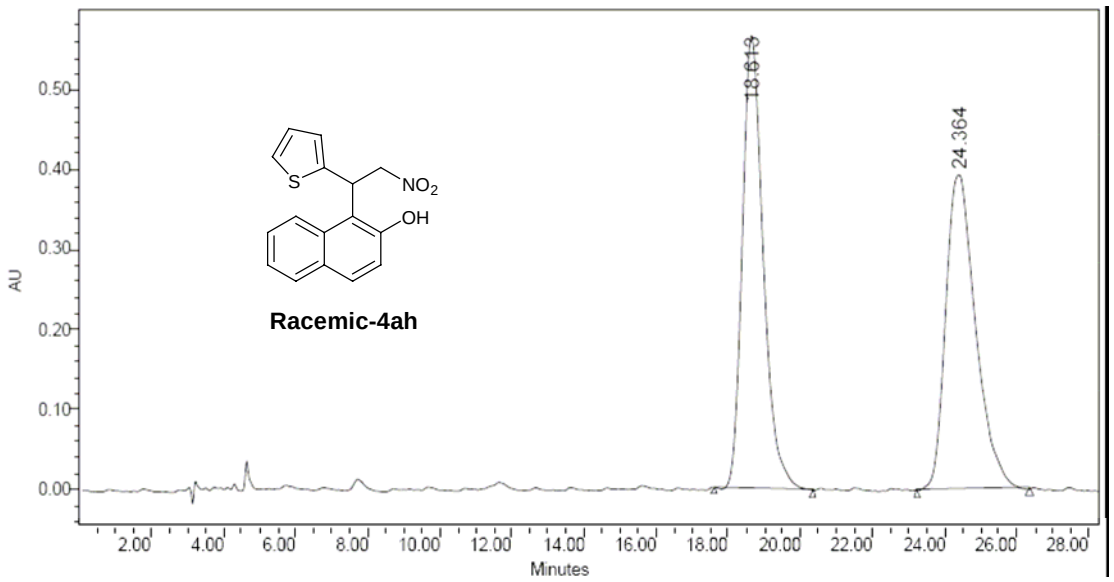
F2 - Acquisition Parameters
Date_ 20060611
Time 19.40
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 793
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

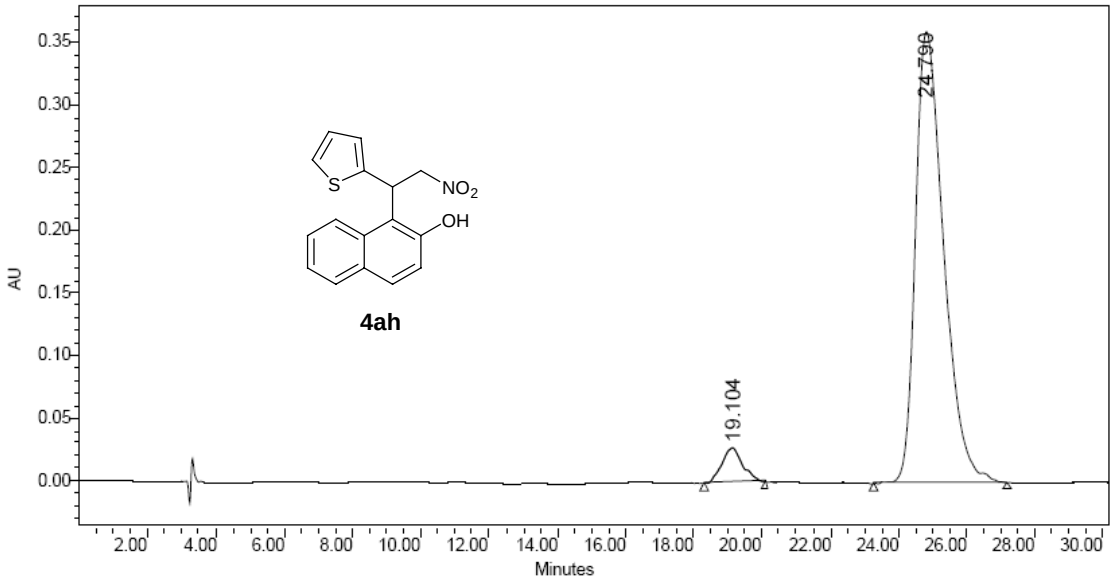
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677547 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

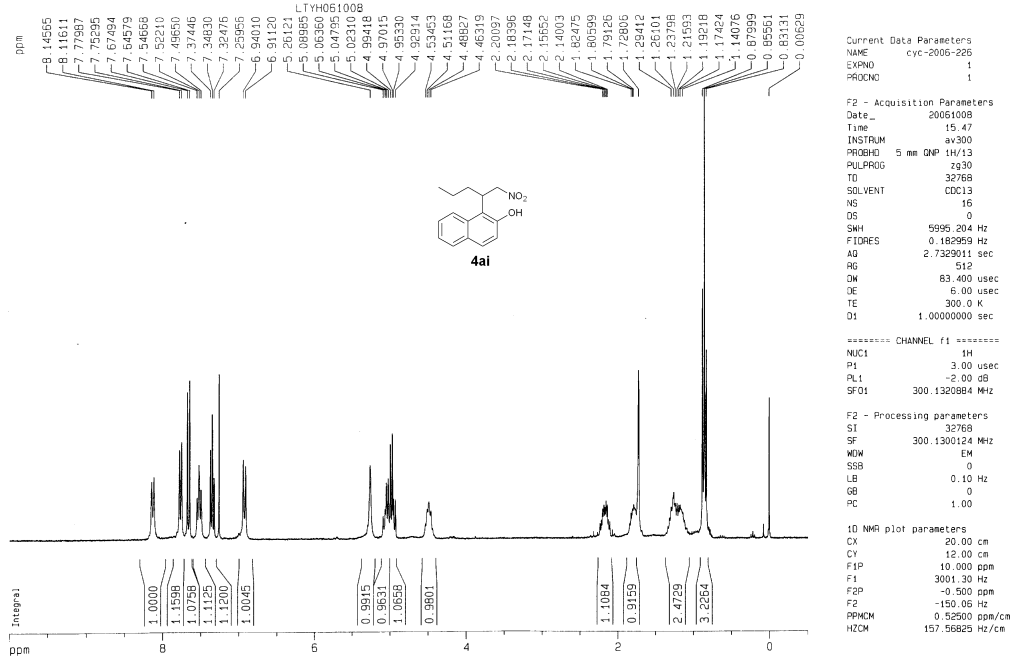
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69647 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	18.613	22821922	50.04	566377	58.98
2	24.364	22786666	49.96	393848	41.02



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	19.104	695915	3.25	21546	5.64
2	24.790	20722681	96.75	360291	94.36



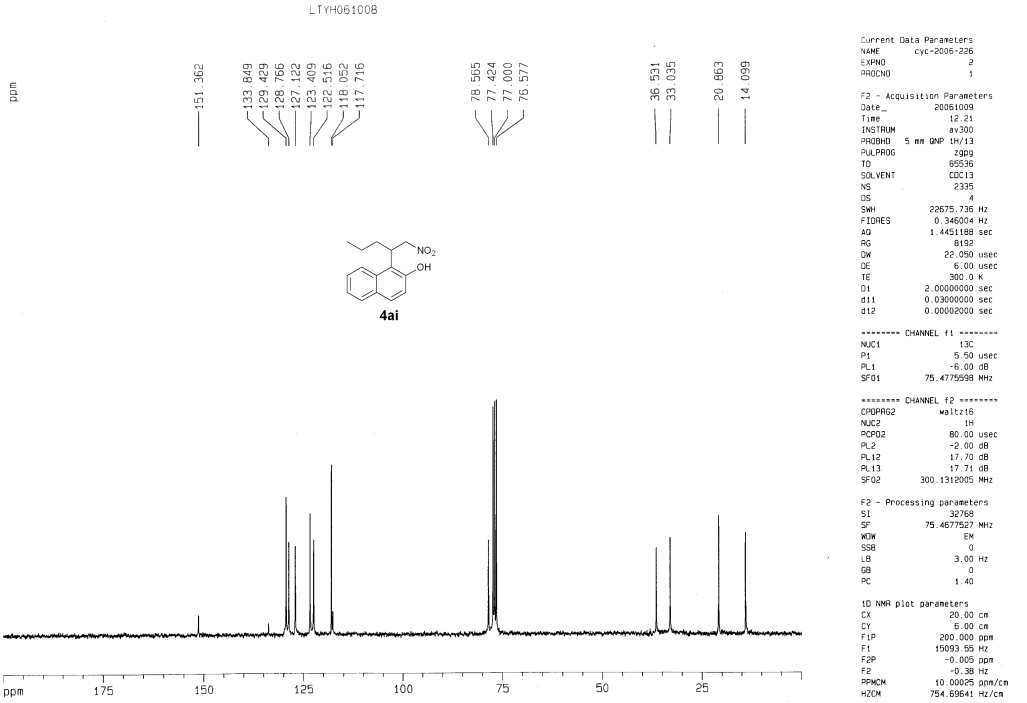
Current Data Parameters
NAME cyc-2006-226
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061008
Time 15.47
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320884 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-226
EXPNO 2
PROCNO 1

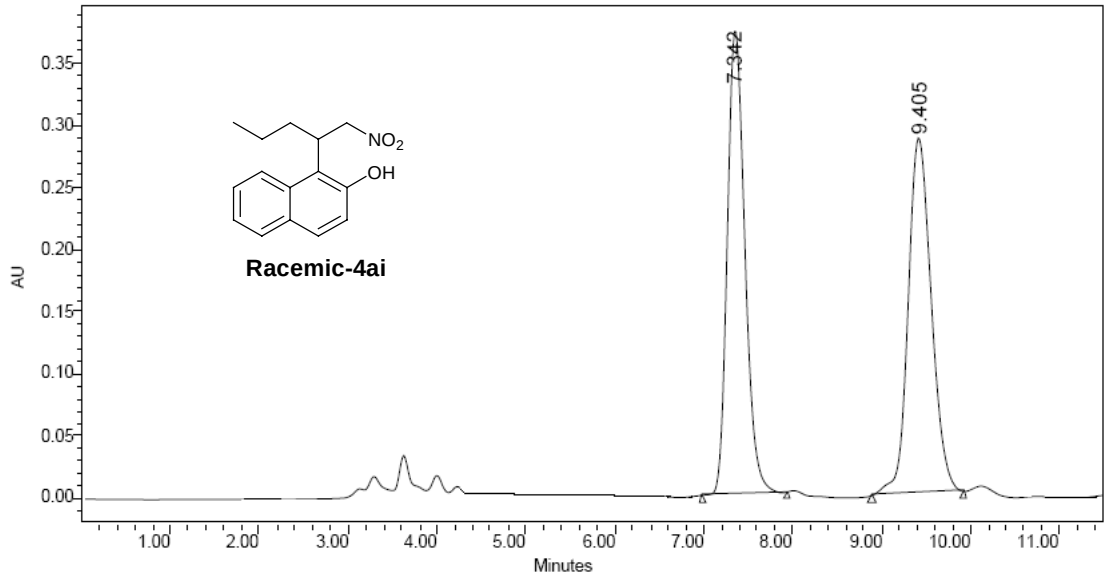
F2 - Acquisition Parameters
Date_ 20061009
Time 12.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2335
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

-------- CHANNEL f1 --------
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

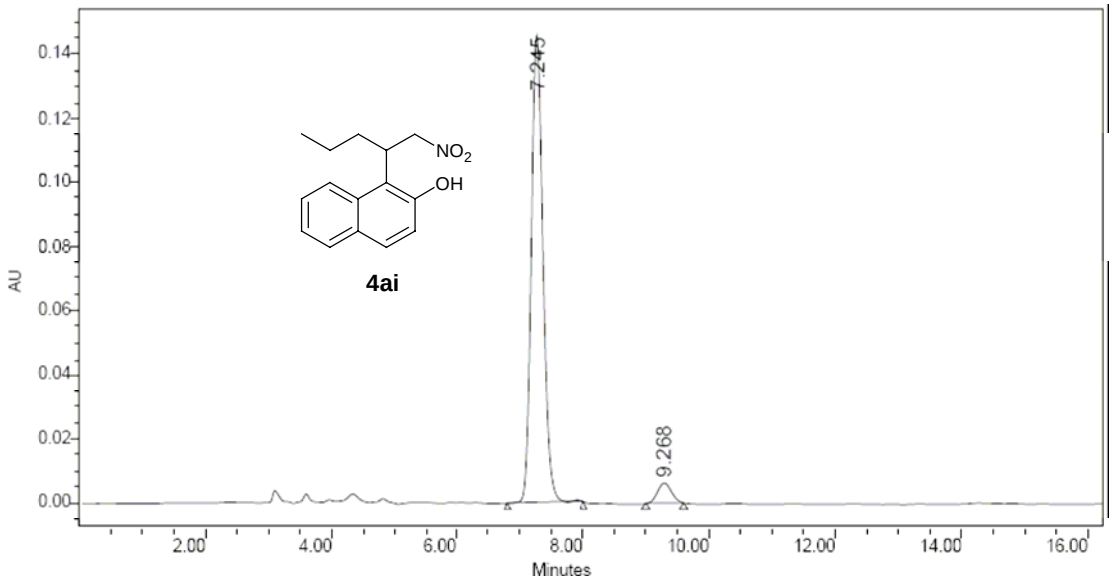
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677527 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

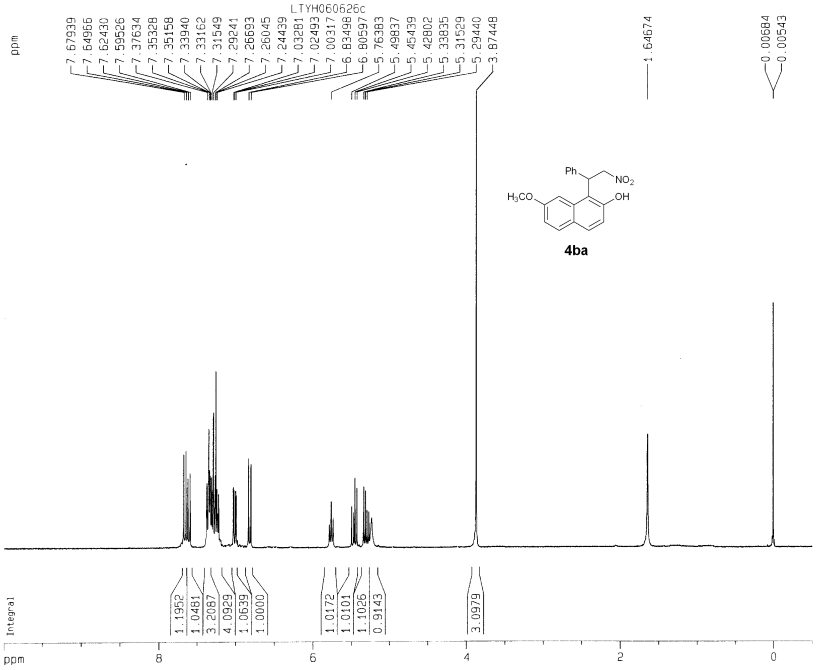
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69641 Hz/cm



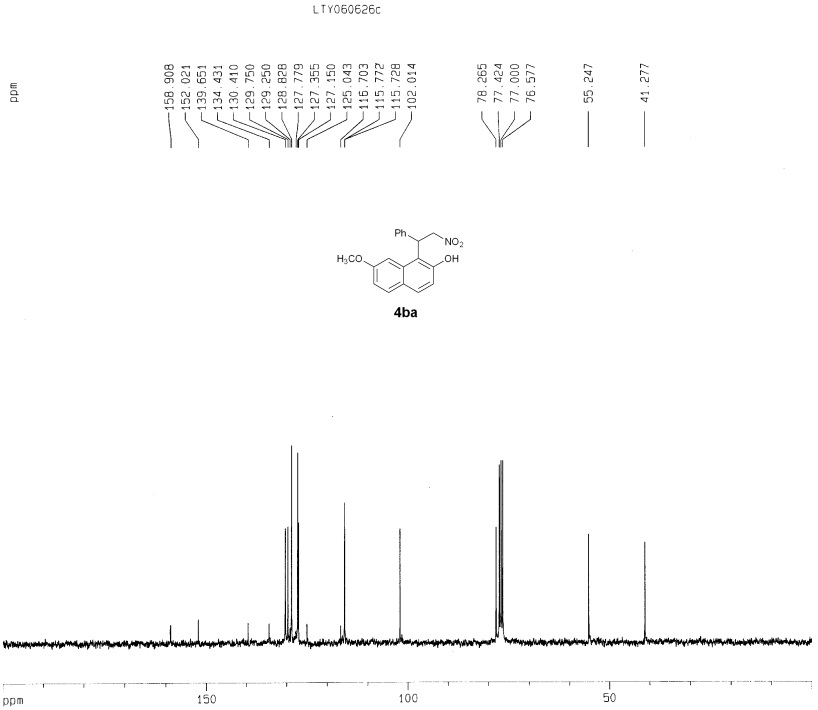
	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.342	5124091	49.64	374140	56.62
2	9.405	5198516	50.36	286676	43.38



