Stereoselective Desymmetrisation of Prochiral α,α-Dicyanoalkenes via Domino Michael-Michael Addition Reactions

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Supplementary Information

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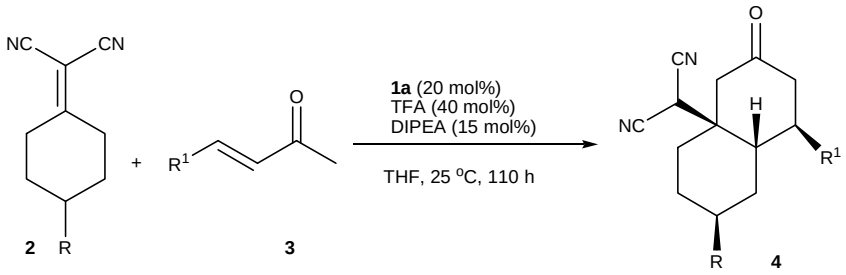
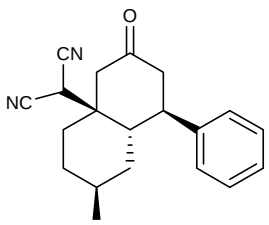
1. General method

TLC was performed on glass-backed silica plates. Column chromatography was performed using silica gel (200–300 mesh) eluting with ethyl acetate and petroleum ether. ^{1}H and ^{13}C NMR were recorded on Bruker 300 or 75 MHz spectrometers, respectively. Chemical shifts were reported in ppm down field with tetramethylsilane resonance as the internal standard. ESI HRMS was recorded on a Bruker Apex-2. Enantiomeric excess was determined by HPLC analysis on Chiralpak columns. All other reagents were used without purification as commercially available.

2. General procedure for the desymmetrisation reaction.

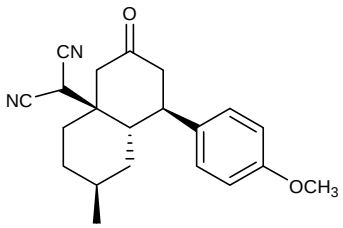


Compound **2** (0.1 mmol)**, 3** (0.12 mmol), catalyst **1a** 6.5 mg (0.02 mmol), TFA 2.9 μL (0.04 mmol) and DIPEA (2.2 μL, 0.015 mmol) were stirred in THF (0.3 mL) at 25 °C for 110 h. Then the reaction was quenched by adding 1 mol/L HCl (0.5 mL). The mixture was diluted with EtOAc (10 mL), washed with water, and dried over anhydrous sodium sulfate. The solvent was removed and flash chromatography on silica gel (ethyl acetate/petroleum ether) gave the product **4**.

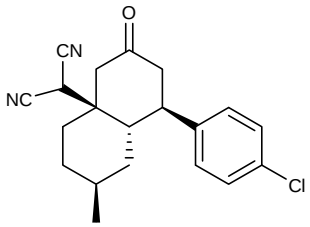


4a 81% yield; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{25}$ = +17.2 (*c* = 0.87 in $CHCl_3$); 99% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 21.14 min, t (major) = 13.46 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.39-7.28 (m, 3H), 7.22-7.20 (m, 2H), 4.43 (s, 1H), 3.26 (td, *J* = 3.4 Hz, 12.0 Hz, 1H), 2.88 (d, *J* = 13.3 Hz, 1H), 2.70-2.66 (m, 1H), 2.62-2.57 (m, 2H), 2.44 (d, *J* = 11.6 Hz, 1H), 1.83-1.78 (m, 3H), 1.70-1.62 (m, 1H), 1.37-1.32 (m, 1H), 0.95-0.89 (m, 2H), 0.79 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.3, 141.4, 129.2, 127.6, 126.9, 111.4, 110.4, 48.9, 48.8, 44.6, 43.7, 41.8, 31.1, 30.9, 29.7, 28.6, 24.9, 22.0; ESI-HRMS: calcd. for $C_{20}H_{22}N_2O$+Na 329.1630, found 329.1630.

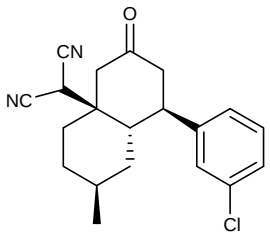
4b 71% yield; R_f = 0.1 (petroleum ether/EtOAc = 15:1); $[\alpha]_D^{25}$ = +18.1 (*c* = 0.66 in $CHCl_3$);



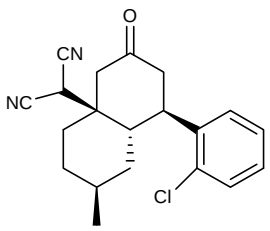
>99.5% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 22.94 min, t (major) = 15.03 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.13 (d, *J* = 7.8 Hz, 2H)，6.89 (d, *J* = 8.0 Hz,, 2 H), 4.43 (s, 1H), 3.81 (s, 3H), 3.22 (td, *J* = 3.3 Hz, 12.7 Hz, 1H), 2.87 (d, *J* = 13.4 Hz, 1H), 2.71-2.62 (m, 1H), 2.60-2.50 (m, 2H), 2.37 (d, *J* = 11.7 Hz, 1H), 1.82-1.76 (m, 3H), 1.68-1.63 (m, 1H), 1.39-1.34 (m, 1H), 0.99-0.83 (m, 2H), 0.79 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.5, 158.8, 133.4, 127.8, 114.5, 111.4, 110.5, 55.3, 49.1, 48.9, 44.5, 42.9, 42.0, 31.1, 30.9, 29.7, 28.6, 24.9, 22.0; ESI-HRMS: calcd. for $C_{21}H_{24}N_2O$+Na 359.1735, found 359.1732.



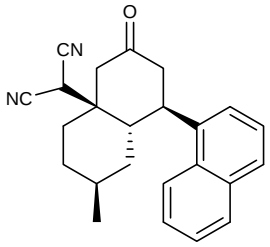
4c 64% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +7.5 (*c* = 0.33 in $CHCl_3$); >99.5% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 23.33 min, t (major) = 10.37 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.35 (d, *J* = 8.5 Hz,, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 4.42 (s, 1H), 3.26 (td, *J* = 4.5 Hz, 12.9 Hz, 1H), 2.87 (d, *J* = 13.3 Hz, 1H), 2.65-2.56 (m, 3H), 2.39 (d, *J* = 11.7 Hz, 1H), 1.81-1.76 (m, 3H), 166-1.59 (m, 1H), 1.35-1.24 (m, 1H), 1.01-0.84 (m, 2H), 0.80 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.8, 139.9, 133.3, 129.5, 128.9, 128.8, 128.2, 111.4, 110.3, 48.8, 48.7, 44.5, 43.2, 41.7, 31.1, 30.9, 29.6, 28.6, 24.9, 22.0; ESI-HRMS: calcd. for $C_{20}H_{21}ClN_2O$+Na 363.1240, found 363.1240.



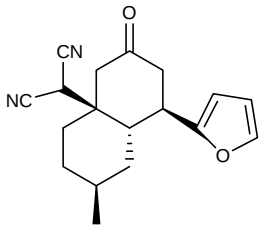
4d 72% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +12.0 (*c* = 0.37 in $CHCl_3$); 95% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) =17.73 min, t (major) = 12.31 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.34-7.26 (m, 2H), 7.20 (s, 1H), 7.10 (d, *J* = 6.7 Hz, 1H), 4.43 (s, 1H), 3.26 (td, *J* = 4.6 Hz, 12.6 Hz, 1H), 2.87 (d, *J* = 13.3 Hz, 1H), 2.71-2.57 (m, 3H), 2.42 (d, *J* = 11.5 Hz, 1H), 1.79-1.77 (m, 3H), 1.66-1.64 (m, 1H), 1.37-1.32 (m, 1H), 0.99-0.88 (m, 2H), 0.81 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.7, 143.4, 135.1, 130.6, 127.9, 127.0, 125.2, 111.4, 110.3, 48.8, 48.6, 44.5, 43.5, 41.5, 31.1, 30.9, 29.6, 28.6, 24.9, 22.0; ESI-HRMS: calcd. for $C_{20}H_{21}ClN_2O$+Na 363.1240, found 363.1240.



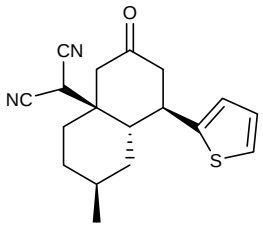
4e 68% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +3.3 (*c* = 0.41 in $CHCl_3$); 92% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 20.53 min, t (major) = 10.89 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.41-7.31 (m, 3H), 7.25-7.19 (m, 1H), 4.44 (s, 1H), 4.01 (td, *J* = 5.0 Hz, 12.1 Hz, 1H), 2.88 (d, *J* = 13.4 Hz, 1H), 2.63-2.50 (m, 4H), 1.86-1.78 (m, 4H), 1.34-1.29 (m, 1H), 1.02-0.86 (m, 2H), 0.81 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.6, 139.1, 133.9, 130.1, 128.5, 128.0, 127.0, 111.5, 110.4, 48.8, 47.9, 44.5, 41.2, 38.6, 31.2, 31.1, 29.7, 28.8, 25.5, 22.0; ESI-HRMS: calcd. for $C_{20}H_{21}ClN_2O$+Na 363.1240, found 363.1240.



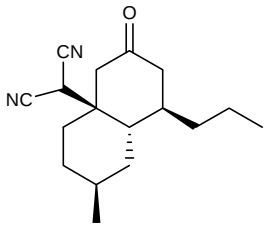
4f 45% yield; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{25}$ = -15.3 (*c* = 0.33 in $CHCl_3$); 98% ee; The enantiomeric excess was determined by HPLC on Chiralpak AD column [20% 2-propanol/hexane, 1.0 mL/min, λ = 254 nm, t (minor) = 7.40 min, t (major) = 5.57 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 8.03 (d, *J* = 7.6 Hz,, 1H), 7.92-7.89 (m, 1H), 7.82-7.79 (m, 1H), 7.58-7.50 (m, 4H), 4.48 (s, 1H), 4.32-4.22 (m, 1H), 2.98 (d, *J* = 13.2 Hz, 1H), 2.79 (d, *J* = 11.6 Hz, 1H), 2.69-2.65 (m, 3H), 1.85-1.81 (m, 2H), 1.62-1.60 (m, 2H), 1.40 (d, *J* = 14.5 Hz, 1H), 1.05-0.89 (m, 2H), 0.64 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.3, 138.0, 134.1, 131.3, 129.3, 127.7, 126.7, 125.9, 125.8, 123.2, 121.6, 111.6, 110.5, 49.3, 49.0, 44.8, 41.5, 36.7, 31.3, 31.0, 29.7, 28.9, 25.8, 21.9; ESI-HRMS: calcd. for $C_{24}H_{24}N_2O$+Na 379.1786, found 379.1748.