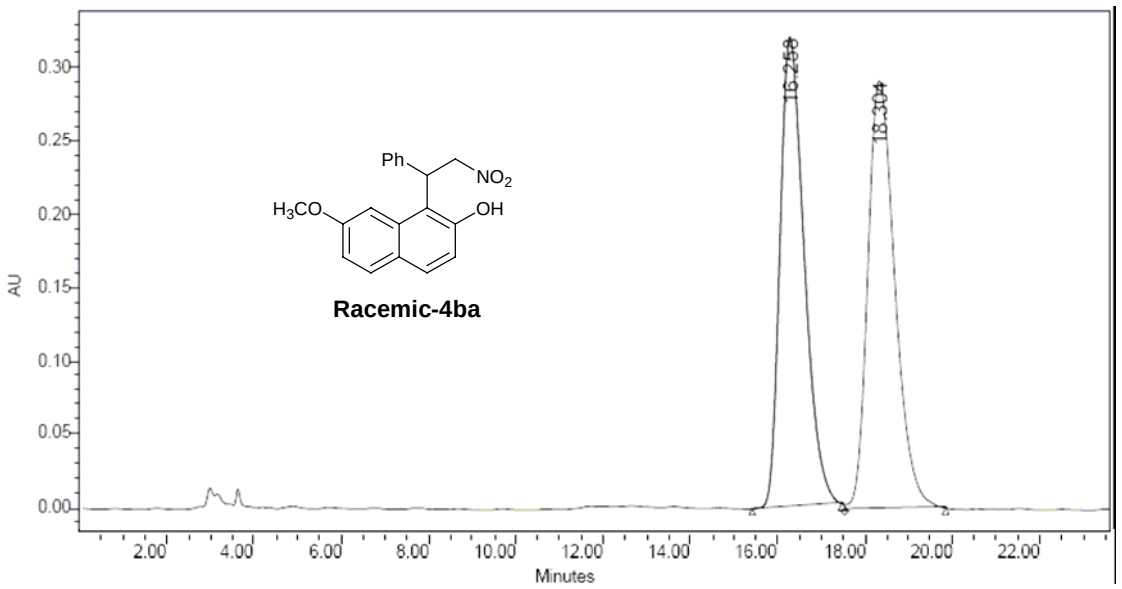
	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.245	1859109	96.77	145956	96.67
2	9.268	62052	3.23	5020	3.33




```
Current Data Parameters
NAME          cyc-2006-155
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_          20060626
Time               16.25
INSTRUM            av300
PROBHD   5 mm GNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           5995.204 Hz
FIDRES        0.182959 Hz
AQ            2.7329011 sec
RG                 512
DW              83.400 usec
DE               6.00 usec
TE              300.0 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                3.00 usec
PL1              -2.00 dB
SFO1        300.1320884 MHz

F2 - Processing parameters
SI                32768
SF          300.1300124 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                15.00 cm
F1P              10.000 ppm
F1             3001.30 Hz
F2P              -0.500 ppm
F2             -150.06 Hz
PPMCM          0.52500 ppm/cm
HZCM         157.56825 Hz/cm
```

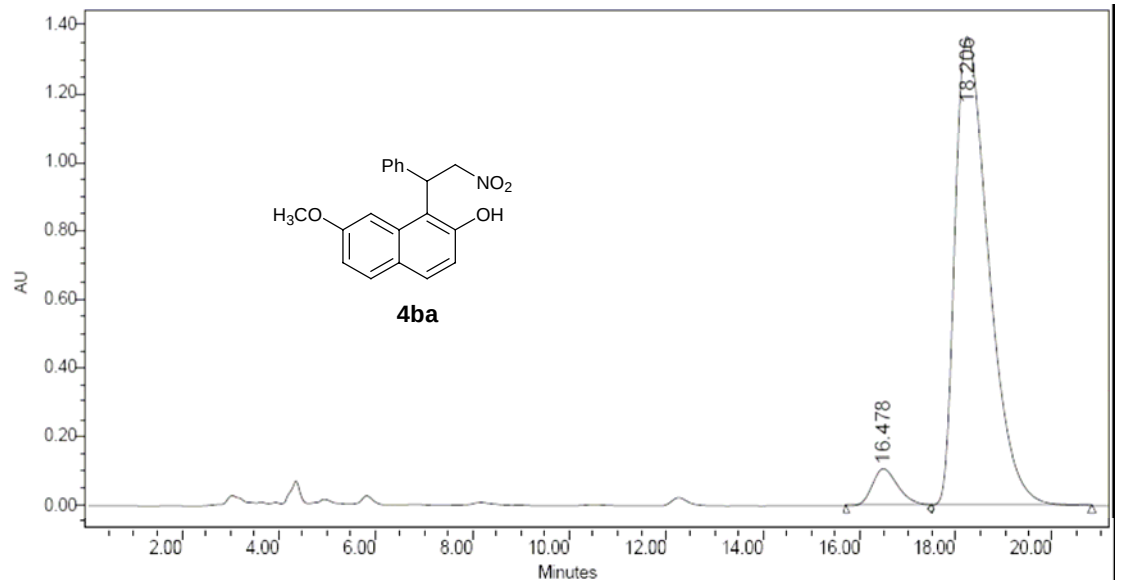



```
Current Data Parameters
NAME          cyc-2006-155
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_          20060629
Time               13.10
INSTRUM            av300
PROBHD   5 mm GNP 1H/13
PULPROG          zgpg
TD                65536
SOLVENT           CDCl3
NS                 1176
DS                    4
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                 8192
DW              22.050 usec
DE               6.00 usec
TE              300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                5.50 usec
PL1              -6.00 dB
SFO1        75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             17.70 dB
PL13             17.71 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677523 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 5.00 cm
F1P             200.005 ppm
F1            15093.93 Hz
F2P              -0.005 ppm
F2              -0.38 Hz
PPMCM         10.00050 ppm/cm
HZCM         754.71527 Hz/cm
```



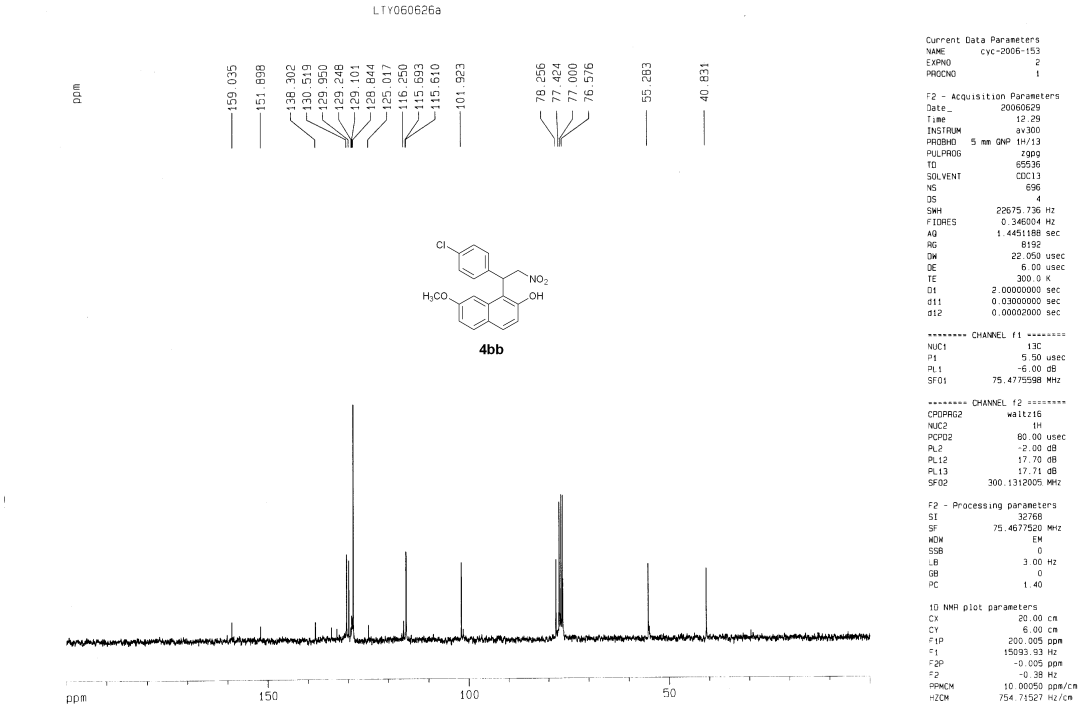
Racemic-4ba

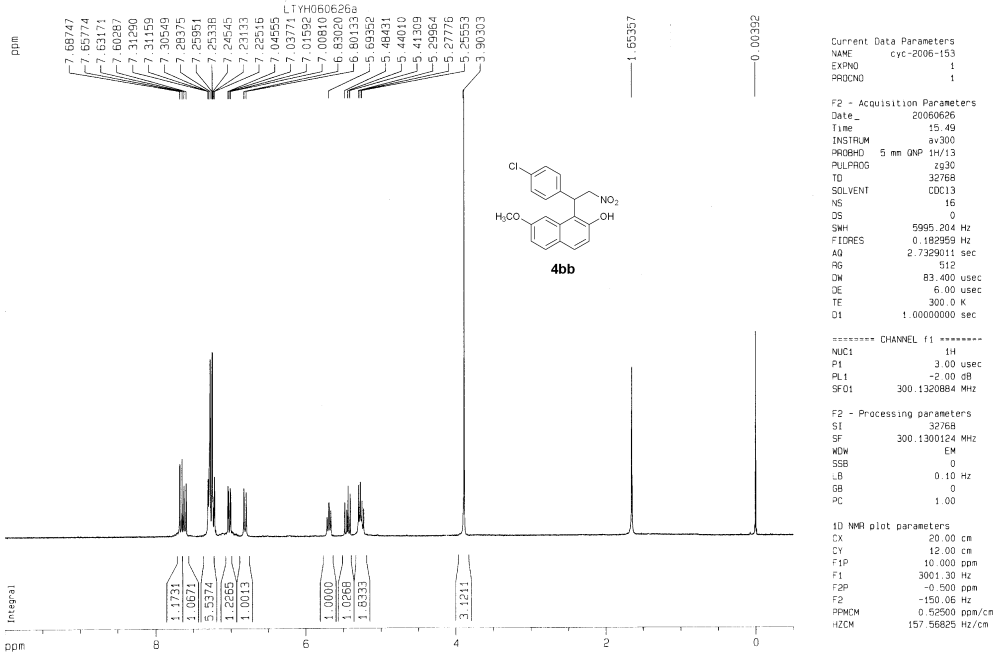
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	16.258	12350938	49.55	320475	52.32
2	18.304	12574205	50.45	292102	47.68

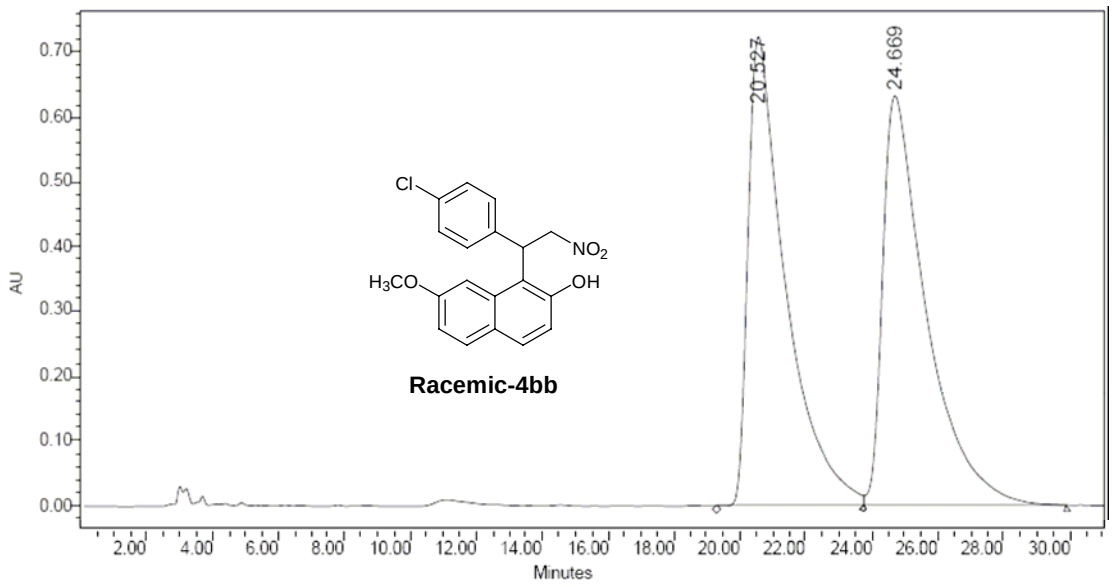


4ba

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	16.478	3232717	4.70	98606	6.74
2	18.206	65494007	95.30	1364339	93.26

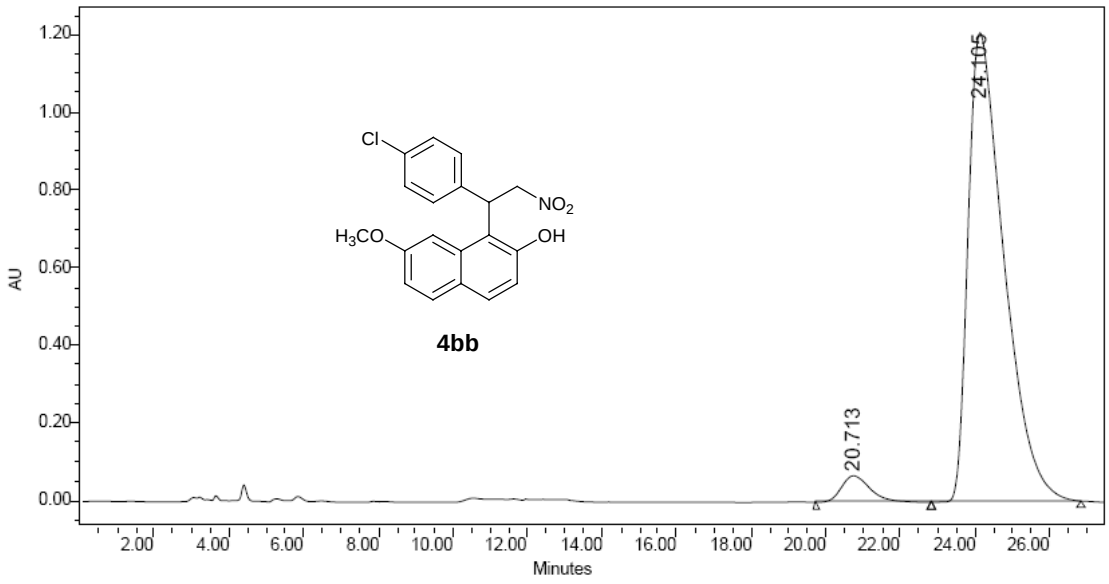

LTY060626a
159.035
151.898
138.302
130.519
129.950
129.248
129.101
128.844
125.017
116.250
115.693
115.610
101.923
78.256
77.424
77.000
76.576
55.283
40.831
ppm
Cl
NO2
H3CO
OH
4bb
150
100
50
Current Data Parameters
NAME cyc-2006-153
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060629
Time 12.29
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 696
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677520 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.005 ppm
F1 15093.93 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71527 Hz/cm


LTYH060626a
7.68747
7.65774
7.63171
7.60287
7.31290
7.31159
7.30549
7.28375
7.25951
7.25338
7.24545
7.23133
7.22516
7.04555
7.03771
7.01592
7.00810
6.83020
6.80133
5.69352
5.48431
5.44010
5.41309
5.29964
5.27776
5.25553
3.90303
1.65357
0.00392
ppm
Cl
NO2
H3CO
OH
4bb
Integral
1.1731
1.0671
5.5374
1.2265
1.0013
1.0000
1.0268
1.8333
3.1211
8
6
4
2
0
Current Data Parameters
NAME cyc-2006-153
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060626
Time 15.49
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320884 MHz
F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