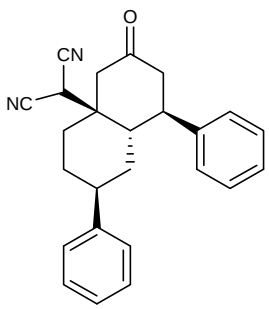
4g 64% yield; R_f = 0.1 (petroleum ether/EtOAc = 18:1); $[\alpha]_D^{25}$ = +17.3 (*c* = 0.68 in $CHCl_3$); 97% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 19.70 min, t (major) = 12.96 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.37 (d, *J* = 1.8 Hz, 1H)，6.32 (dd, *J* = 1.9 Hz, 3.2 Hz, 1H)，6.11 (d, *J* = 3.2 Hz, 1H), 4.40 (s, 1H), 3.43 (td, *J* = 4.2 Hz, 13.3 Hz, 1H), 2.88-2.79 (m, 2H), 2.59-2.48 (m, 3H), 1.75-1.70 (m, 4H), 1.45-1.40 (m, 1H), 1.05-0.94 (m, 2H), 0.87 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.9, 153.7, 142.1, 111.3, 110.4, 110.2, 106.6, 48.6, 46.0, 44.1, 40.3, 37.3, 31.3, 31.0, 29.6, 28.4, 24.9, 22.0; ESI-HRMS: calcd. for $C_{18}H_{20}N_2O_2$+Na 319.1422, found 319.1400.



4h 60% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +16.7 (*c* = 0.69 in $CHCl_3$); 97% ee; The enantiomeric excess was determined by HPLC on Chiralpak AD column [10% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 15.34 min, t (major) = 12.68 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.26-7.22 (m, 1H), 6.97 (dd, *J* = 3.5 Hz, 5.1 Hz, 1H), 6.88-6.87 (m, 1H), 4.43 (s, 1H), 3.62 (td, *J* = 6.8 Hz, 11.2 Hz, 1H), 2.86 (d, *J* = 13.4 Hz, 1H), 2.78-2.70 (m, 2H), 2.58 (d, *J* = 13.4 Hz, 1H), 2.35-2.32 (m, 1H), 1.82-1.63 (m, 4H), 1.57-1.53 (m, 1H), 1.02-0.93 (m, 2H), 0.85 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.2, 145.1, 127.0, 124.5, 124.2, 111.3, 110.4, 49.9, 48.9, 44.4, 43.7, 39.1, 31.1, 29.7, 28.7, 25.0, 22.0; ESI-HRMS: calcd. for $C_{18}H_{20}N_2OS$+Na 335.1194, found 335.1214.

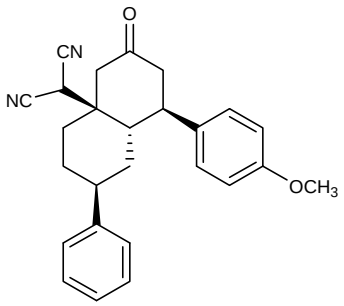


4i 54% yield; R_f = 0.1 (petroleum ether/EtOAc = 20:1); $[\alpha]_D^{25}$ = +12.0 (*c* = 0.37 in $CHCl_3$); 98% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 254 nm, t (minor) = 12.58 min, t (major) = 29.58 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 4.42 (s, 1H), 2.69 (d, *J* = 13.0 Hz, 1H), 2.50-2.46 (m, 2H), 2.18-2.14 (m, 2H), 1.96-1.92 (m, 2H), 1.73-1.64 (m, 3H), 1.61-1.54 (m, 2H), 1.42-1.29 (m, 1H), 1.28-1.23 (m, 3H), 1.08-0.98 (m, 1H), 0.94-0.87 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 206.6, 111.4, 110.6, 48.9, 45.8, 44.9, 41.0, 35.7, 35.5, 31.0, 30.6, 29.9, 28.7, 24.9, 22.2, 18.6, 14.1; ESI-HRMS: calcd. for $C_{17}H_{24}N_2O$+Na 295.1786, found 295.1766.

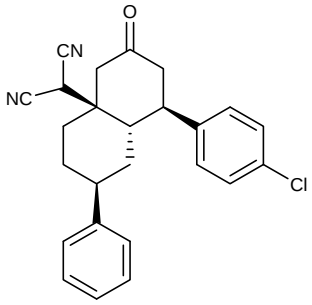


4j 61% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = -9.3 (*c* = 0.78 in $CHCl_3$); 95% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 23.86 min, t (major) = 14.38 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.38-7.35 (m, 2H), 7.30-7.27 (m, 4 H), 7.23-7.18 (m, 2H), 7.06-7.02 (m, 2H), 4.58 (s, 1H), 3.46 (td, *J* = 4.3 Hz, 13.0 Hz, 1H), 2.94 (d, *J* = 13.3 Hz, 1H), 2.90-2.80 (m, 1H), 2.77-2.56 (m, 4H), 2.05-1.96 (m, 3H), 1.60-1.51 (m, 2H), 1.45-1.34 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.0, 143.9, 141.0, 129.4, 128.7, 127.8, 126.9, 126.8, 126.3, 111.4, 110.4, 48.9, 48.8, 44.5, 43.6, 42.0, 35.8, 31.3, 30.1, 28.8, 28.0; ESI-HRMS: calcd. for $C_{25}H_{24}N_2O$+Na 391.1786, found 391.1743.

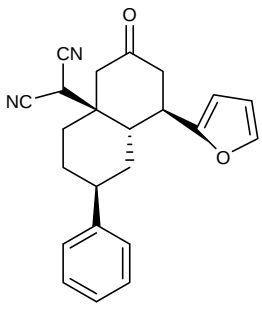
4k 66% yield; R_f = 0.1 (petroleum ether/EtOAc = 18:1); $[\alpha]_D^{25}$ = -7.1 (*c* = 0.87 in $CHCl_3$); 97% ee;



The enantiomeric excess was determined by HPLC on Chiralpak ADcolumn [20% 2-propanol/ hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 12.62 min, t (major) = 7.99 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.29-7.24 (m, 2H), 7.21-7.16 (m, 3 H), 7.05 (d, *J* = 8.3 Hz,, 2H), 6.90 (d, *J* = 8.5 Hz,, 2H), 4.58 (s, 1H), 3.79 (s, 3H), 3.40 (td, *J* = 4.4 Hz, 12.4 Hz, 1H), 2.93 (d, *J* = 13.4 Hz, 1H), 2.88-2.80 (m, 1H), 2.78-2.58 (m, 3H), 2.52-2.49 (m, 1H), 2.05-1.95 (m, 3H), 1.64-1.50 (m, 2H), 1.43-1.33 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.2, 158.9, 143.9, 133.1, 128.7, 127.8, 126.8, 126.3, 114.7, 111.4, 110.4, 55.3, 49.0, 48.9, 44.4, 42.8, 42.3, 35.8, 31.3, 30.1, 28.8, 28.1; ESI-HRMS: calcd. for $C_{26}H_{26}N_2O_2$+Na 421.1892, found 421.1895.

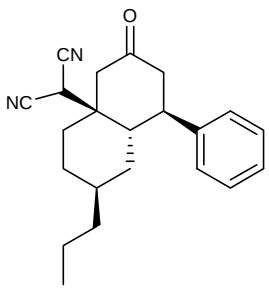


4l 67% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = -2.8 (*c* = 1.1 in $CHCl_3$); 97% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 28.01 min, t (major) = 11.36 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.36-7.33 (m, 2H), 7.30-7.17 (m, 5H), 7.05-7.03 (m, 2H), 4.58 (s, 1H), 3.44 (td, *J* = 4.2 Hz, 12.2 Hz, 1H), 2.93 (d, *J* = 13.3 Hz, 1H), 2.86-2.77 (m, 1H), 2.72-2.51 (m, 4H), 2.05-1.94 (m, 3H), 1.61-1.50 (m, 2H), 1.48-1.42 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.5, 143.6, 139.5, 133.5, 129.6, 128.7, 128.2, 126.9, 126.3, 111.3, 110.3, 48.8, 48.6, 44.4, 43.0, 41.9, 35.8, 31.3, 30.0, 28.8, 28.0; ESI-HRMS: calcd. for $C_{25}H_{23}ClN_2O$+Na 425.1397, found 425.1422.

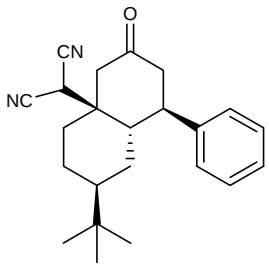


4m 62% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = -15.8 (*c* = 0.35 in $CHCl_3$); 90% ee; The enantiomeric excess was determined by HPLC on Chiralpak AD column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 11.23 min, t (major) = 7.94 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.36 (d, *J* = 9.0 Hz, 1H), 7.32-7.19 (m, 3 H), 7.12 (d, *J* = 7.0 Hz, 2H), 6.32 (dd, *J* = 2.0 Hz, 3.1 Hz, 1H), 6.20 (d, *J* = 3.1 Hz, 1H), 4.57 (s, 1H), 3.63 (td, *J* = 3.8 Hz, 11.7 Hz, 1H), 2.95-2.82 (m, 3H), 2.64 (d, *J* = 13.3 Hz, 3H), 2.04-2.00 (m, 1H), 1.93-1.89 (m, 2H), 1.69-1.61 (m, 1H), 1.57-1.49 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.6, 153.4, 143.9, 142.2, 128.7, 126.9, 126.4, 111.2, 110.3, 106.8, 48.7, 46.0, 44.0, 40.7, 37.1, 35.8, 31.2, 30.5, 28.7, 28.0; ESI-HRMS: calcd. for $C_{23}H_{23}N_2O_2$+Na 381.1579, found 381.1563.

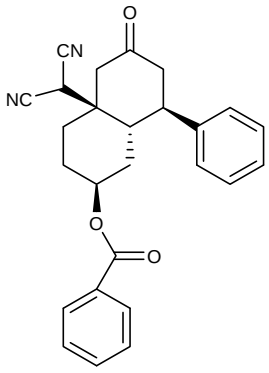
4n 61% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +8.9 (*c* = 0.75 in $CHCl_3$); 94% ee;



The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 18.64 min, t (major) = 9.47 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.39-7.30 (m, 3H), 7.29-7.20 (m, 2H), 4.42 (s, 1H), 3.27 (td, *J* = 4.3 Hz, 13.2 Hz, 1H), 2.88 (d, *J* = 13.3 Hz, 1H), 2.67 (t, *J* = 13.3 Hz, 1H), 2.61-2.53 (m, 2H), 2.45-2.41 (m, 1H), 1.86-1.78 (m, 3H), 1.54-1.53 (m, 1H), 1.42-1.37 (m, 1H), 1.16-1.08 (m, 3H), 1.07-0.84 (m, 3H), 0.78 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.4, 141.3, 129.2, 127.6, 126.9, 111.5, 110.5, 48.9, 48.7, 44.9, 43.7, 41.8, 38.9, 31.1, 29.4, 28.9, 28.6, 27.9, 19.5, 14.0; ESI-HRMS: calcd. for $C_{22}H_{26}N_2O$+Na 357.1943, found 357.1949.