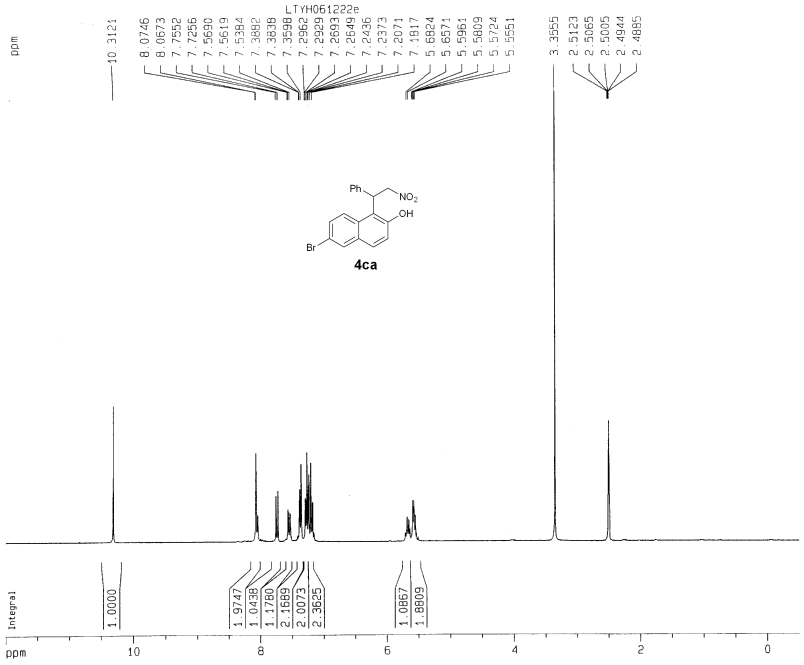
Racemic-4bb

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	20.527	57402003	49.63	723749	53.32
2	24.669	58258832	50.37	633599	46.68

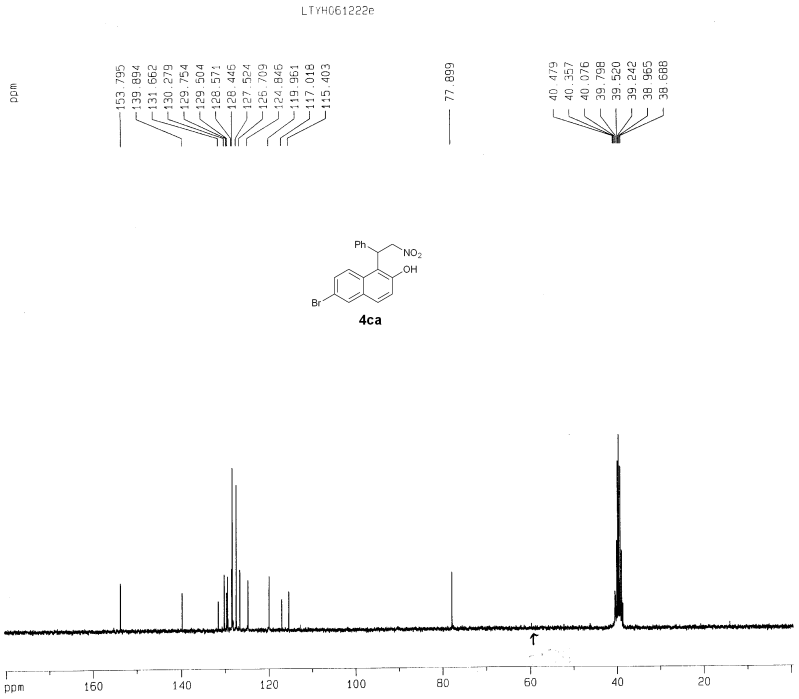


4bb

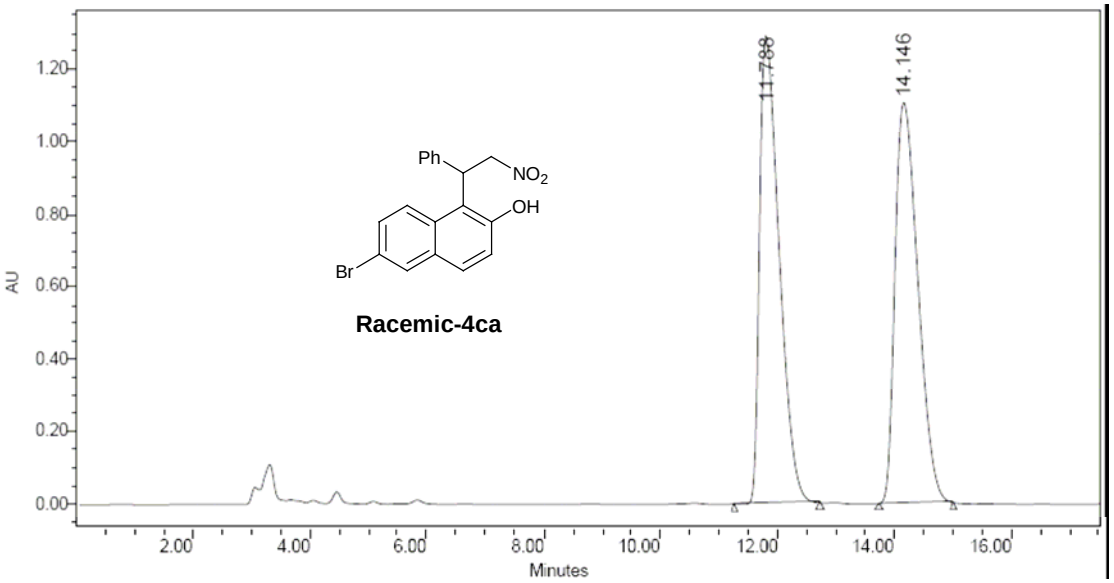
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	20.713	2267292	2.74	54635	4.33
2	24.105	80559117	97.26	1207296	95.67




```
Current Data Parameters
NAME        cyc-2006-288
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061222
Time               17.23
INSTRUM            av300
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT             DMSO
NS                    32
DS                     0
SWH             7485.030 Hz
FIDRES          0.228425 Hz
AQ              2.1889524 sec
RG                  1024
DW                66.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 3.00 usec
PL1               -2.00 dB
SFO1         300.1320882 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300013 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
CY                15.00 cm
F1P              12.000 ppm
F1             3601.56 Hz
F2P              -0.500 ppm
F2             -150.06 Hz
PPMCM          0.62500 ppm/cm
HZCM          187.58125 Hz/cm
```

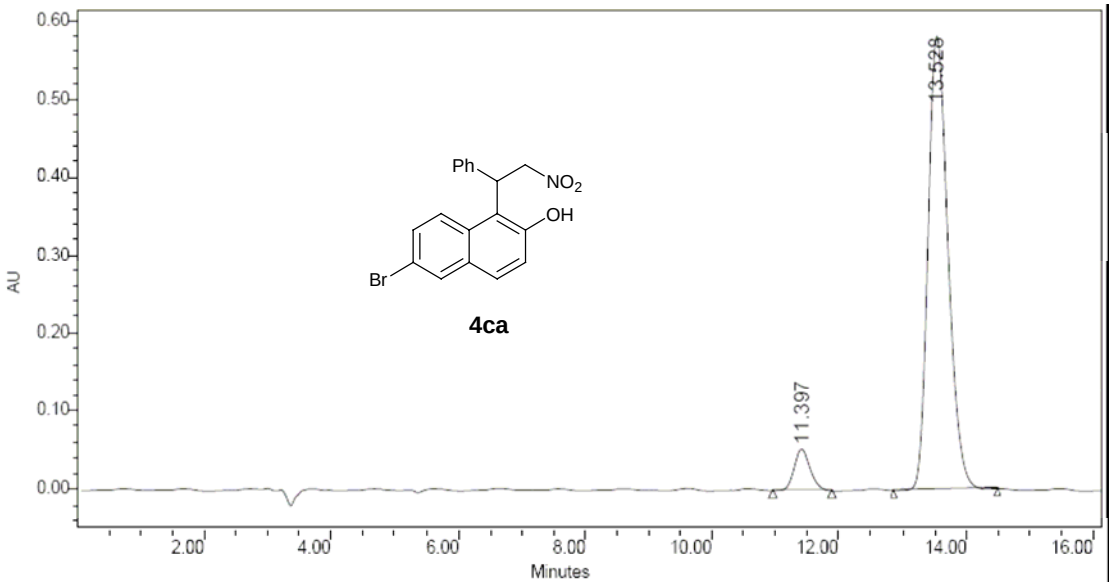



```
Current Data Parameters
NAME        cyc-2006-288
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061222
Time               20.08
INSTRUM            av300
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg
TD                65536
SOLVENT             DMSO
NS                   817
DS                     4
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ              1.4451188 sec
RG                  8192
DW                22.050 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -6.00 dB
SFO1         75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              17.70 dB
PL13              17.71 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 65536
SF          75.4677845 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                20.00 cm
CY                 5.00 cm
F1P             180.500 ppm
F1            13621.93 Hz
F2P              -0.500 ppm
F2             -37.73 Hz
PPMCM          9.05000 ppm/cm
HZCM          682.98340 Hz/cm
```



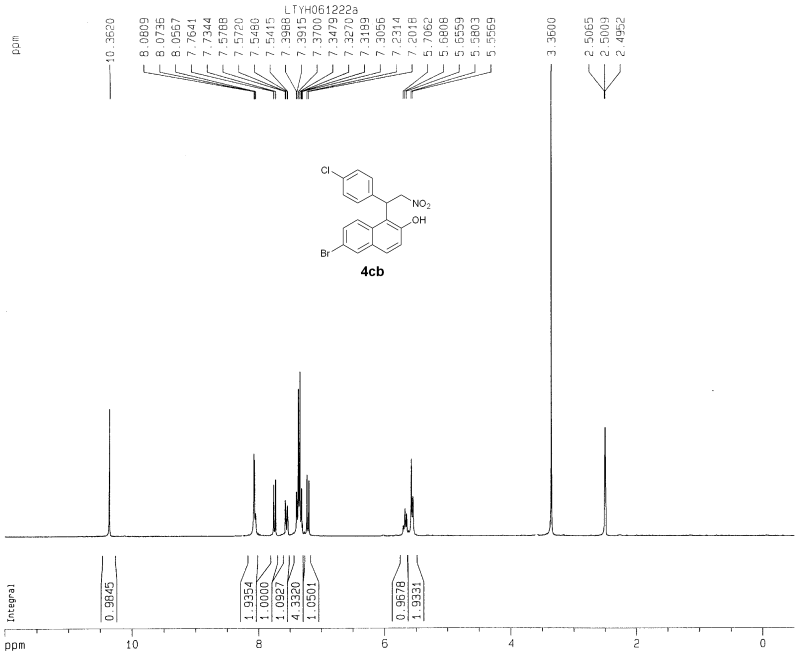
Racemic-4ca

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.788	30240332	50.05	1292196	53.85
2	14.146	30185857	49.95	1107609	46.15



4ca

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.397	680978	5.09	45615	7.29
2	13.528	12699825	94.91	580475	92.71



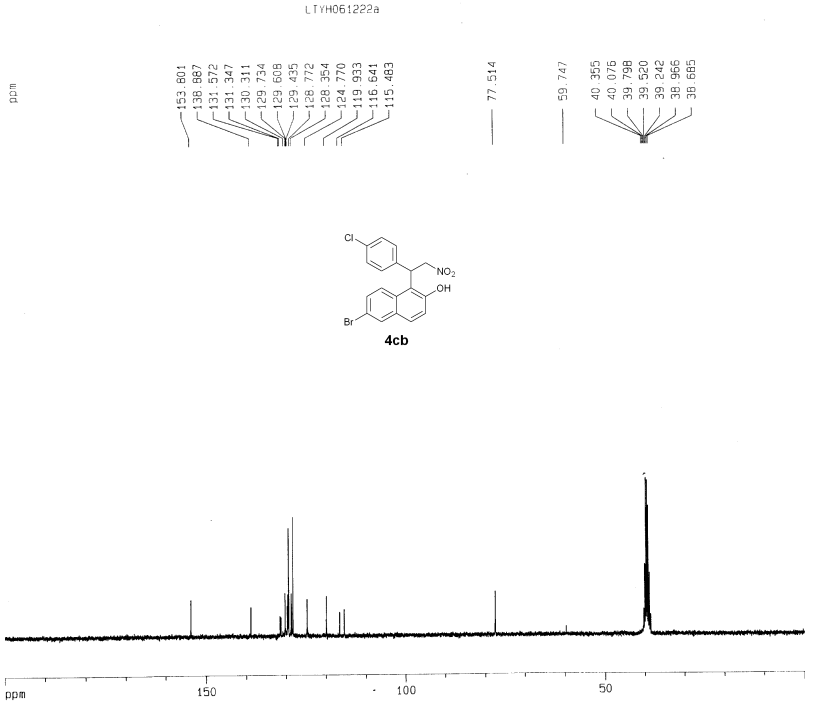
Current Data Parameters
NAME cyc-2006-286
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061222
Time 17.03
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 0
SWH 7485.030 Hz
FIDRES 0.228425 Hz
AQ 2.1889524 sec
RG 1024
DW 66.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300013 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 14.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.58125 Hz/cm



Current Data Parameters
NAME cyc-2006-286
EXPNO 2
PROCNO 1

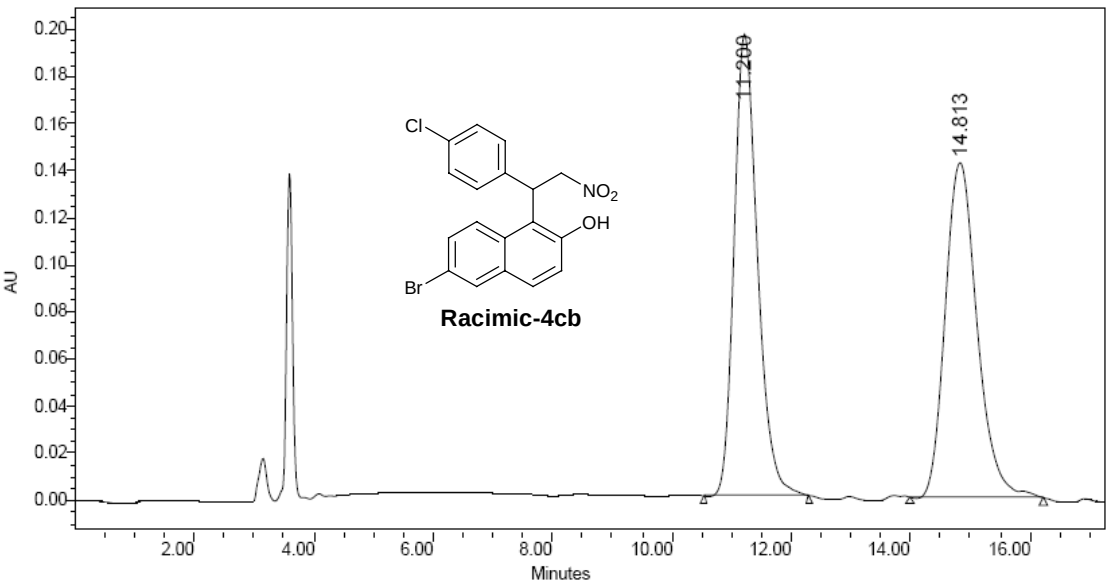
F2 - Acquisition Parameters
Date_ 20061222
Time 17.43
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 670
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

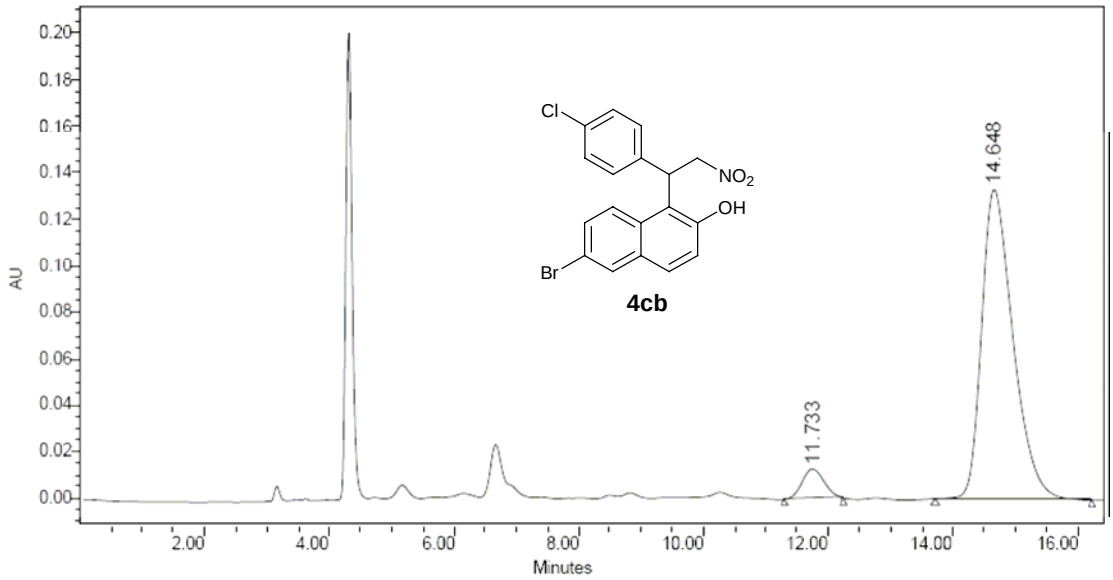
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677846 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