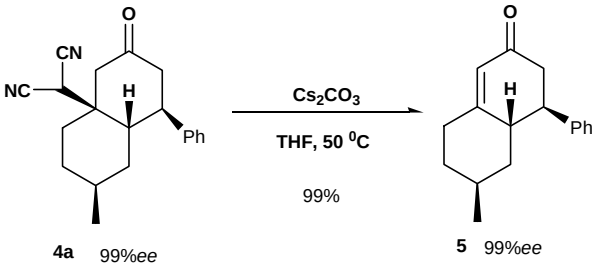


4o 71% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = -9.3 (*c* = 0.78 in $CHCl_3$); 99.5% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 24.08 min, t (major) = 9.08 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.46-7.30 (m, 3H), 7.29-7.21 (m, 2H), 4.35 (s, 1H), 3.28 (td, *J* = 4.1 Hz, 11.7 Hz, 1H), 2.88 (d, *J* = 13.3 Hz, 1H), 2.78-2.69 (m, 1H), 2.62-2.54 (m, 2H), 2.49-2.44 (m, 1H), 1.84-1.79 (m, 3H), 1.38-1.29 (m, 2H), 1.05-0.93 (m, 2H), 0.70 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 205.2, 141.3, 129.1, 127.7, 127.0, 111.5, 110.5, 48.9, 48.7, 44.7, 43.7, 42.1, 39.6, 32.2, 31.2, 29.0, 27.0, 23.3, 22.2; ESI-HRMS: calcd. for $C_{23}H_{28}N_2O$+Na 371.2099, found 371.2074.



4p 60% yield; R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = -16.9 (*c* = 0.74 in $CHCl_3$); 98% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/ hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 16.26 min, t (major) = 13.92 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.93 (d, *J* = 7.2 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 6.2 Hz, 4H), 7.34-7.31 (m, 3H), 5.29-5.22 (m, 1H), 4.54 (s, 1H), 3.31 (td, *J* = 4.4 Hz, 11.9 Hz, 1H), (m, 1H), 2.92 (d, *J* = 13.6 Hz, 1H), 2.81-2.62 (m, 4H), 2.31-2.25 (m, 1H), 2.00-1.97 (m, 2H), 1.84-1.79 (m, 1H), 1.52-1.48 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 204.5, 165.4, 140.7, 133.2, 129.7, 129.5, 129.4, 128.4, 128.0, 127.0, 111.1, 110.1, 66.6, 48.3, 48.0, 44.4, 44.2, 42.2, 31.4, 28.1, 27.7, 26.7; ESI-HRMS: calcd. for $C_{26}H_{24}N_2O_3$+Na 435.1685, found 435.1641.

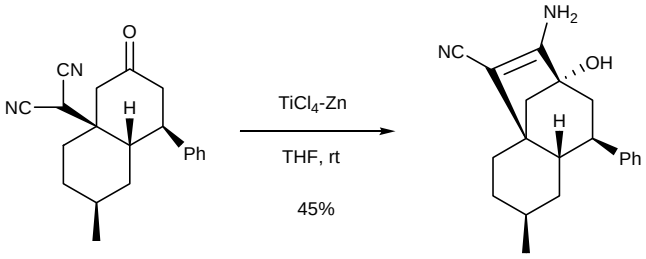
3. Procedure for the Synthesis of Compound 5



4a 99%*ee* **5** 99%*ee*

The mixture of **4a** (30.6 mg, 0.1 mmol) and Cs_2CO_3 (65 mg, 0.2 mmol) in freshly distilled dry THF (0.3 mL) was stirred at 50 °C until the reaction was complete. The mixture was diluted with EtOAc (10 mL) and washed with water. After dried with anhydrous sodium sulfate, the solvent was removed and flash chromatography on silica gel (4% ethyl acetate/petroleum ether) gave compound **5** (23.8 mg, 99% yield). R_f = 0.1 (petroleum ether/EtOAc = 25:1); $[\alpha]_D^{25}$ = +11.0 (*c* = 0.75 in $CHCl_3$); 99% ee; The enantiomeric excess was determined by HPLC on Chiralpak AS column [20% 2-propanol/hexane, 1.0 mL/min, λ = 220 nm, t (minor) = 33.19 min, t (major) = 26.55 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.36-7.29 (m, 2H), 7.28-7.23 (m, 1H), 7.22-7.17 (m, 2H), 5.93 (s, 1H), 2.88-2.84 (m, 1H), 2.72-2.68 (m, 1H), 2.63-2.48 (m, 3H), 2.38-2.29 (m, 1H), 2.06-2.03 (m, 1H), 1.73-1.62 (m, 2H), 1.56-1.50 (m, 1H), 1.33-1.25 (m, 1H), 0.98 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 199.0, 166.9, 142.5, 128.8, 127.3, 126.8, 124.6, 47.8, 45.2, 37.7, 36.2, 31.4, 30.0, 26.9, 17.0; ESI-HRMS: calcd. for $C_{17}H_{20}O$+Na 263.1412, found 263.1439.

4. Procedure for the Synthesis of Compound 6

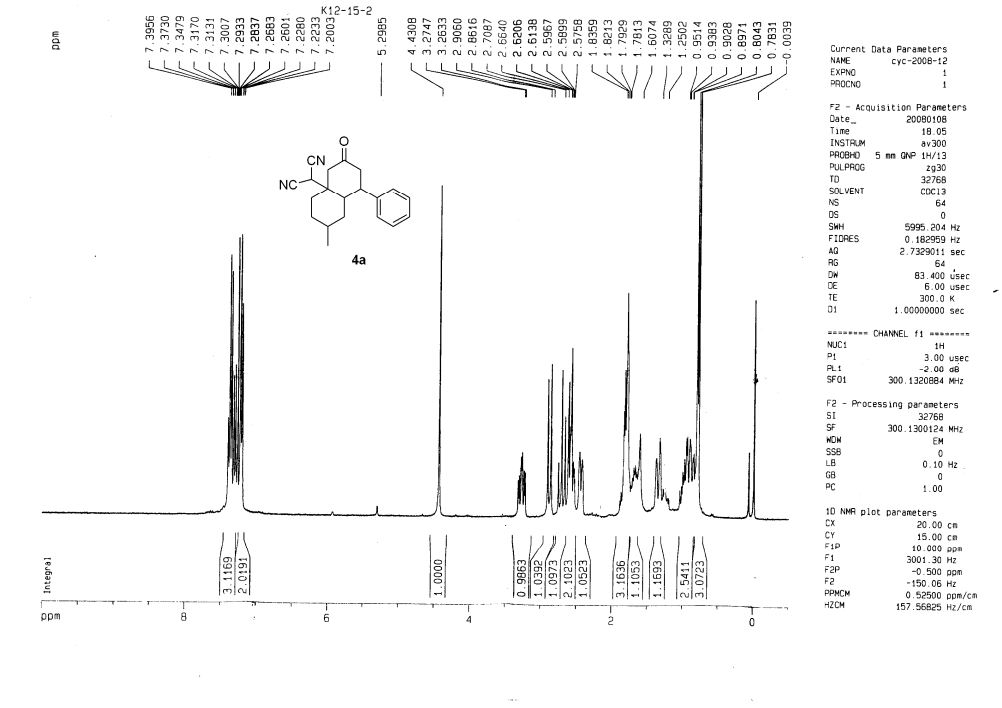


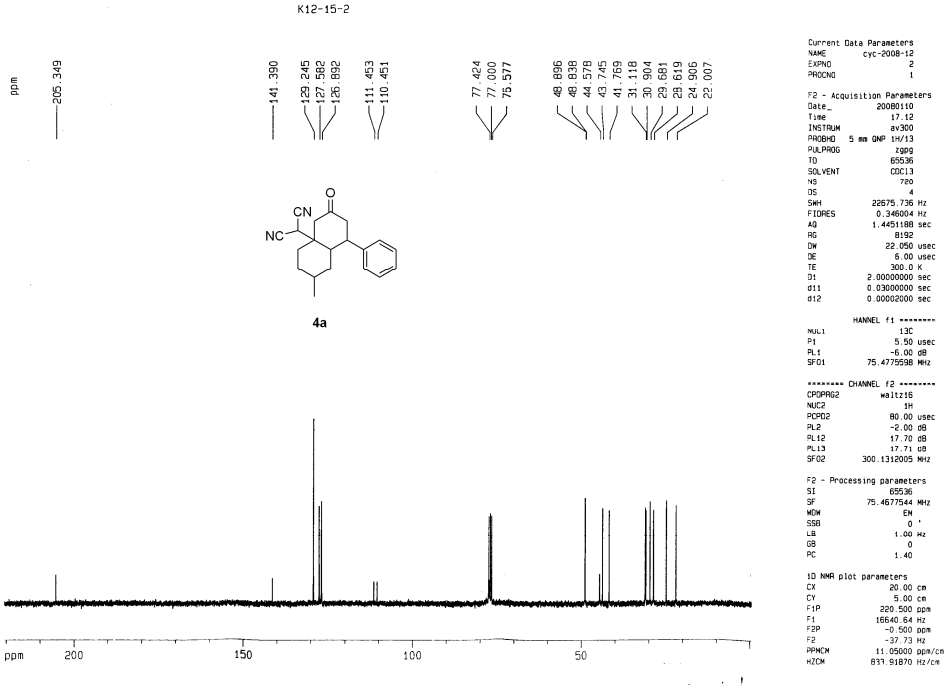
4a 99%*ee* **6** 99%*ee*

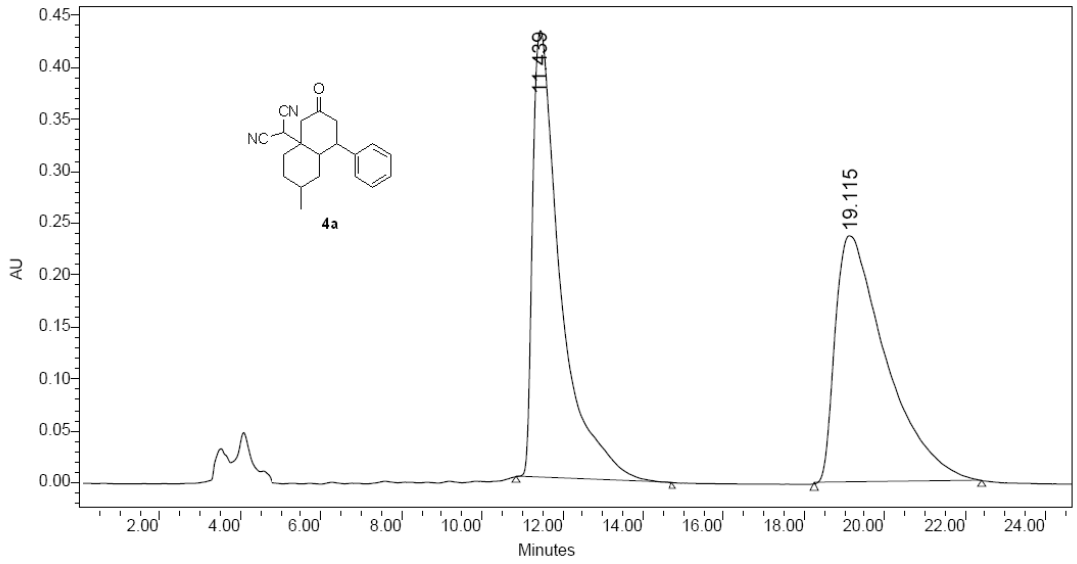
$TiCl_4$ (44 μL, 0.4mmol) was added dropwise by using a syringe to a stirred suspension of Zn powder (52 mg, 0.80 mmol) in freshly distilled dry THF (0.3 mL) at room temperature under Ar atmosphere. Then the mixture was refluxed for 2 h. The suspension of the formed low-valent titanium reagent was cooled to room temperature and the solution of **4a** (30.6 mg, 0.1mmol) in anhydrous THF (0.20 mL) was added. The mixture was stirred at room temperature for 5 h, and then quenched with 5% HCl (1.0 mL). The mixture was extracted with $CHCl_3$ (3×10 mL). The combined organic phase was washed with water (2×10 mL) and dried over anhydrous Na_2SO_4.