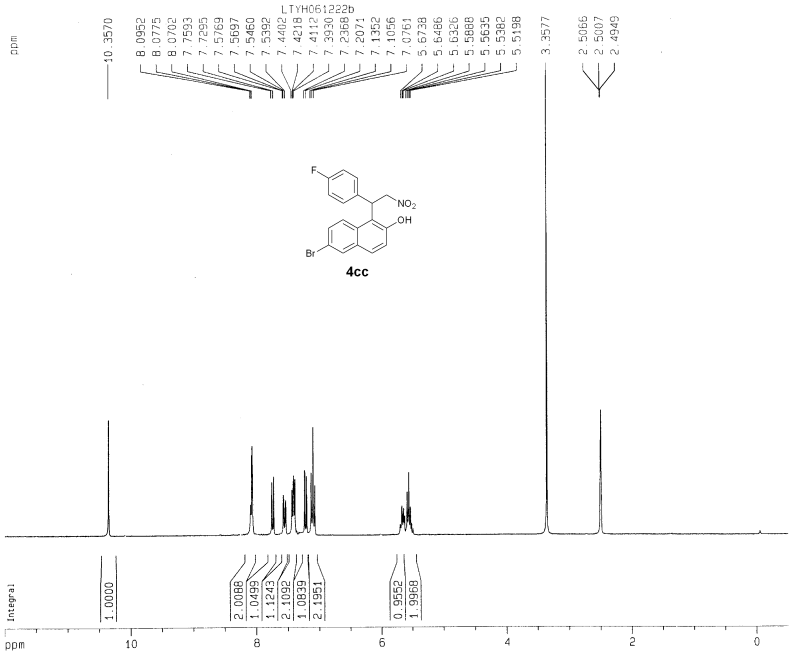
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 15093.56 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69672 Hz/cm



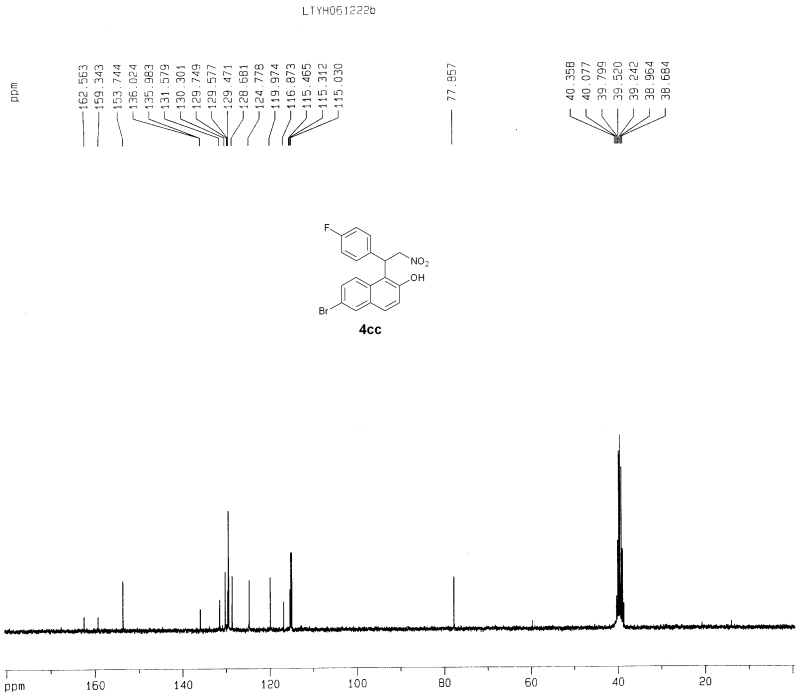
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.200	5351325	50.20	196314	57.96
2	14.813	5309066	49.80	142402	42.04



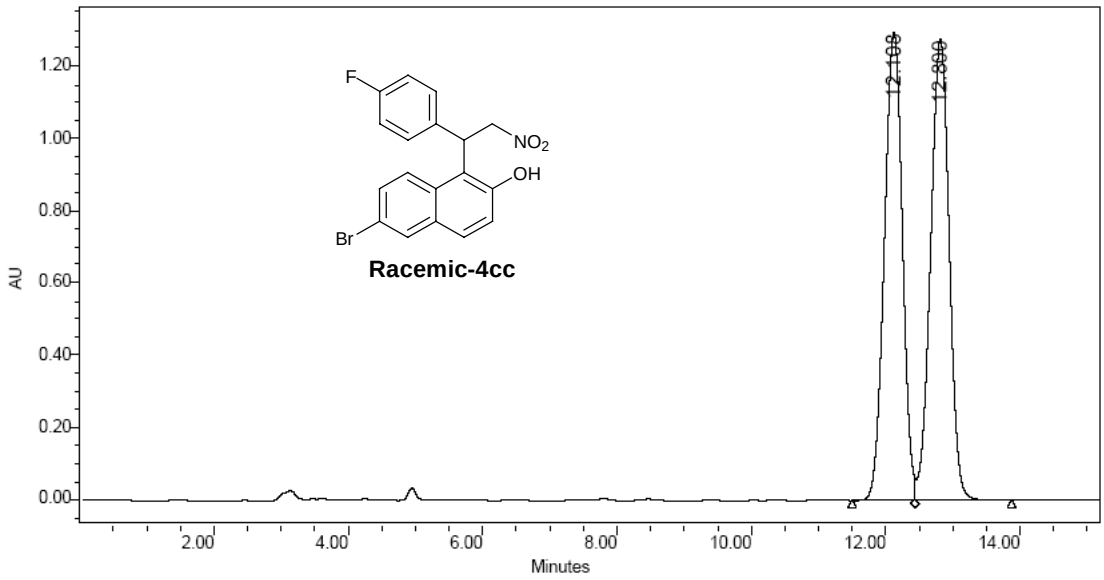
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.733	239472	4.98	11173	7.78
2	14.648	4568792	95.02	132428	92.22



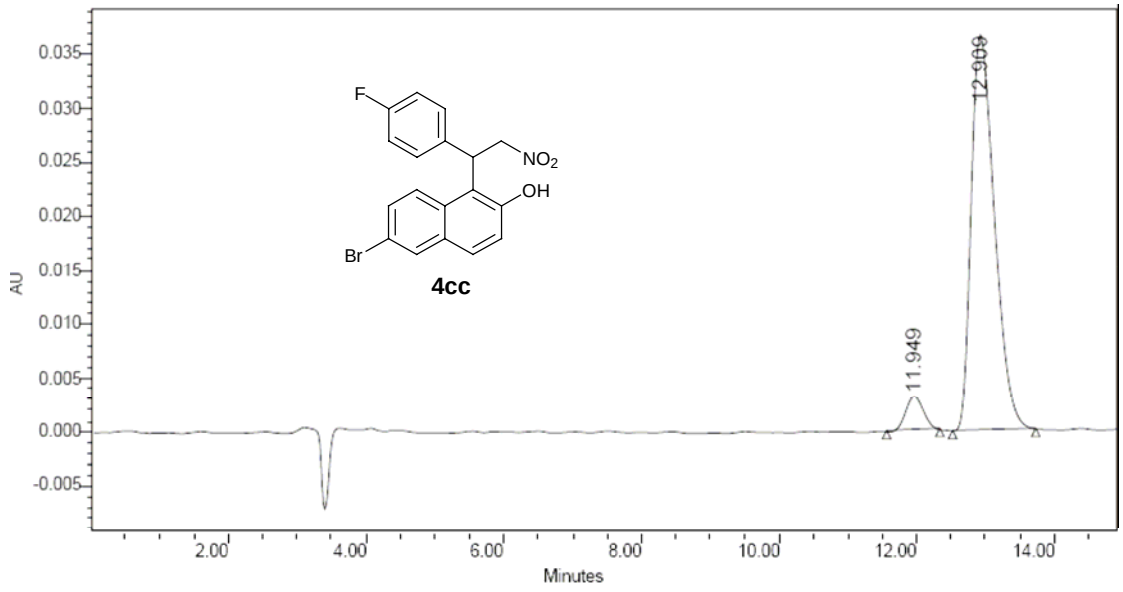

```
Current Data Parameters
NAME          cyc-2006-287
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20061222
Time                  17.14
INSTRUM               av300
PROBHD   5 mm GNP 1H/13
PULPROG               zg30
TD                   32768
SOLVENT              DMSO
NS                     32
DS                      0
SWH              7485.030 Hz
FIDRES           0.228425 Hz
AQ               2.1889524 sec
RG                    1024
DW                66.800 usec
DE                 6.00 usec
TE                 300.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 3.00 usec
PL1                -2.00 dB
SFO1         300.1320882 MHz

F2 - Processing parameters
SI                   32768
SF           300.1300013 MHz
WDW                    EM
SSB                     0
LB                 0.10 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  15.00 cm
F1P                12.000 ppm
F1               3601.56 Hz
F2P                -0.500 ppm
F2                -150.06 Hz
PPMCM           0.62500 ppm/cm
HZCM          187.58125 Hz/cm
```



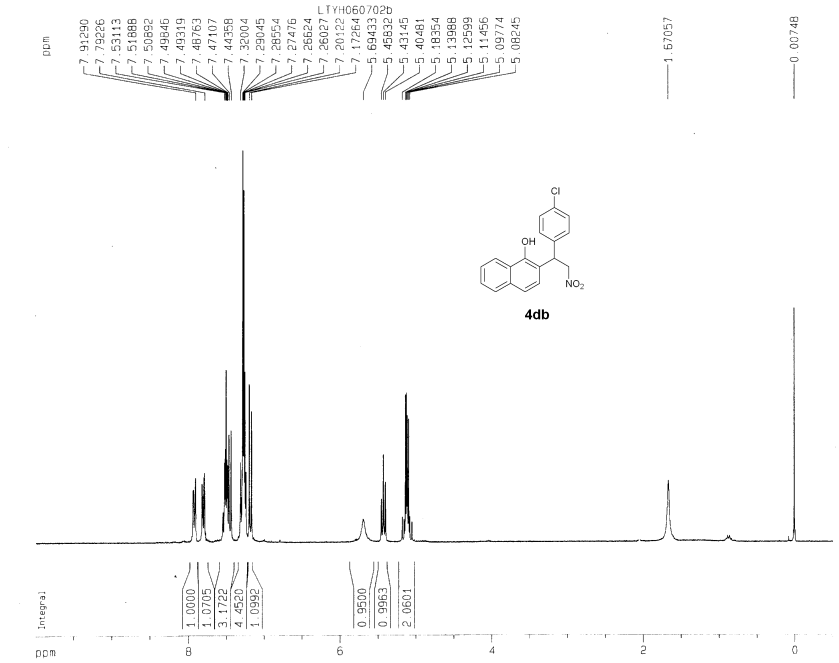

```
Current Data Parameters
NAME          cyc-2006-287
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20061222
Time                  18.26
INSTRUM               av300
PROBHD   5 mm GNP 1H/13
PULPROG             zgpg
TD                   65536
SOLVENT              DMSO
NS                     762
DS                       4
SWH              22675.736 Hz
FIDRES           0.346004 Hz
AQ               1.4451188 sec
RG                    8192
DW                22.050 usec
DE                 6.00 usec
TE                 300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

-------- CHANNEL f1 --------
NUC1                   13C
P1                 5.50 usec
PL1                -6.00 dB
SFO1         75.4775998 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2             80.00 usec
PL2                -2.00 dB
PL12               17.70 dB
PL13               17.71 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                   65536
SF           75.4677843 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   5.00 cm
F1P                180.500 ppm
F1              13621.93 Hz
F2P                -0.500 ppm
F2                 -37.73 Hz
PPMCM           9.05000 ppm/cm
HZCM          682.98340 Hz/cm
```



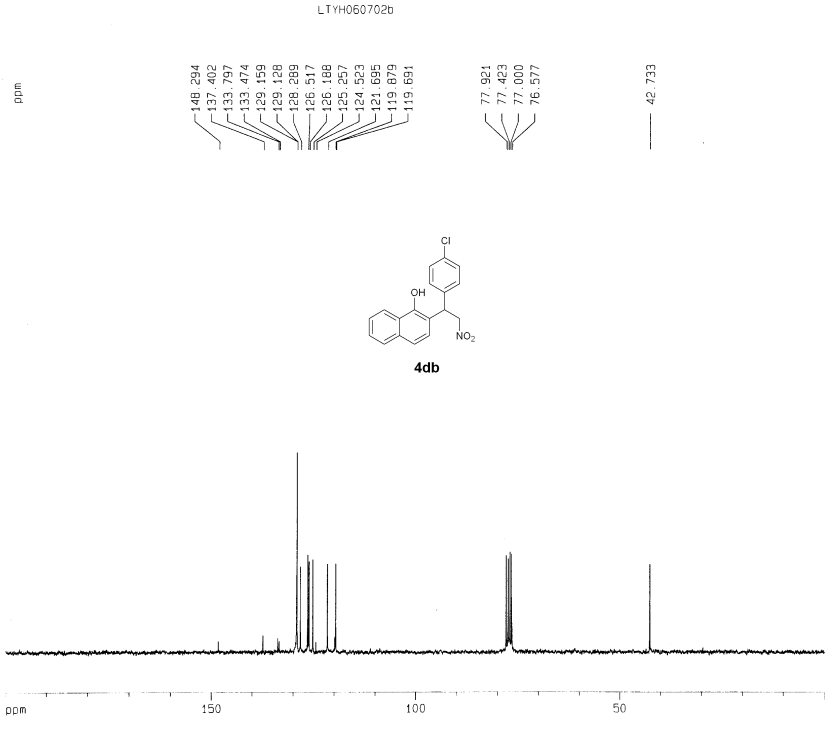
	RT (min)	Area (μV *sec)	% Area	Height (μV)	% Height
1	12.108	24195658	49.66	1297975	50.40
2	12.800	24529396	50.34	1277452	49.60



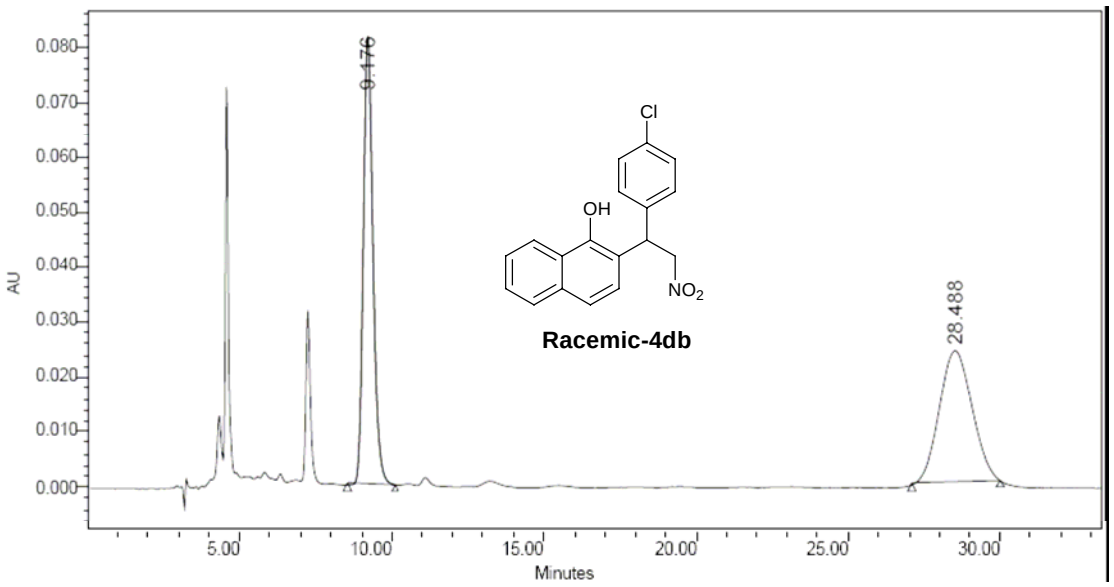
	RT (min)	Area (μV *sec)	% Area	Height (μV)	% Height
1	11.949	41803	4.61	2725	6.91
2	12.909	864357	95.39	36703	93.09




```
Current Data Parameters
NAME        cyc-2006-162
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060703
Time                9.49
INSTRUM            av300
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             5995.204 Hz
FIDRES          0.182959 Hz
AQ              2.7329011 sec
RG                   512
DW                83.400 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 3.00 usec
PL1                -2.00 dB
SFO1         300.1320884 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300124 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               10.000 ppm
F1              3001.30 Hz
F2P               -0.500 ppm
F2              -150.06 Hz
PPMCM          0.52500 ppm/cm
HZCM          157.56825 Hz/cm
```