After concentration, the crude product was purified by column chromatography (silica gel, ethyl acetate/petroleum ether, 1:3) to give pure compound **6** (13.8 mg, yield 45%). R_f = 0.1 (petroleum ether/EtOAc = 4:1); $[\alpha]_D^{25}$ = +14.0 (*c* = 0.61 in $CHCl_3$); 99% ee; The enantiomeric excess was determined by HPLC on Chiralpak IC column [20% 2-propanol/hexane, 1.0 mL/min, λ = 254 nm, t (minor) = 6.21 min, t (major) = 5.33 min]; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.31-7.27 (m, 2H), 7.21-7.19 (m, 2H), 7.17-7.12 (m, 1H), 4.66 (s, 2H), 2.90-2.86 (m, 1H), 2.28 (d, *J* = 9.9 Hz, 1H), 2.17-2.13 (m, 2H), 2.08-2.01 (m, 3H), 1.98-1.97 (m, 1H), 1.78-1.70 (m, 2H), 1.68-1.57 (m, 1H), 1.44-1.41 (m, 2H), 1.37-1.32 (m, 1H), 0.98 (d, *J* = 7.21 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 163.7, 147.2, 128.3, 127.0, 125.6, 117.5, 81.8, 79.2, 46.5, 46.0, 44.8, 38.3, 37.9, 36.1, 30.0, 27.5, 27.4, 18.0; ESI-HRMS: calcd. for $C_{20}H_{24}N_2O$+Na 331.1786 found 331.1749.

5. NMR and HPLC spectra







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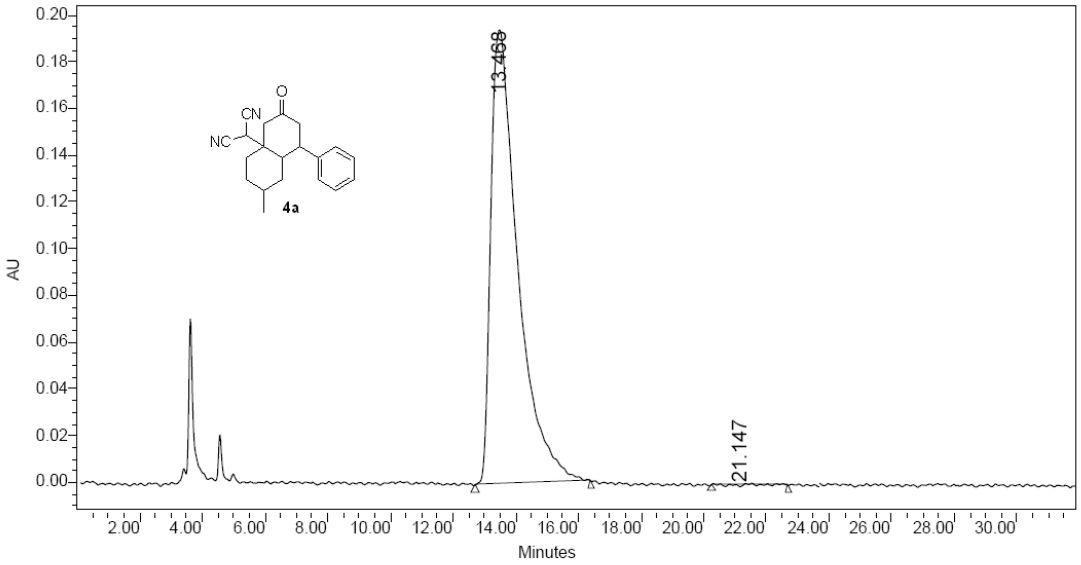

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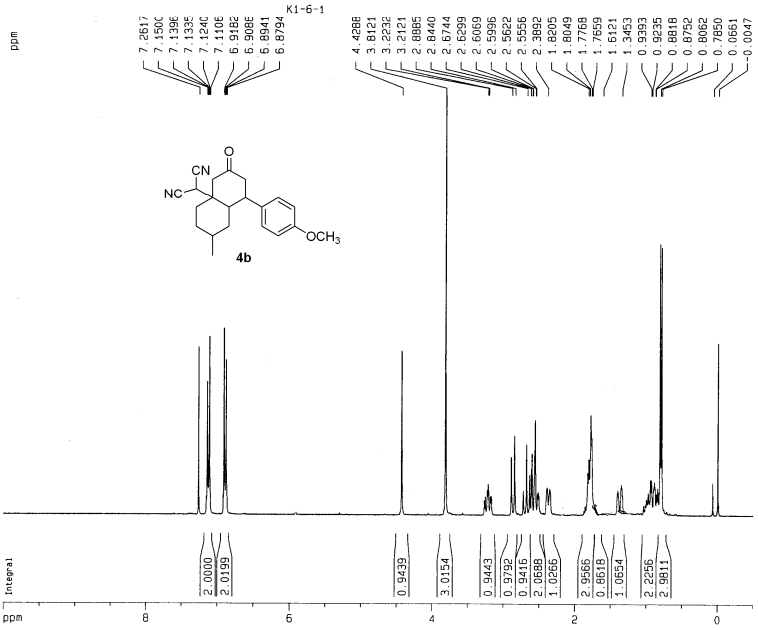


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PROCNO                  1

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FIDRES       0.182959 Hz
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RG                 512
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DE               6.00 usec
TE              300.0 K
D1           1.00000000 sec

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PL1             -2.00 dB
SFO1       300.1320884 MHz

F2 - Processing parameters
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SF          300.1300121 MHz
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GB                   0
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1D NMR plot parameters
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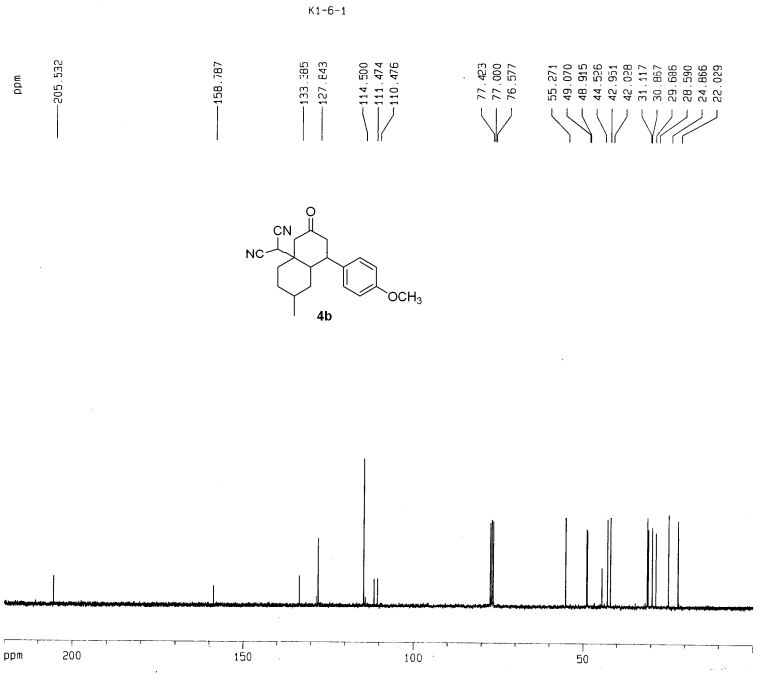


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PROCNO                  1

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D1           2.00000000 sec
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d12          0.00002000 sec

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PL1             -6.00 dB
SFO1       75.4775598 MHz

======== CHANNEL f2 ========
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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
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GB                   0
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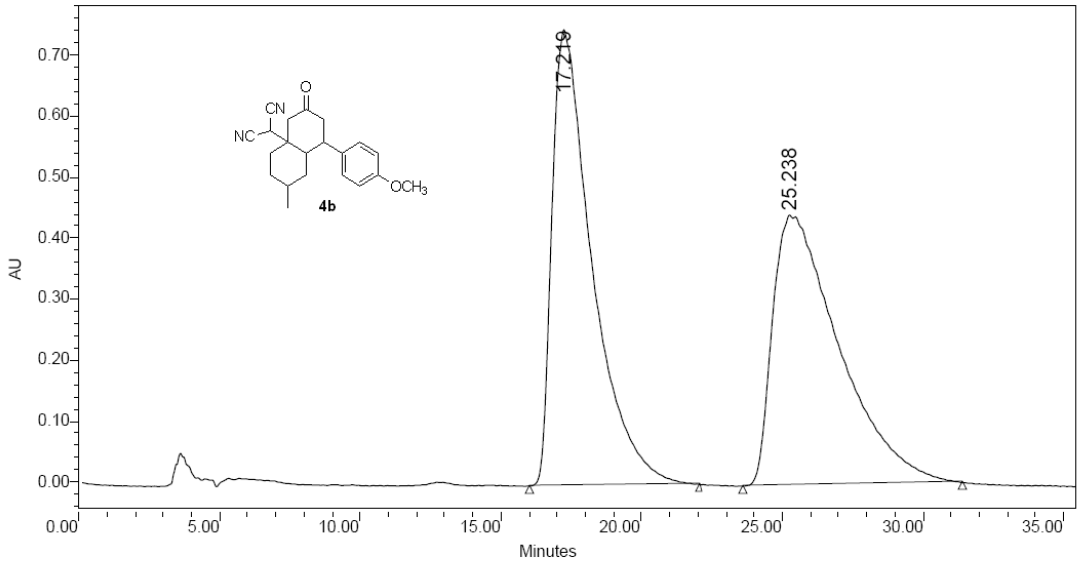

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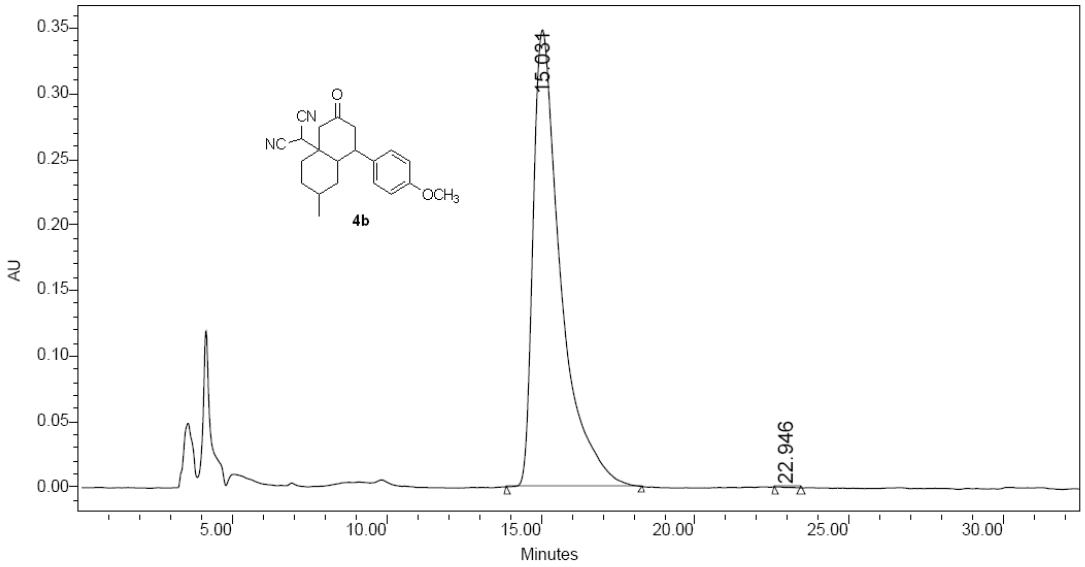