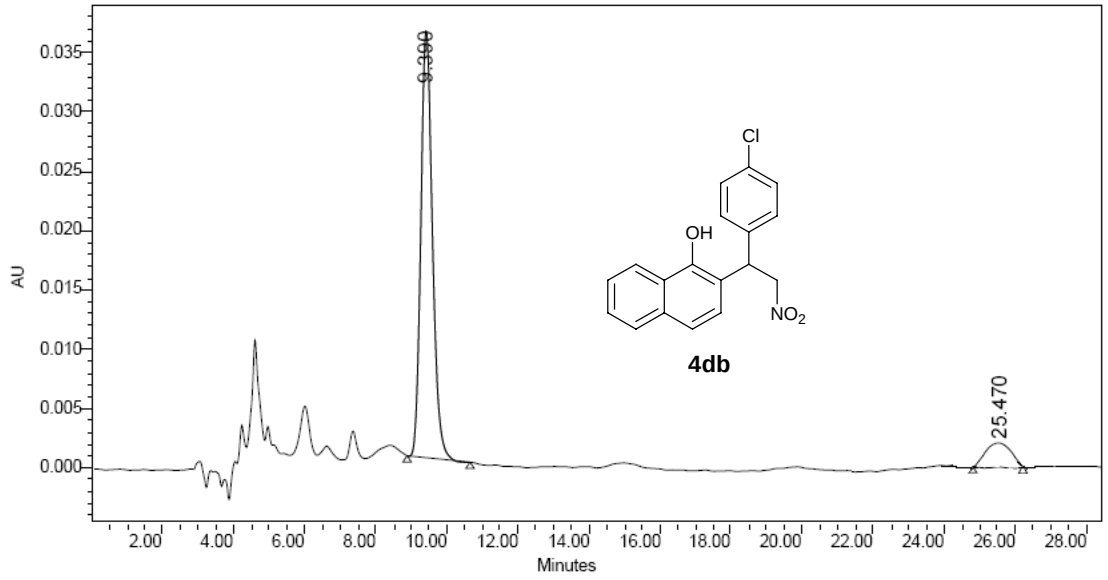



```
Current Data Parameters
NAME        cyc-2006-162
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060706
Time               10.19
INSTRUM            av300
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg
TD                 65536
SOLVENT            CDCl3
NS                   869
DS                     4
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ              1.4451188 sec
RG                  8192
DW                22.050 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 5.50 usec
PL1                -6.00 dB
SFO1         75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                -2.00 dB
PL12               17.70 dB
PL13               17.71 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF          75.4677525 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  5.00 cm
F1P              200.000 ppm
F1              15093.55 Hz
F2P               -0.005 ppm
F2                -0.38 Hz
PPMCM          10.00025 ppm/cm
HZCM          754.69641 Hz/cm
```



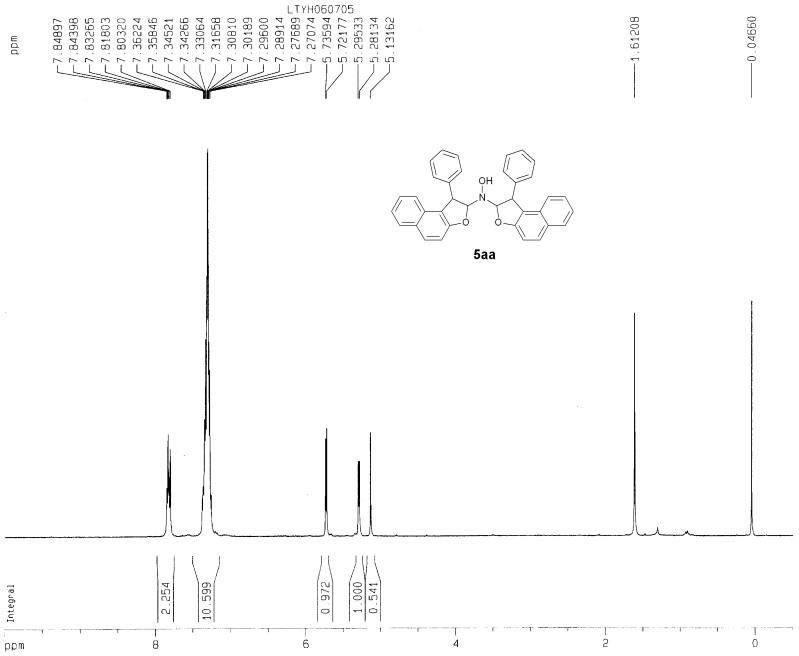
Racemic-4db

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.176	1799821	49.97	81340	77.22
2	28.488	1802047	50.03	23998	22.78



4db

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.390	815364	90.15	35992	95.05
2	25.470	89137	9.85	1873	4.95



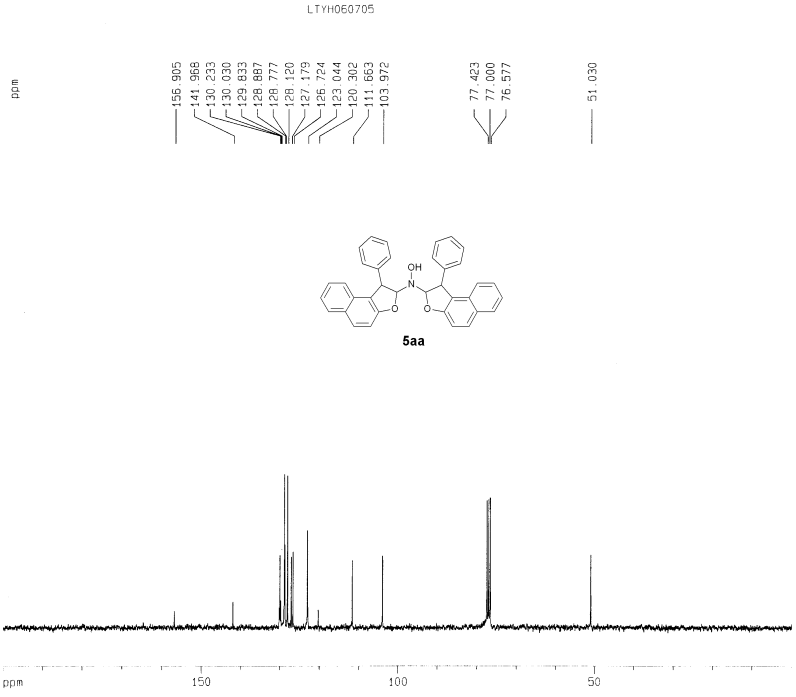
Current Data Parameters
NAME cyc-2006-167
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060705
Time 16.10
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300017 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-167
EXPNO 2
PROCNO 1

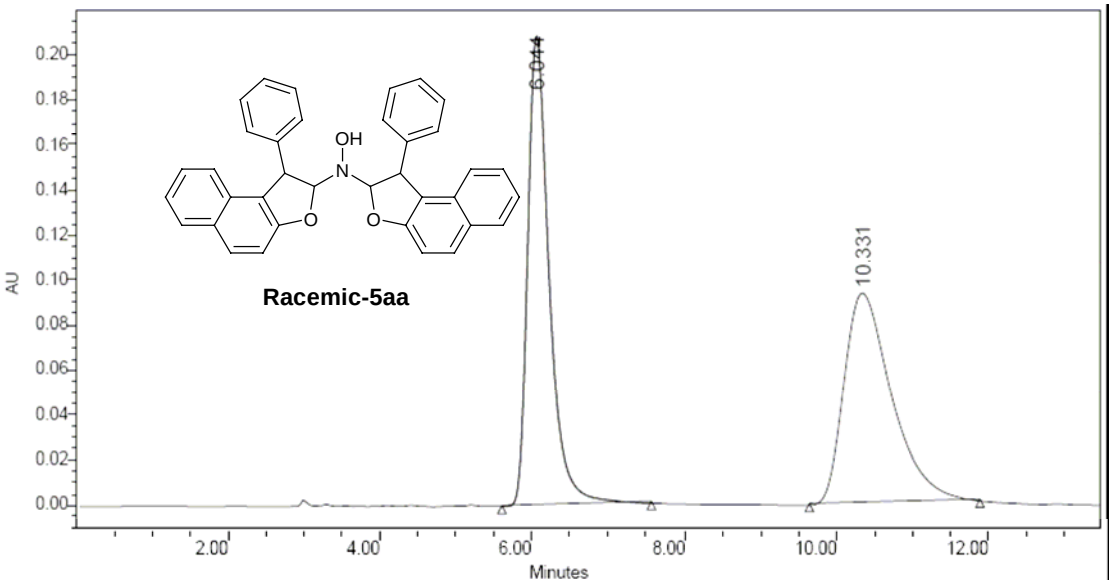
F2 - Acquisition Parameters
Date_ 20060705
Time 21.01
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 408
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451088 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

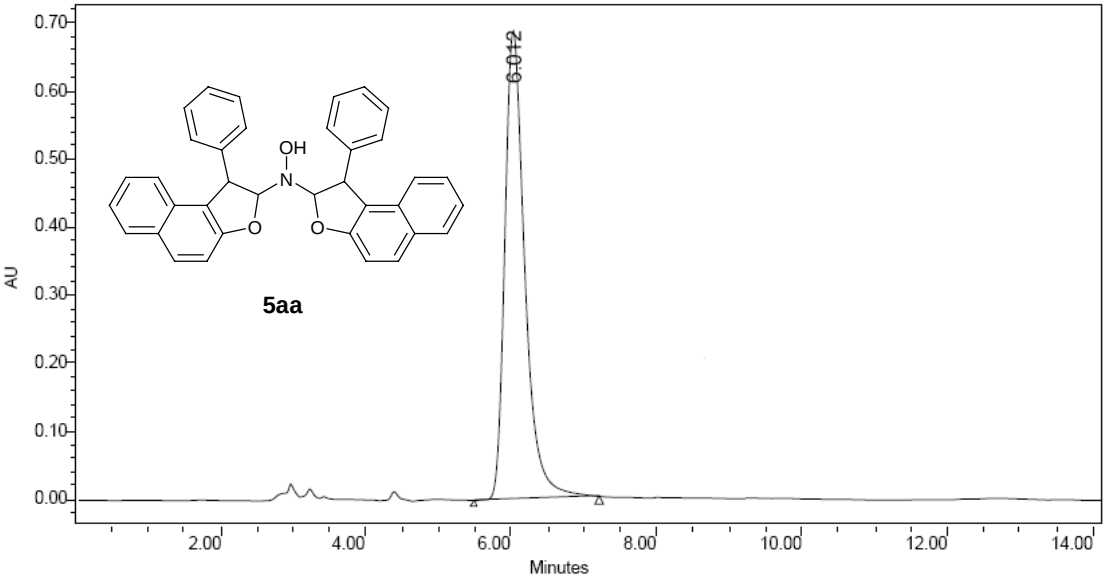
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677519 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

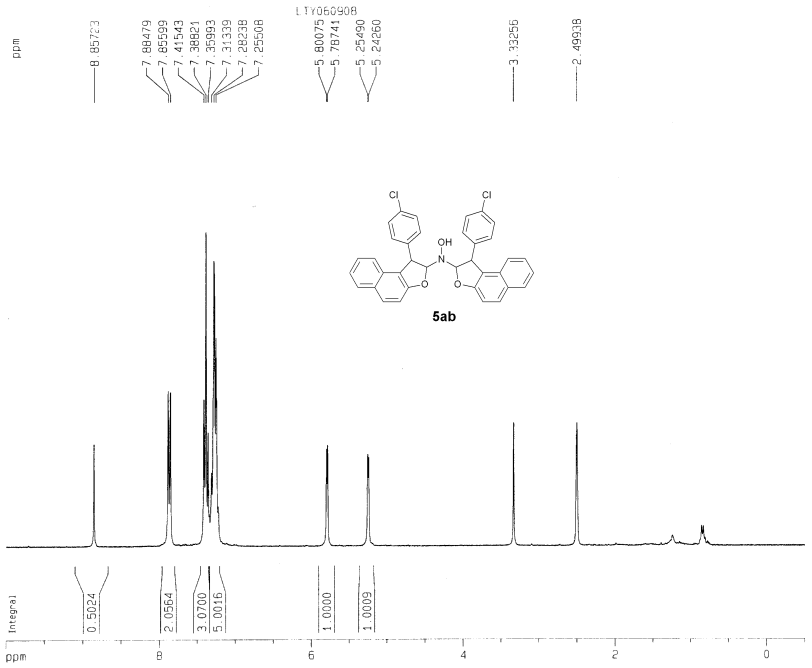
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.005 ppm
F1 15093.93 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71527 Hz/cm



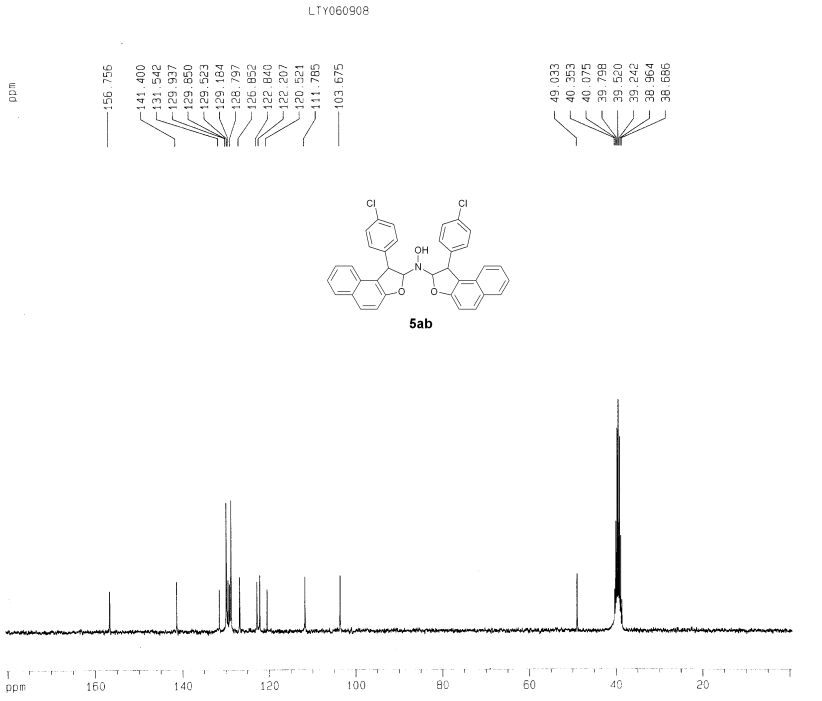
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.044	4098489	50.01	208360	69.02
2	10.331	4096162	49.99	93507	30.98



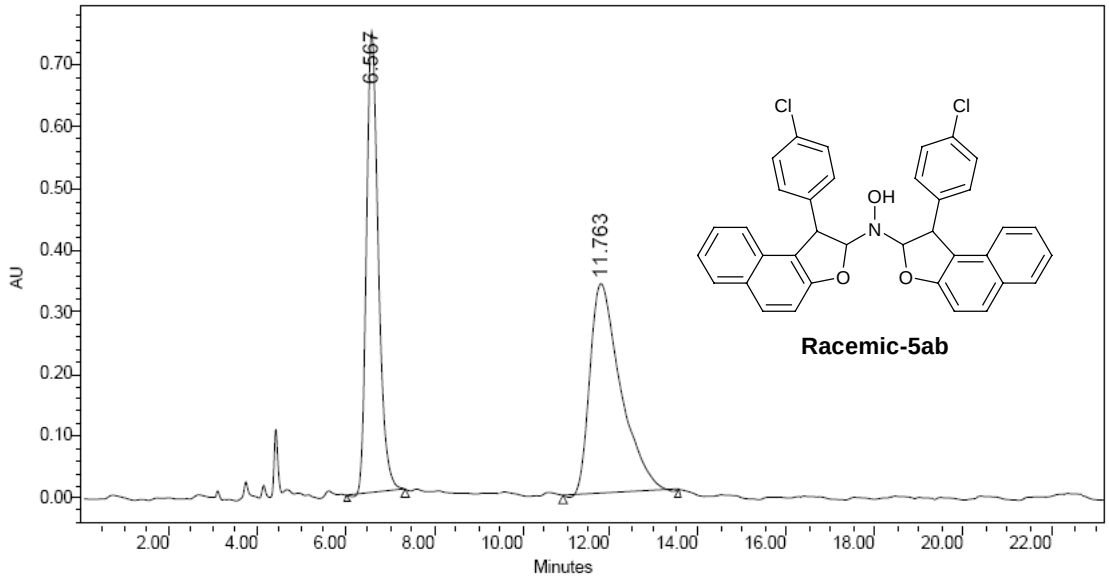
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.012	13135652	100.00	688513	100.00