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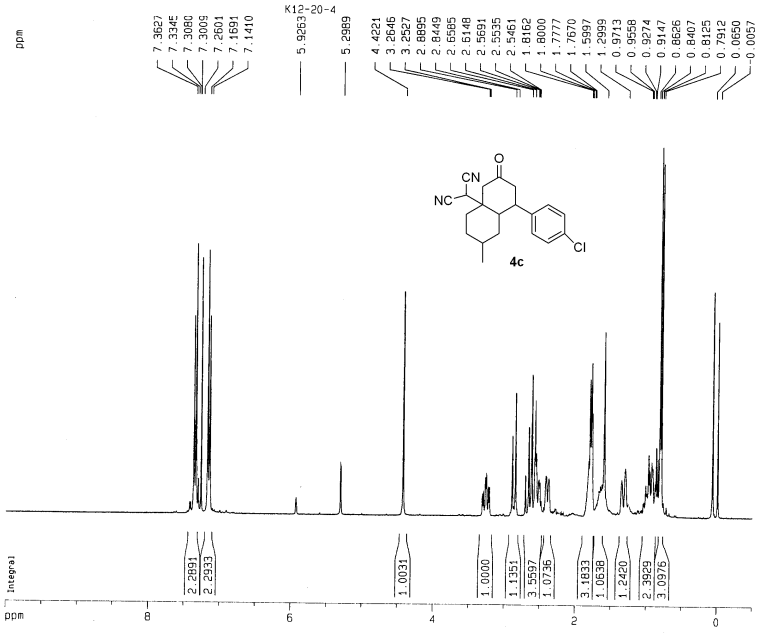

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PROCNO 1

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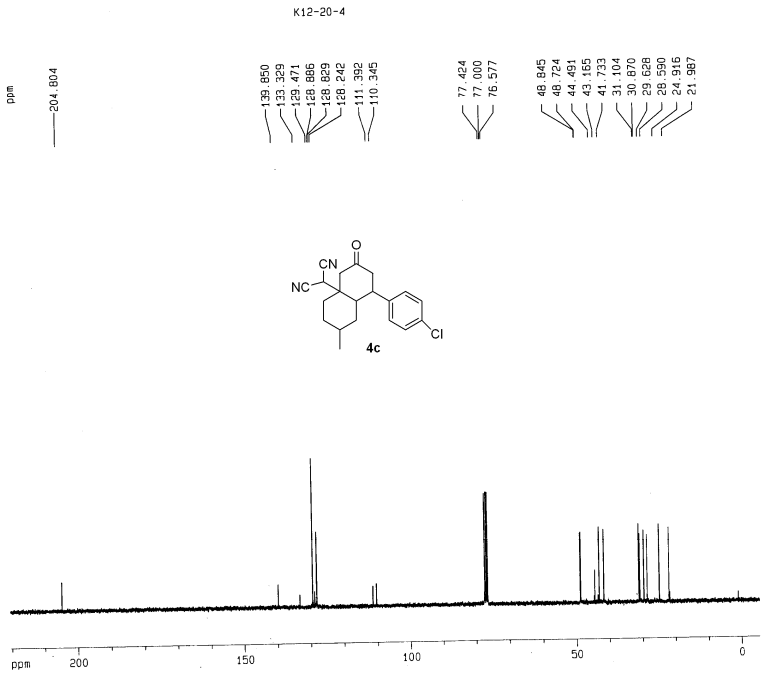

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PROCNO 1

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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

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P1 5.50 usec
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======== CHANNEL f2 ========
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PCPD2 80.00 usec
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PL12 17.70 dB
PL13 17.71 dB
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F2 - Processing parameters
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SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
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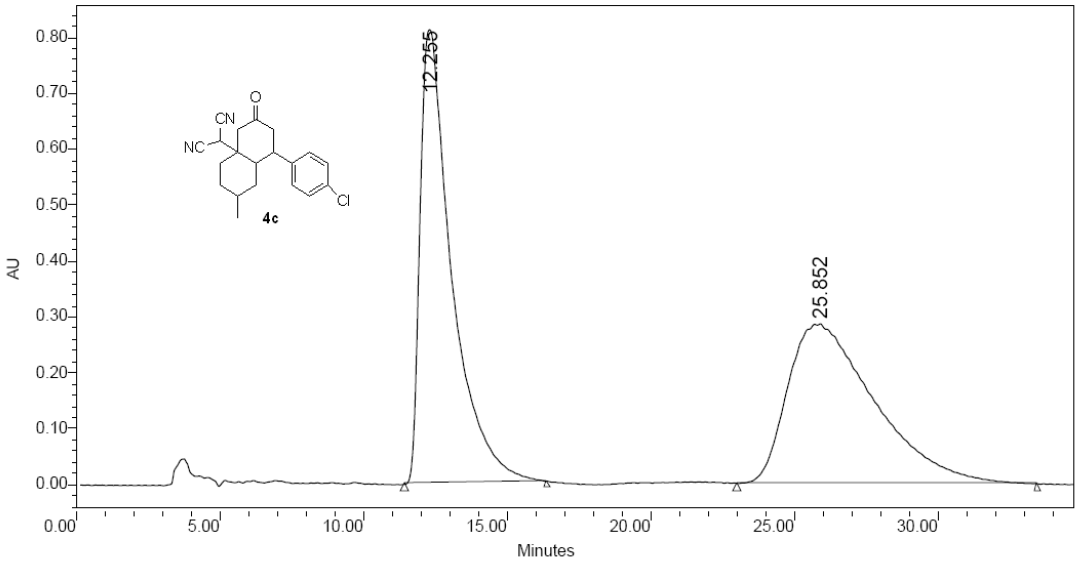

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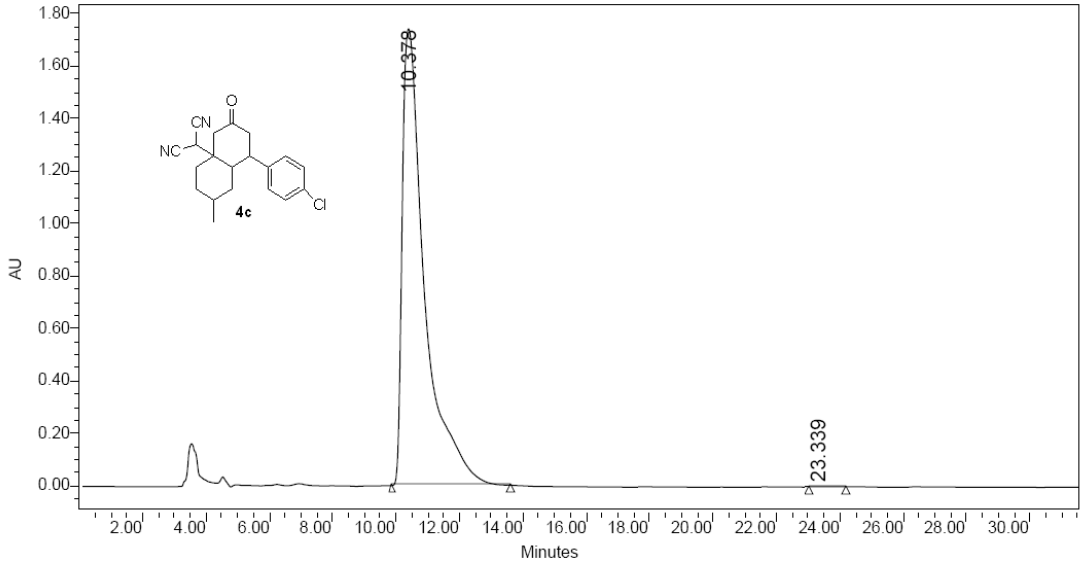

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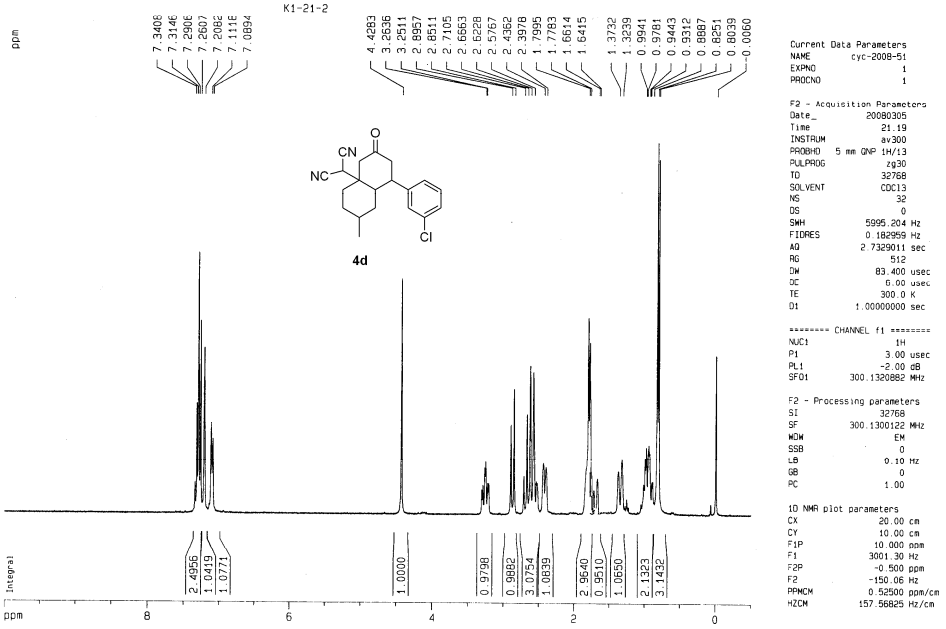

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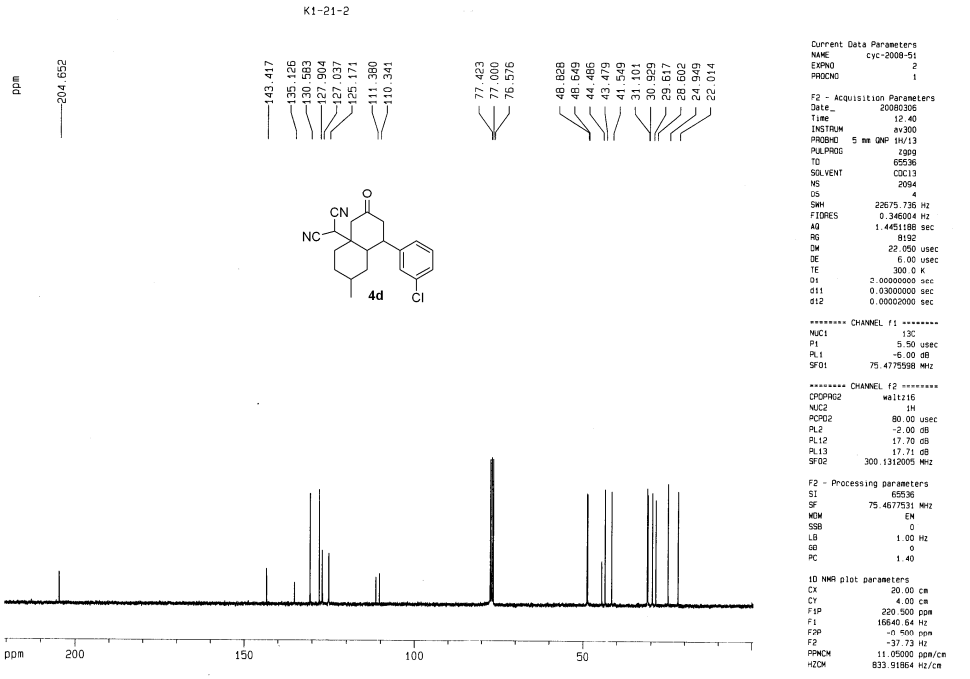
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 32768
SF 300.1300122 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-2008-51
EXPNO 2
PROCNO 1

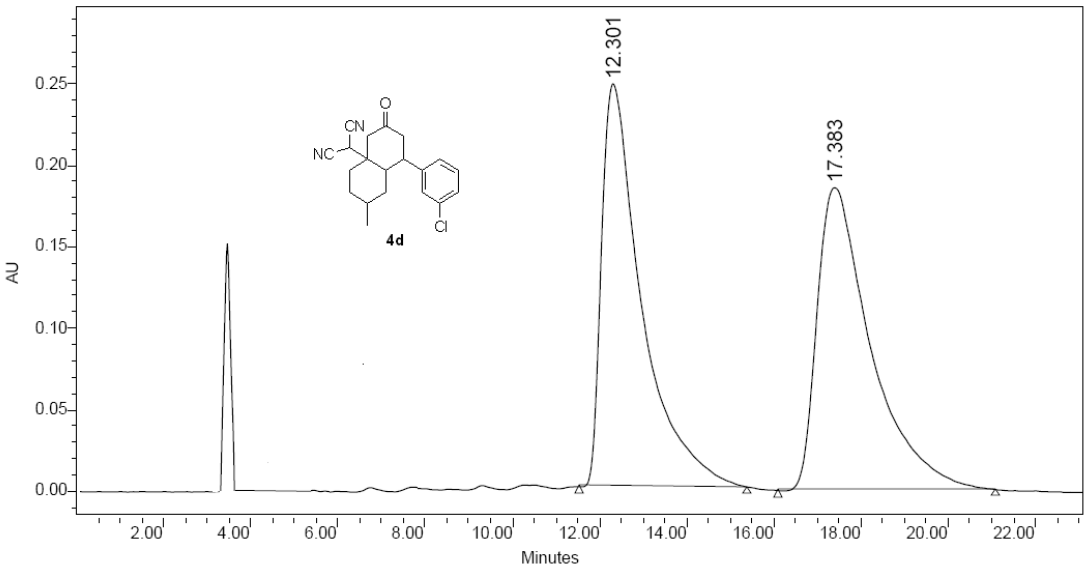
F2 - Acquisition Parameters
Date_ 20080306
Time 12.40
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2094
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

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

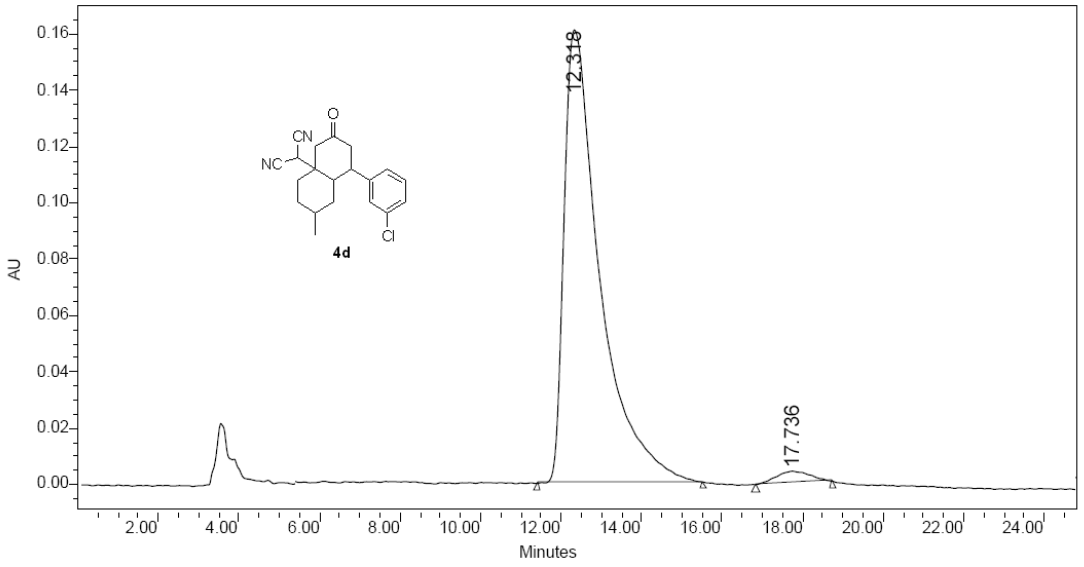
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

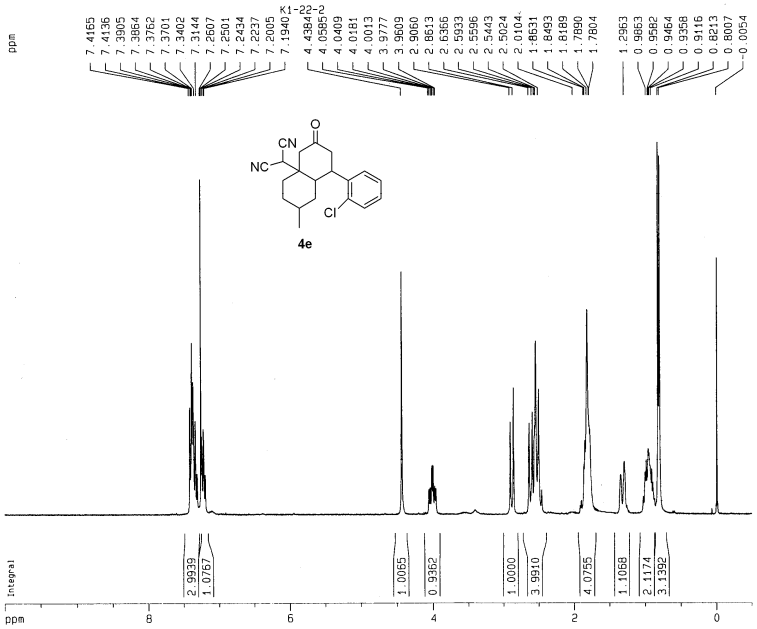
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91864 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.301	15782590	49.83	247221	57.12
2	17.383	15889662	50.17	185593	42.88



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.318	9922376	97.52	160915	97.42
2	17.736	252244	2.48	4254	2.58



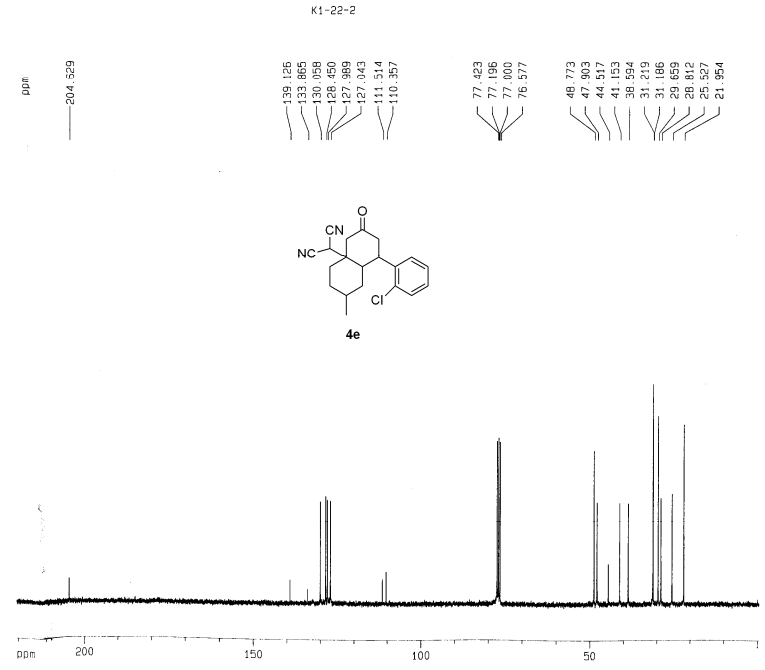
Current Data Parameters
NAME cyc-2008-52
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080305
Time 21.14
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 300.1300122 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-2008-52
EXPNO 2
PROCNO 1

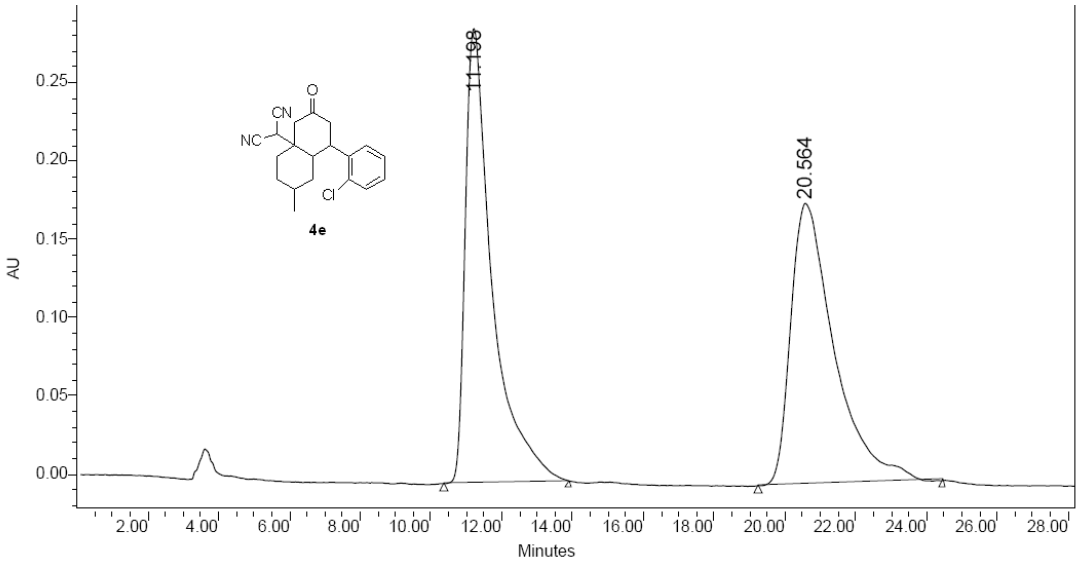
F2 - Acquisition Parameters
Date_ 20080310
Time 12.48
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2260
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