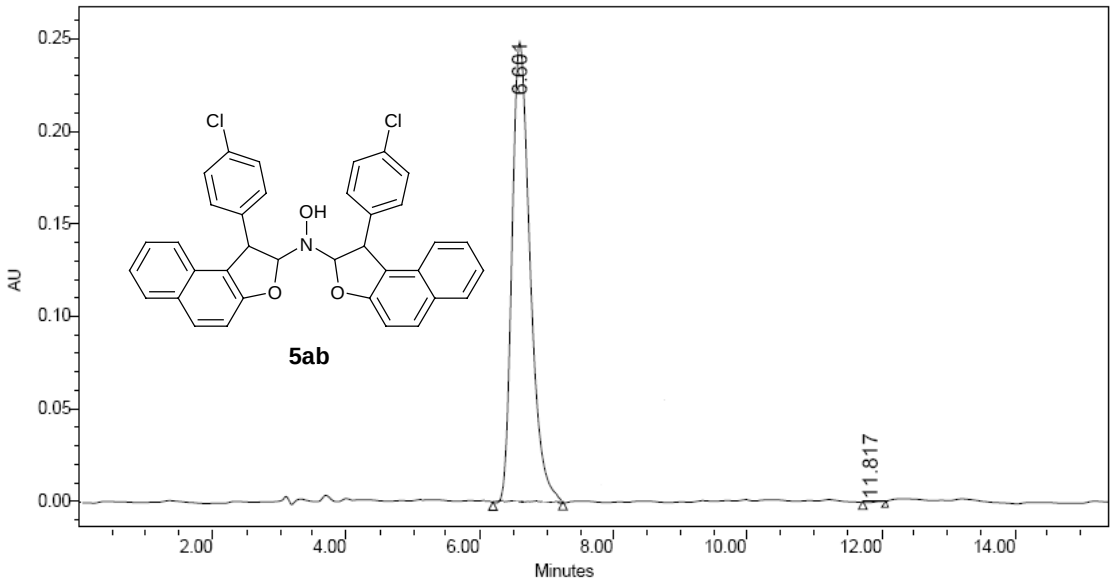

```
Current Data Parameters
NAME          cyc-2006-210
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060908
Time                  16.41
INSTRUM               av300
PROBHD   5 mm GNP 1H/13
PULPROG               zg30
TD                   32768
SOLVENT               DMSO
NS                      20
DS                       0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                     512
DW                  83.400 usec
DE                   6.00 usec
TE                  300.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    3.00 usec
PL1                  -2.00 dB
SFO1           300.1320882 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300026 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                    8.00 cm
F1P                 10.000 ppm
F1                3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM              0.52500 ppm/cm
HZCM             157.56825 Hz/cm
```