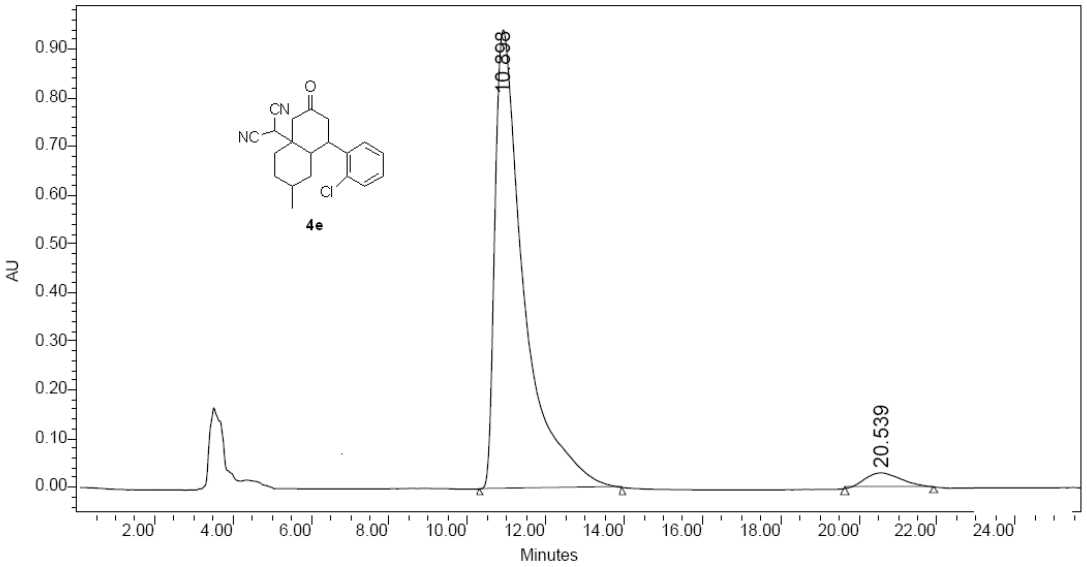
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677528 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

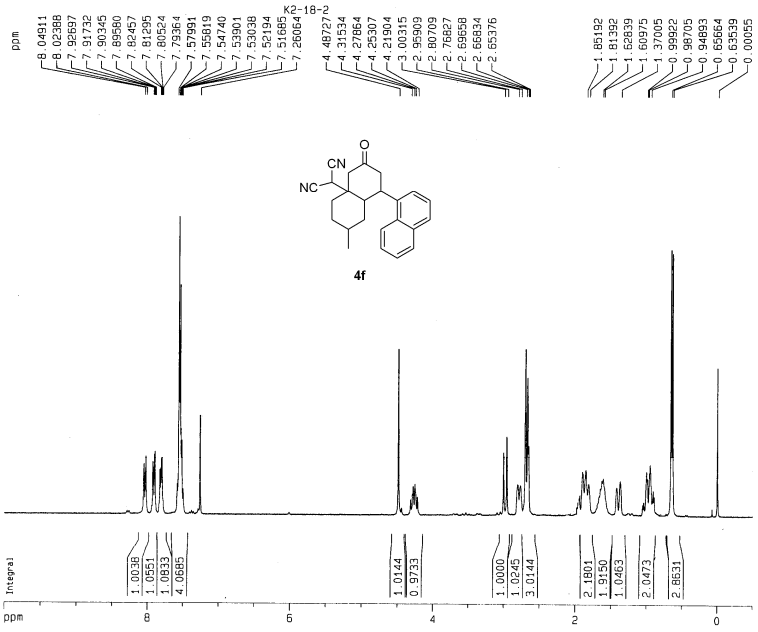
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91864 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.198	15336962	49.91	290172	61.77
2	20.564	15389211	50.09	179621	38.23



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	10.898	48520119	96.05	942078	96.86
2	20.539	1993509	3.95	30560	3.14



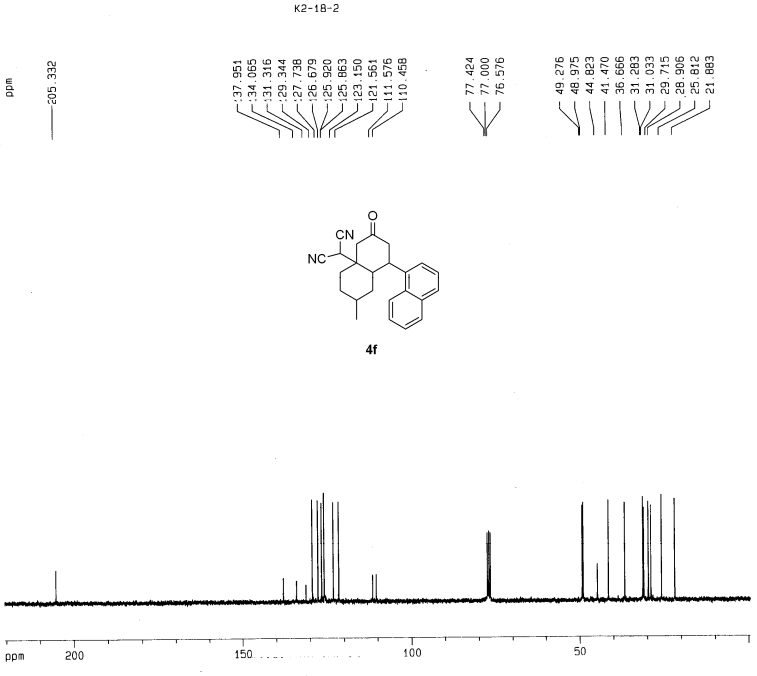
Current Data Parameters
NAME cyc-2008-63
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080314
Time 9.52
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
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 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-2008-63
EXPNO 2
PROCNO 1

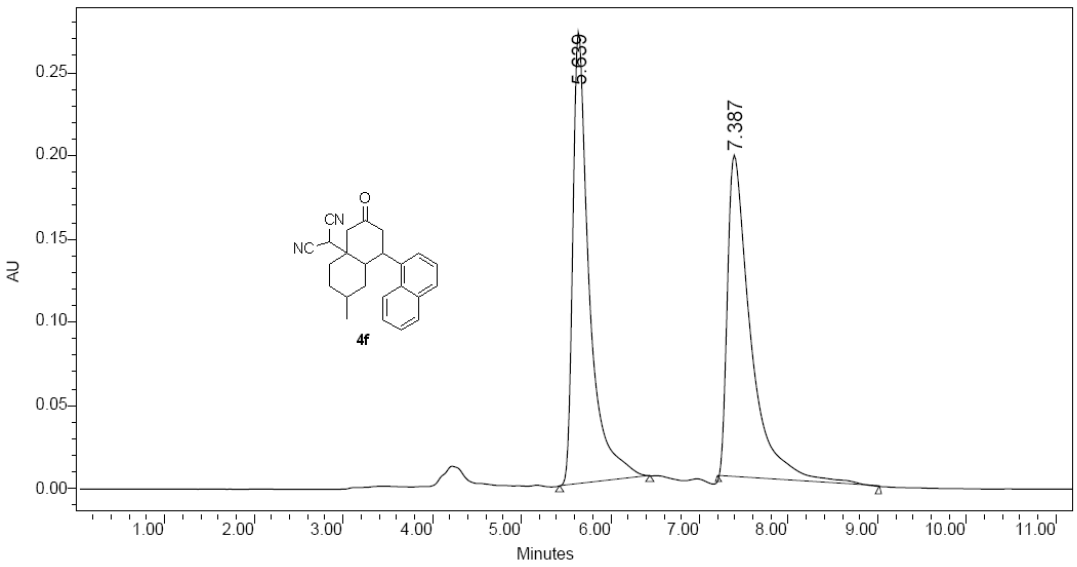
F2 - Acquisition Parameters
Date_ 20080319
Time 20.04
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1406
DS 4
SWH 22675.736 Hz
FIDRES 0.346094 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

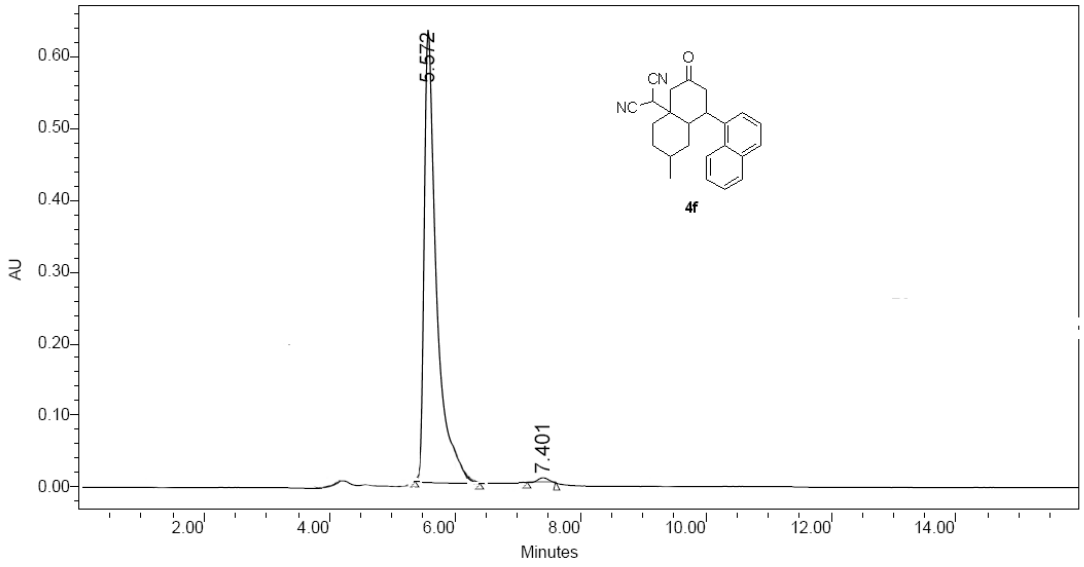
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

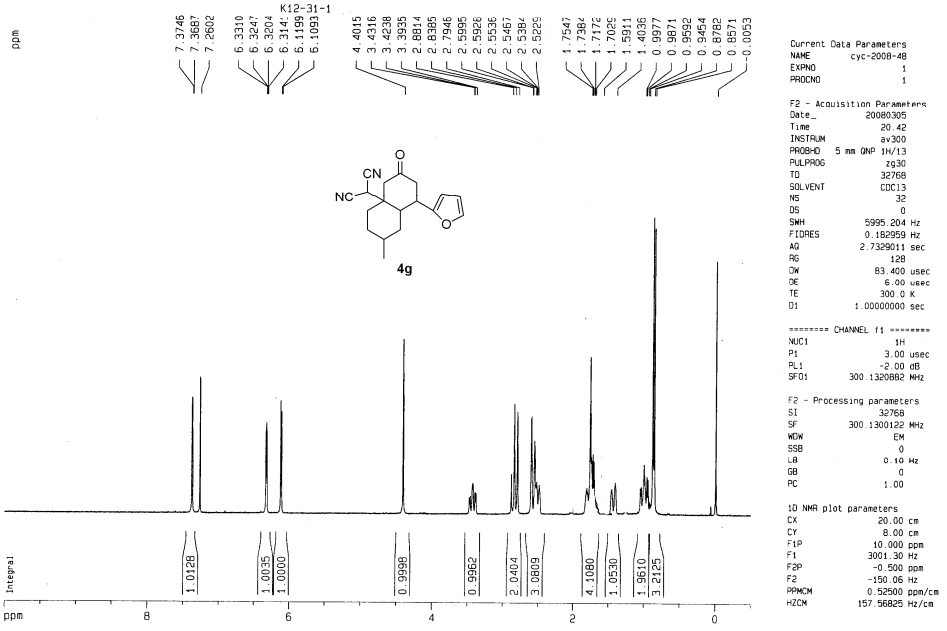
1D NMR plot parameters
CX 20.00 cm
CY 3.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91864 Hz/cm