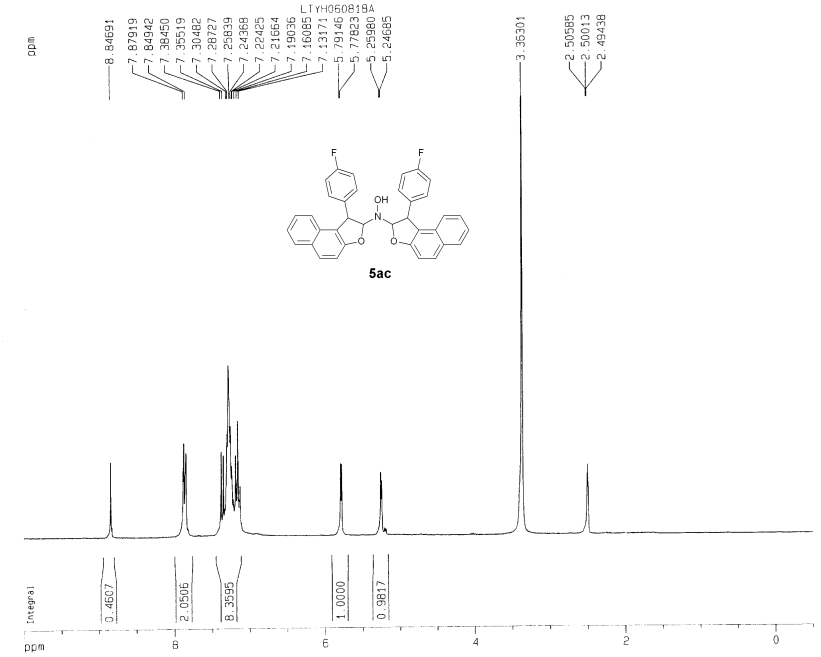

```
Current Data Parameters
NAME          cyc-2006-210
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060911
Time                   9.30
INSTRUM               av300
PROBHD   5 mm GNP 1H/13
PULPROG             zgpg
TD                   65536
SOLVENT               DMSO
NS                     466
DS                       4
SWH               22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                     8192
DW                  22.050 usec
DE                   6.00 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    5.50 usec
PL1                   -6.00 dB
SFO1           75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 17.70 dB
PL13                 17.71 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677848 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    6.00 cm
F1P                180.500 ppm
F1               13621.93 Hz
F2P                 -0.500 ppm
F2                 -37.73 Hz
PPMCM              9.05000 ppm/cm
HZCM             682.98340 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.567	14089440	49.70	747273	69.72
2	11.763	14262165	50.30	324608	30.28



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.601	4573572	99.97	246985	99.92
2	11.817	1596	0.03	-205	0.08



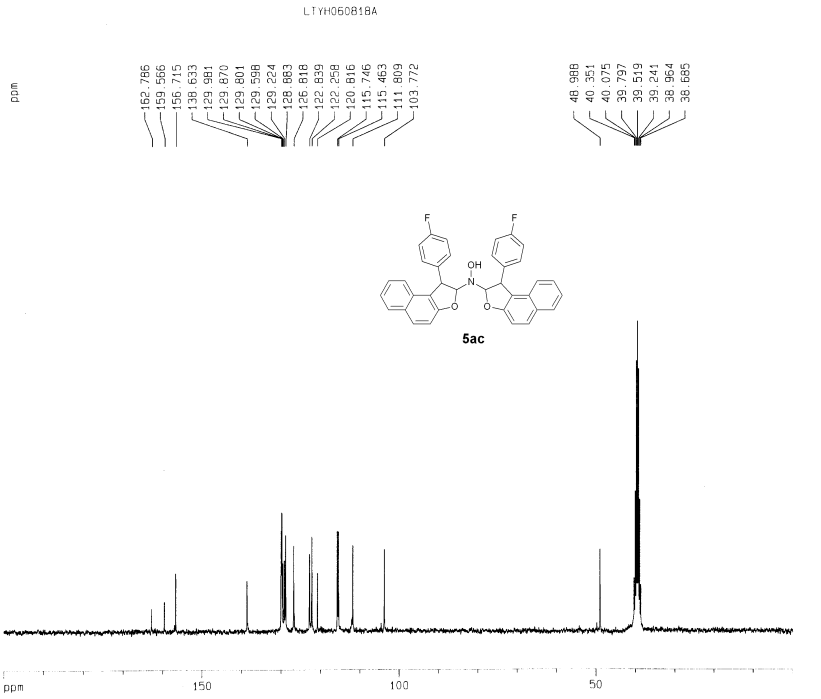
Current Data Parameters
NAME cyc-2006-196
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060818
Time 18.29
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300015 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 35.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-196
EXPNO 2
PROCNO 1

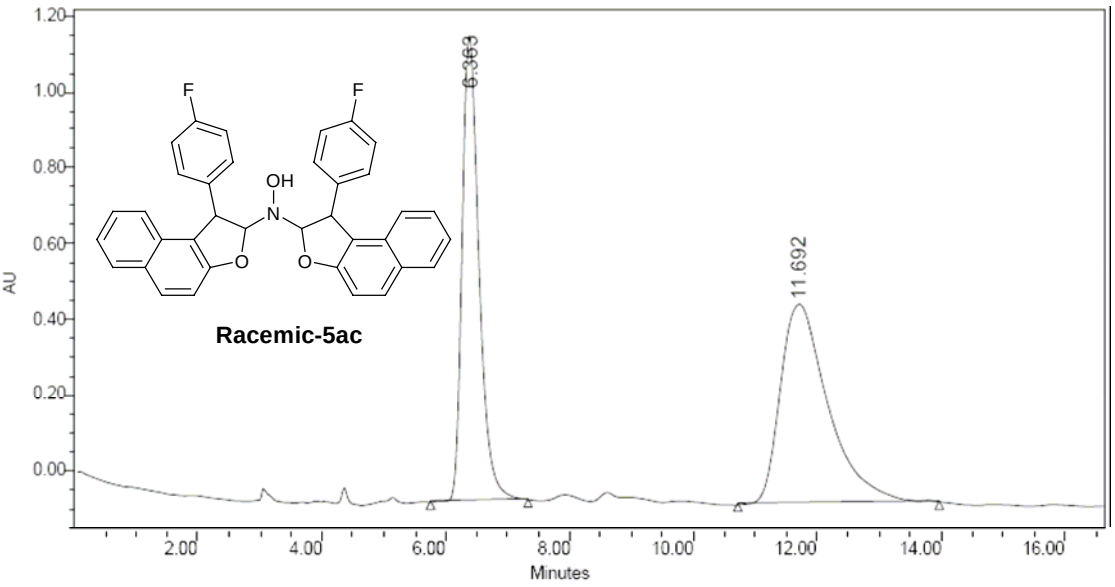
F2 - Acquisition Parameters
Date_ 20060821
Time 9.02
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 922
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz

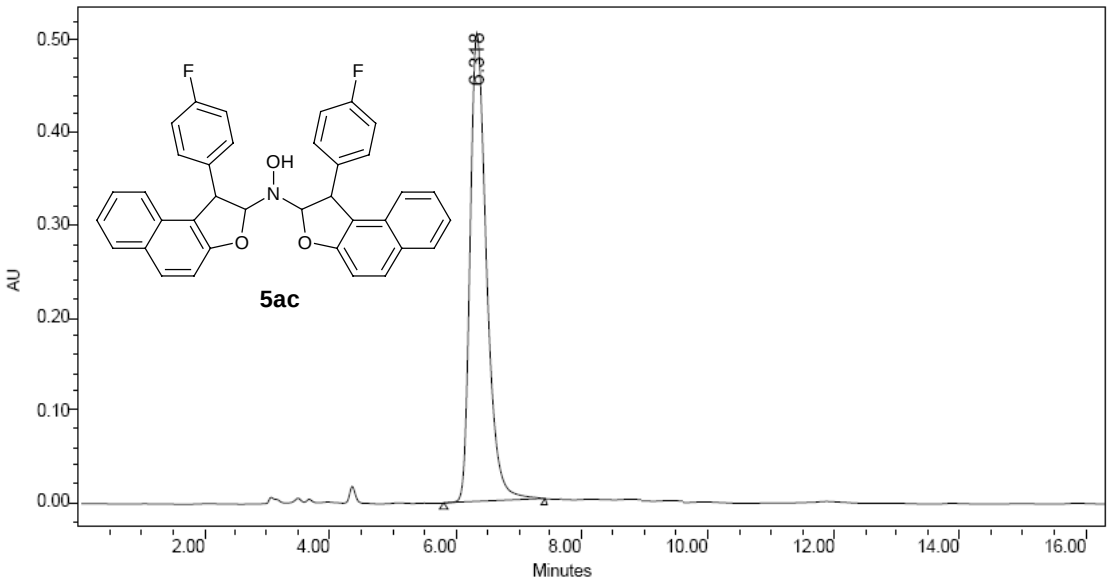
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677825 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

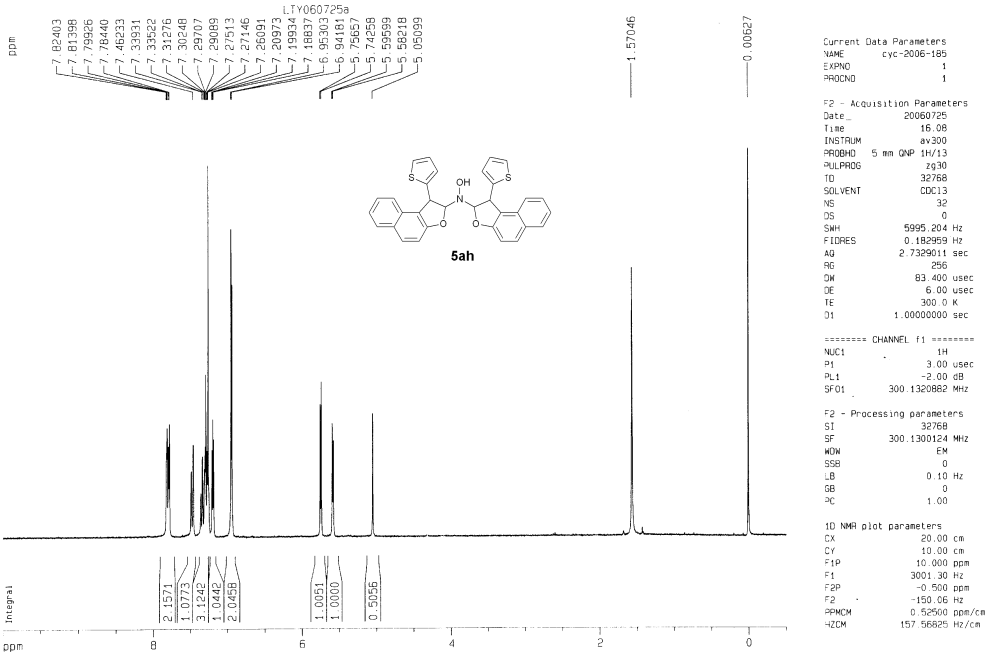
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 200.005 ppm
F1 15093.94 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00050 ppm/cm
HZCM 754.71558 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.363	23235582	49.75	1225854	71.23
2	11.692	23466088	50.25	495134	28.77



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.318	9194414	100.00	506795	100.00



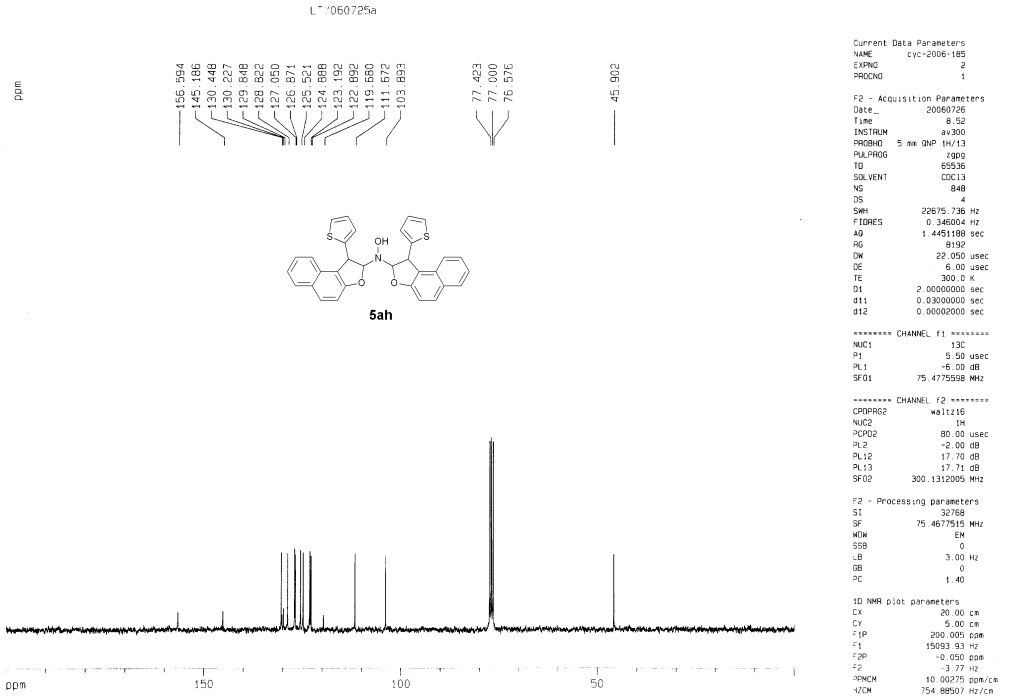
Current Data Parameters
NAME cyc-2006-185
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060725
Time 16.08
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-185
EXPNO 2
PROCNO 1

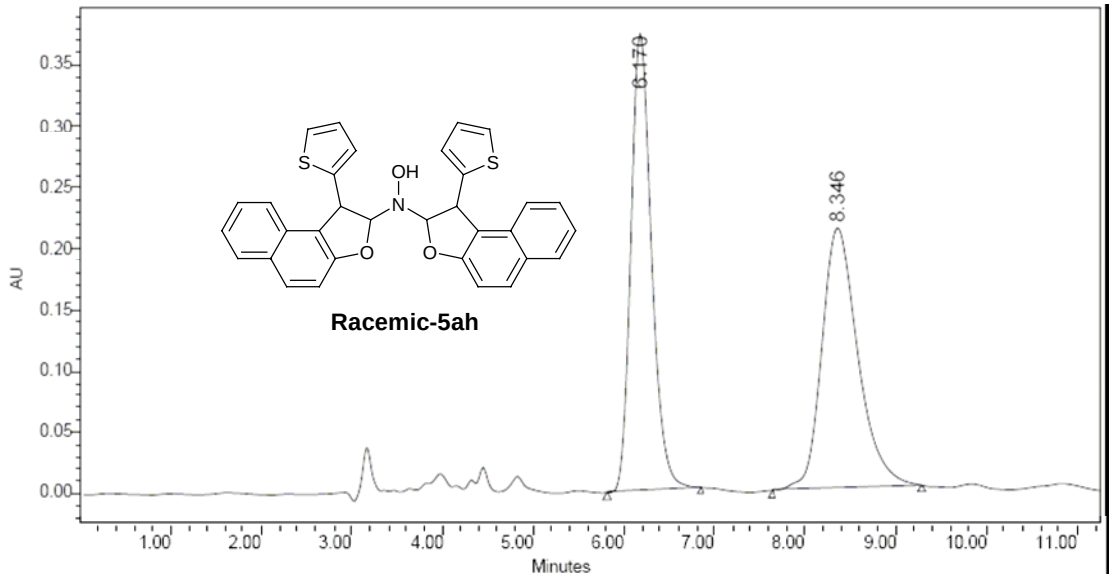
F2 - Acquisition Parameters
Date_ 20060726
Time 8.52
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 848
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

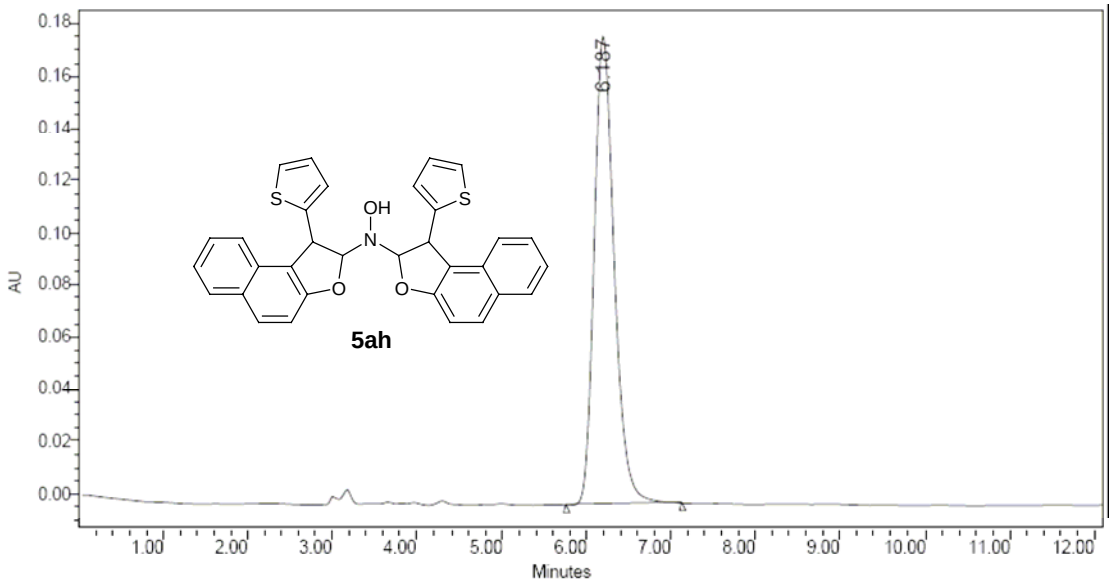
F2 - Processing parameters
SI 32768
SF 75.4677515 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.005 ppm
F1 15093.93 Hz
F2P -0.050 ppm
F2 -3.77 Hz
PPMCM 10.00275 ppm/cm
HZCM 754.88507 Hz/cm



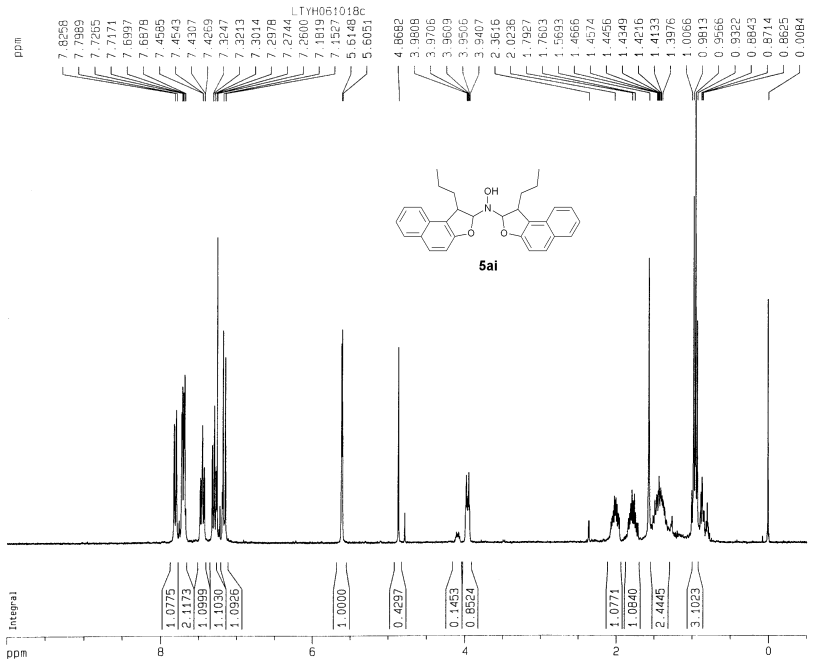
Racemic-5ah

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.170	5905723	50.10	373507	63.70
2	8.346	5881499	49.90	212850	36.30



5ah

	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	6.187	2952881	100.00	179203	100.00



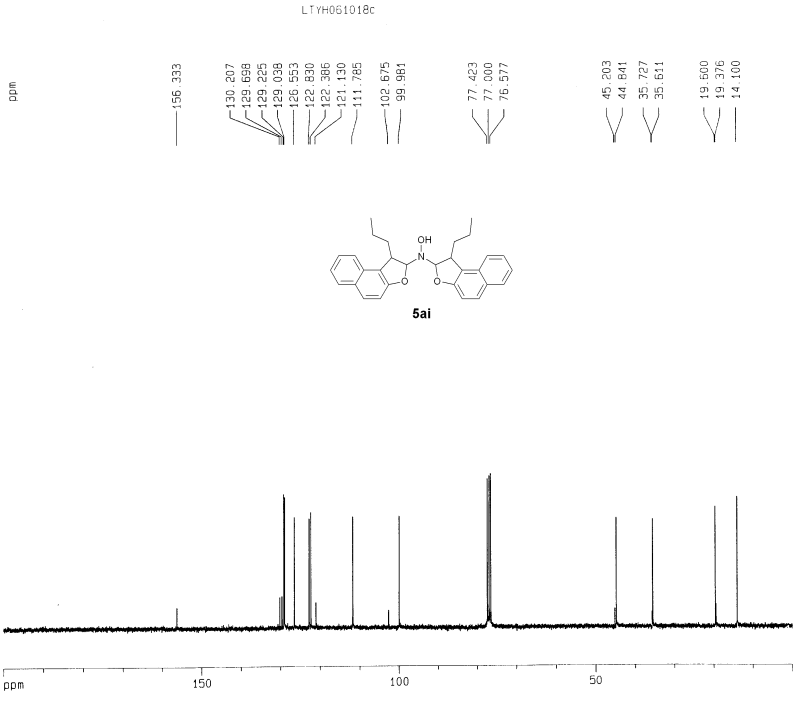
Current Data Parameters
NAME cyc-2006-233
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061018
Time 16.54
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm



Current Data Parameters
NAME cyc-2006-233
EXPNO 2
PROCNO 1

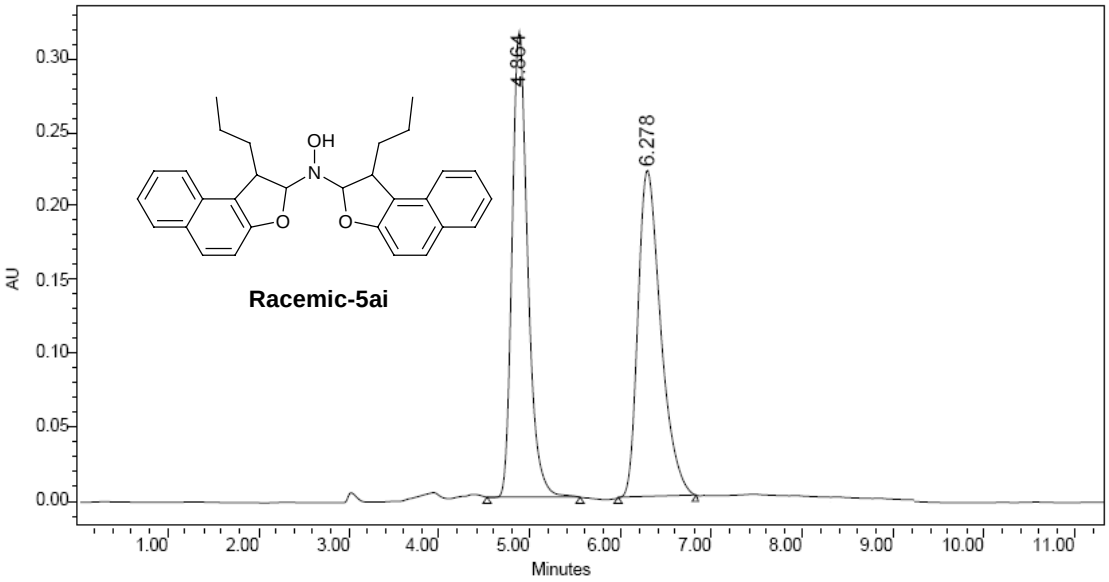
F2 - Acquisition Parameters
Date_ 20061019
Time 12.43
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1870
DS 4
SWH 21097.047 Hz
FIDRES 0.321915 Hz
AQ 1.5532532 sec
RG 8192
DW 23.700 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775998 MHz

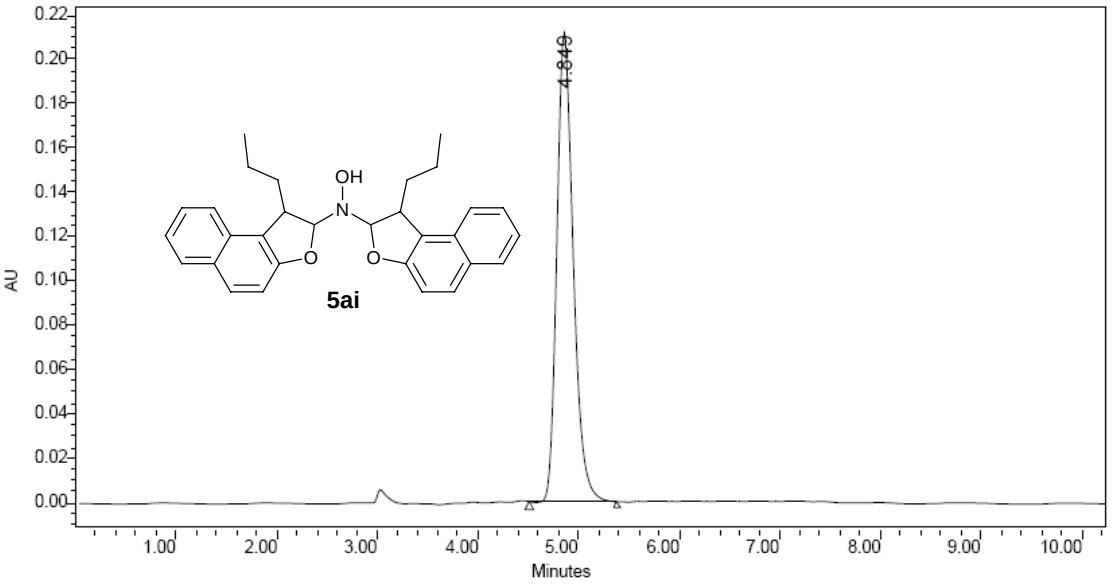
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677514 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

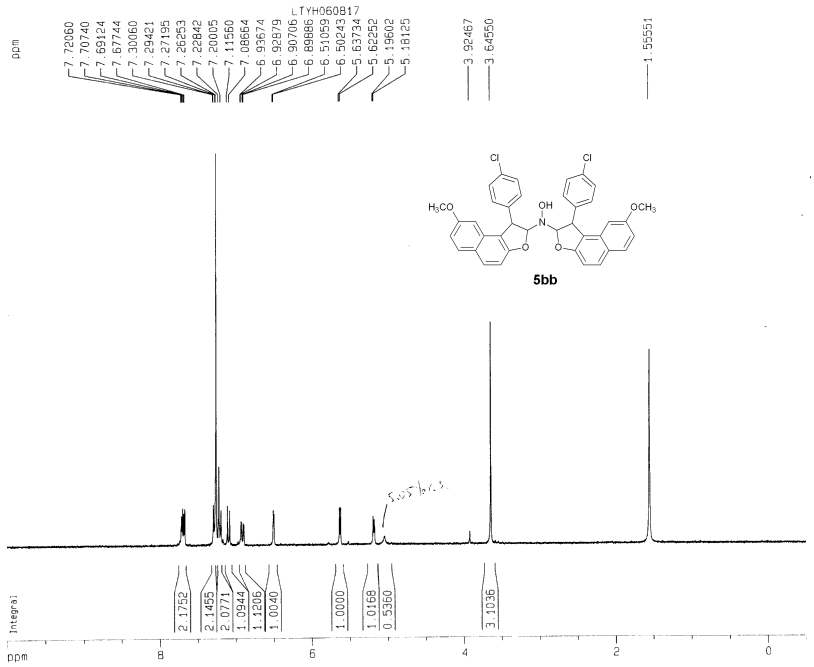
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69641 Hz/cm



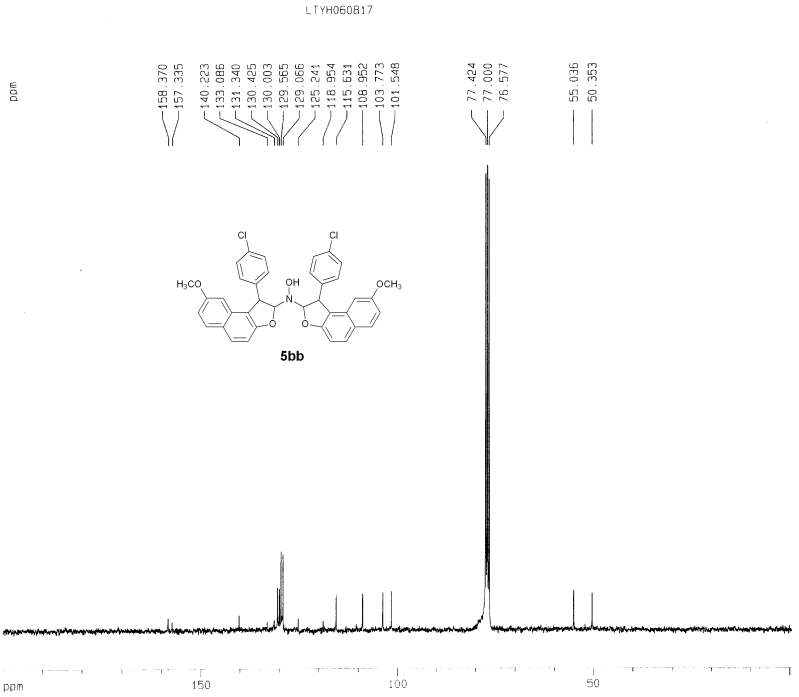
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	4.864	3788454	49.54	315653	58.77
2	6.278	3859146	50.46	221479	41.23



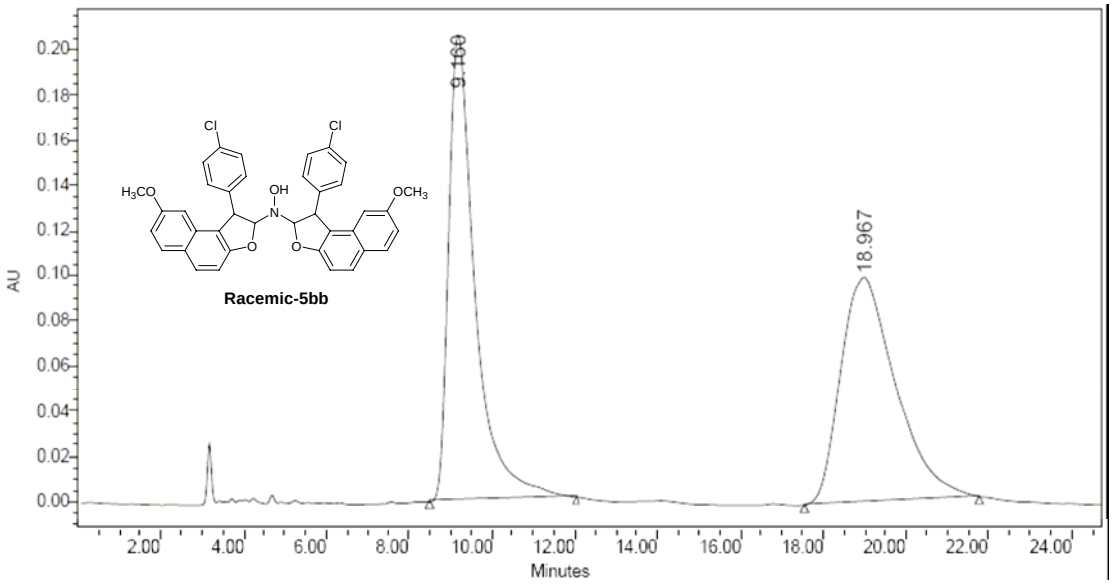
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	4.849	2402481	100.00	211380	100.00



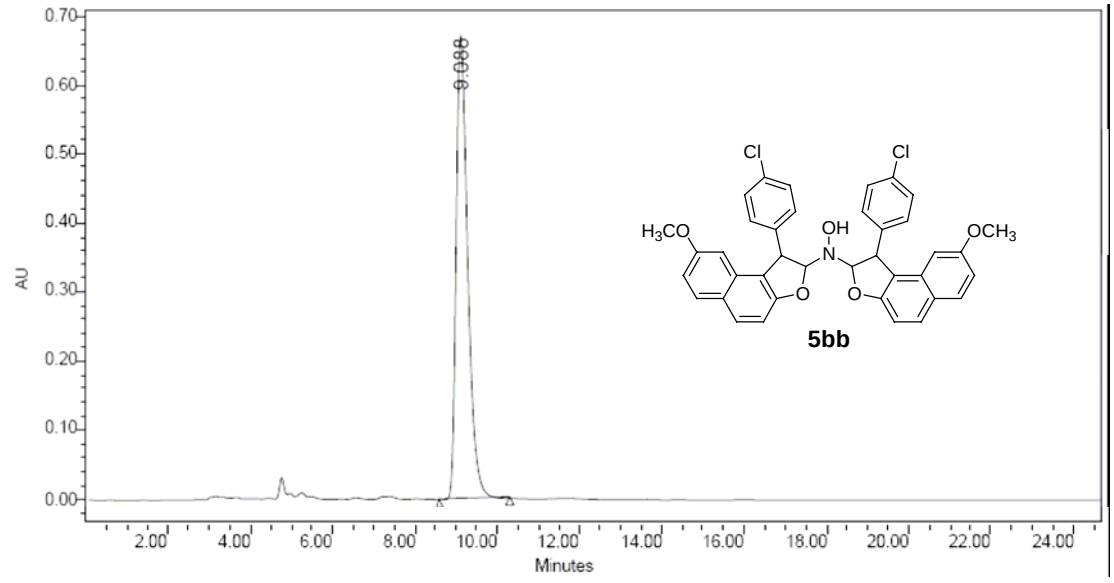

```
Current Data Parameters
NAME          cyc-2006-193
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060817
Time                  15.42
INSTRUM               av300
PROBHD   5 mm QNP 1H/13
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      64
DS                       0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                    1024
DW                  83.400 usec
DE                   6.00 usec
TE                  300.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    3.00 usec
PL1                  -2.00 dB
SF01           300.1320882 MHz

F2 - Processing parameters
SI                   32768
SF             300.1300119 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                 10.000 ppm
F1                3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM              0.52500 ppm/cm
HZCM             157.56825 Hz/cm
```



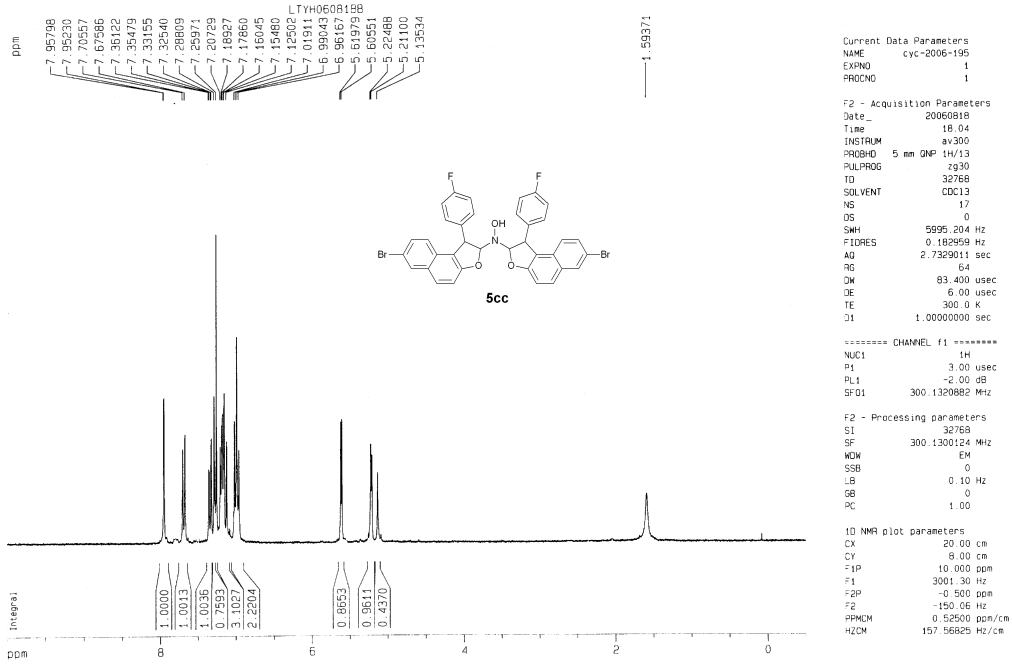

```
Current Data Parameters
NAME          cyc-2006-193
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060820
Time                   9.46
INSTRUM               av300
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg
TD                   65536
SOLVENT              CDCl3
NS                   16767
DS                       4
SWH               22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                    8192
DW                  22.050 usec
DE                   6.00 usec
TE                  300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

-------- CHANNEL f1 --------
NUC1                     13C
P1                    5.50 usec
PL1                  -6.00 dB
SF01           75.4775598 MHz

-------- CHANNEL f2 --------
CPDPRG2              waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 17.70 dB
PL13                 17.71 dB
SF02           300.1312005 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677517 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.00 cm
F1P                200.005 ppm
F1               15093.93 Hz
F2P                 -0.500 ppm
F2                 -37.73 Hz
PPMCM              10.02625 ppm/cm
HZCM             756.58307 Hz/cm
```



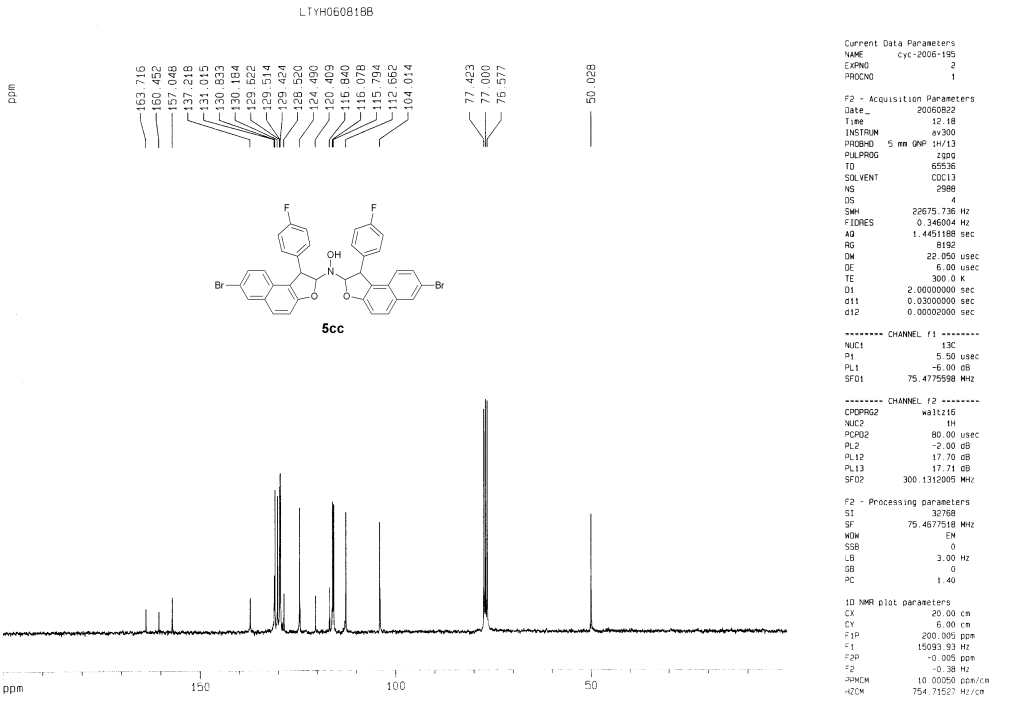
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.160	9166094	49.98	206075	67.54
2	18.967	9171695	50.02	99049	32.46



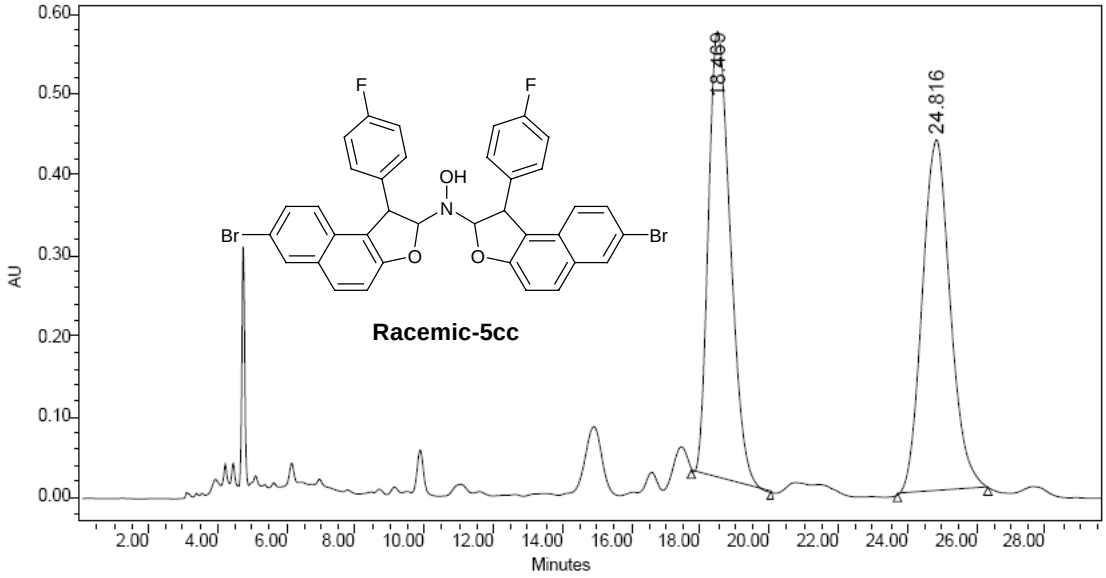
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.088	13141056	100.00	672023	100.00



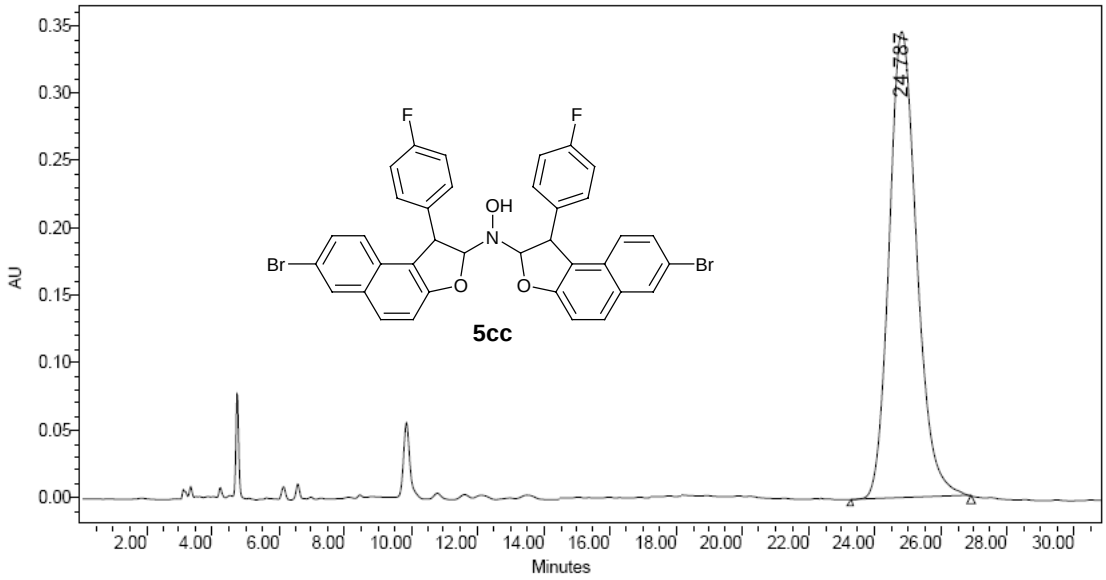

```
Current Data Parameters
NAME        cyc-2006-195
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060818
Time               18.04
INSTRUM            av300
PROBHD   5 mm GNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    17
DS                     0
SWH             5995.204 Hz
FIDRES          0.182959 Hz
AQ              2.7329011 sec
RG                    64
DW               83.400 usec
DE                6.00 usec
TE                300.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 3.00 usec
PL1               -2.00 dB
SFO1         300.1320882 MHz

F2 - Processing parameters
SI                32768
SF          300.1300124 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P              10.000 ppm
F1             3001.30 Hz
F2P              -0.500 ppm
F2             -150.06 Hz
PPMCM          0.52500 ppm/cm
HZCM          157.56825 Hz/cm
```




```
Current Data Parameters
NAME        cyc-2006-195
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060822
Time               12.18
INSTRUM            av300
PROBHD   5 mm GNP 1H/13
PULPROG            zgpg
TD                65536
SOLVENT           CDCl3
NS                  2988
DS                     4
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ              1.4451188 sec
RG                  8192
DW               22.050 usec
DE                6.00 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

-------- CHANNEL f1 --------
NUC1                  13C
P1                 5.50 usec
PL1               -6.00 dB
SFO1         75.4775998 MHz

-------- CHANNEL f2 --------
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              17.70 dB
PL13              17.71 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677518 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 6.00 cm
F1P             200.005 ppm
F1            15093.93 Hz
F2P              -0.005 ppm
F2              -0.38 Hz
PPMCM          10.00050 ppm/cm
HZCM          754.71527 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	18.469	24442292	49.68	553541	55.90
2	24.816	24753929	50.32	436759	44.10



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	24.787	20134247	100.00	346040	100.00