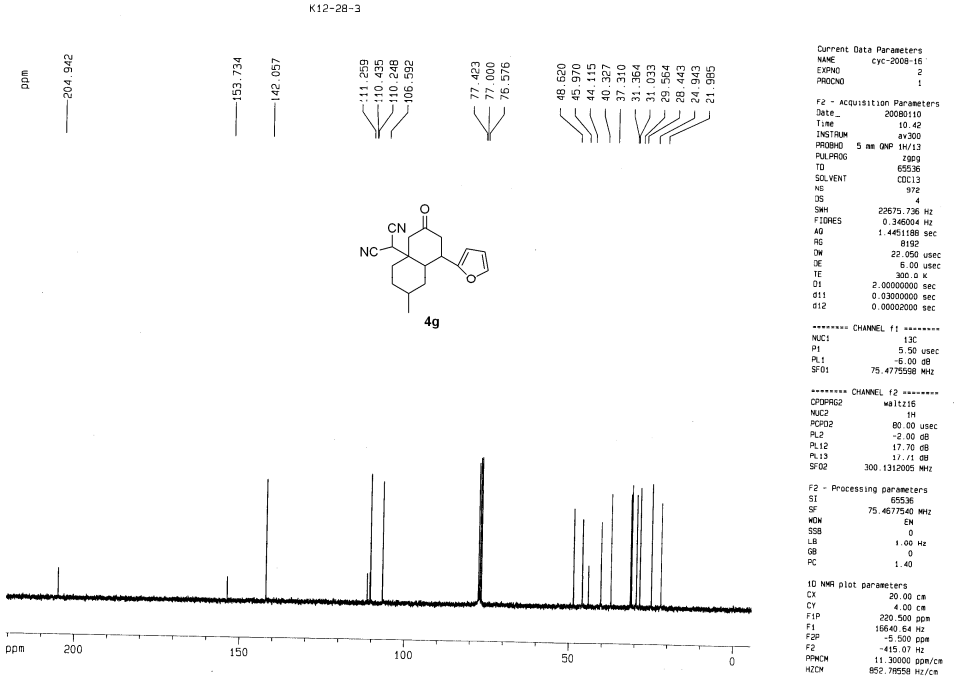


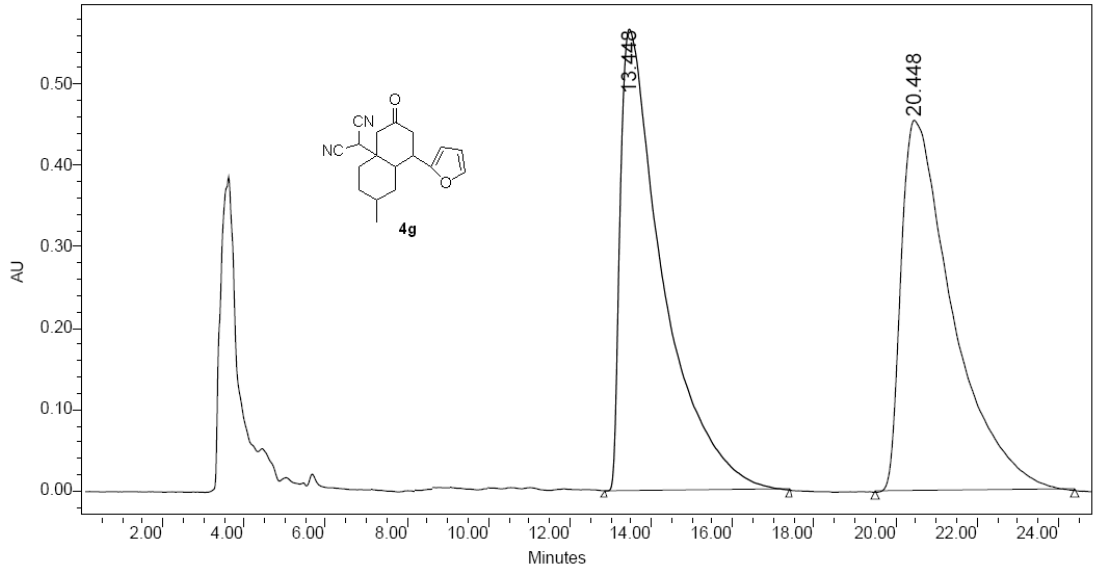
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.639	3507326	50.08	272052	58.46
2	7.387	3496647	49.92	193292	41.54



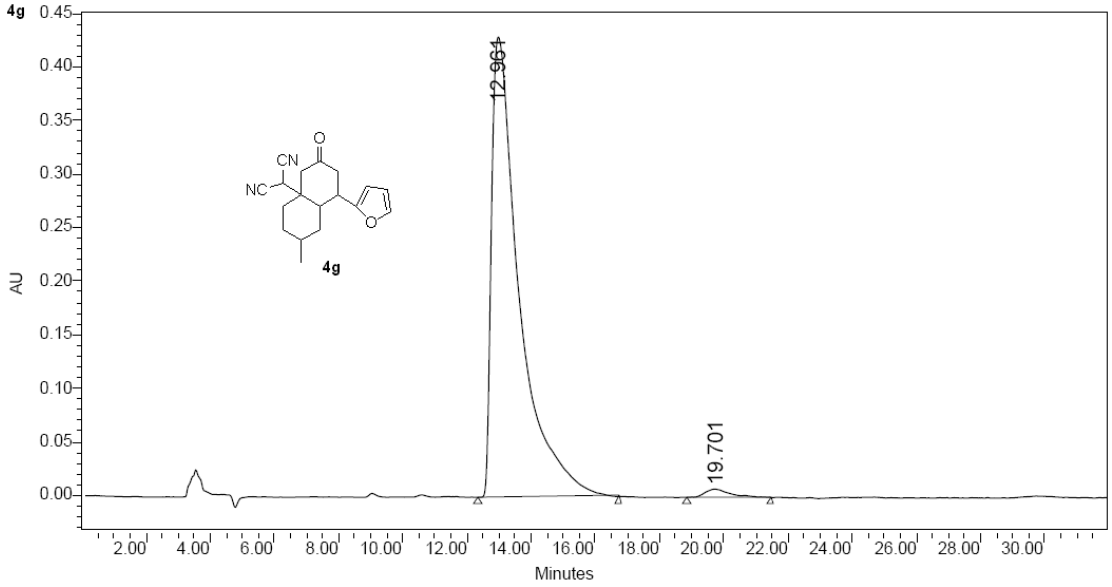
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.572	8451618	98.93	632022	98.87
2	7.401	91605	1.07	7196	1.13


K12-31-1
ppm
7.3746
7.3687
7.2602
6.3310
6.3247
6.3204
6.3140
6.1199
6.1093
4.4015
3.4316
3.4238
3.3935
2.8814
2.8385
2.7946
2.5995
2.5928
2.5536
2.5467
2.5384
2.5229
1.7547
1.7384
1.7172
1.7025
1.5911
1.4036
0.9977
0.9871
0.9592
0.9454
0.8782
0.8571
-0.0053
CN
NC
O
4g
Integral
1.0128
1.0035
1.0000
0.9998
0.9962
2.0404
3.0809
4.1080
1.0530
1.9610
3.2125
ppm
8
6
4
2
0
Current Data Parameters
NAME cyc-2008-48
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080305
Time 20.42
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 128
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 300.1320862 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 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

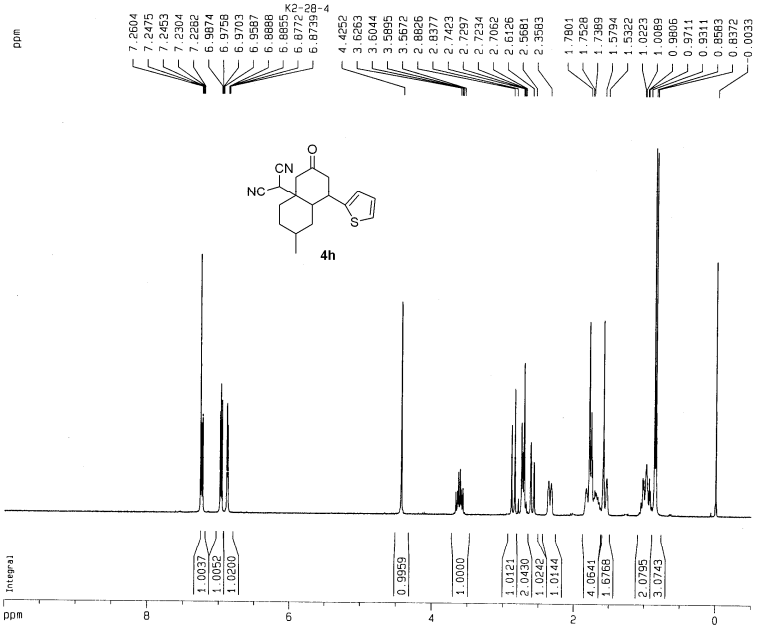

K12-28-3
ppm
204.942
153.734
142.057
111.259
110.435
110.248
106.592
77.423
77.000
76.576
48.620
45.970
44.115
40.327
37.310
31.364
31.033
29.564
28.443
24.943
21.985
CN
NC
O
4g
ppm
200
150
100
50
0
Current Data Parameters
NAME cyc-2008-16
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080110
Time 10.42
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 972
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 75.4677540 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 220.500 ppm
F1 16640.64 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 11.30000 ppm/cm
HZCM 852.78558 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	13.448	41228525	50.56	566943	55.40
2	20.448	40318277	49.44	456503	44.60



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.961	25726409	98.42	429590	98.25
2	19.701	411815	1.58	7633	1.75



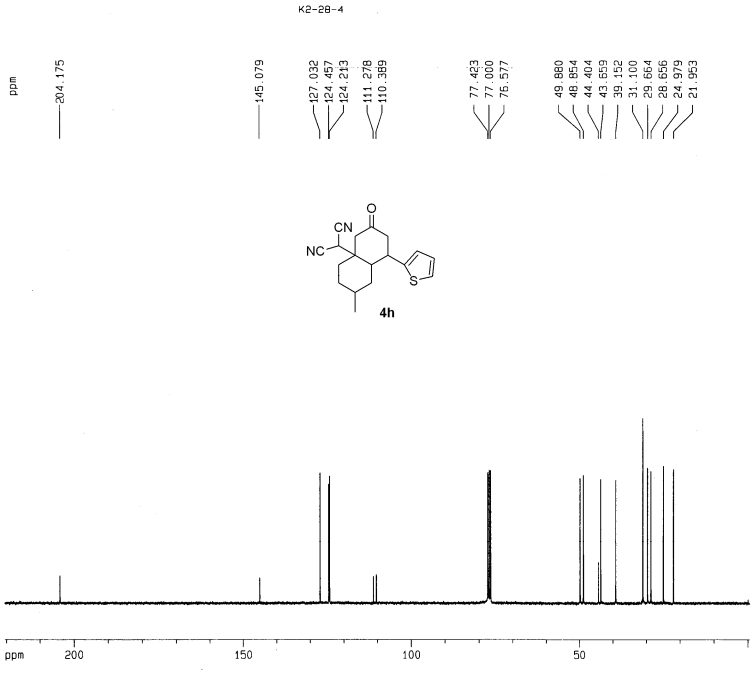
Current Data Parameters
NAME cyc-2008-64
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080314
Time 10.07
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
SFO1 300.1320882 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 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-2008-64
EXPNO 2
PROCNO 1

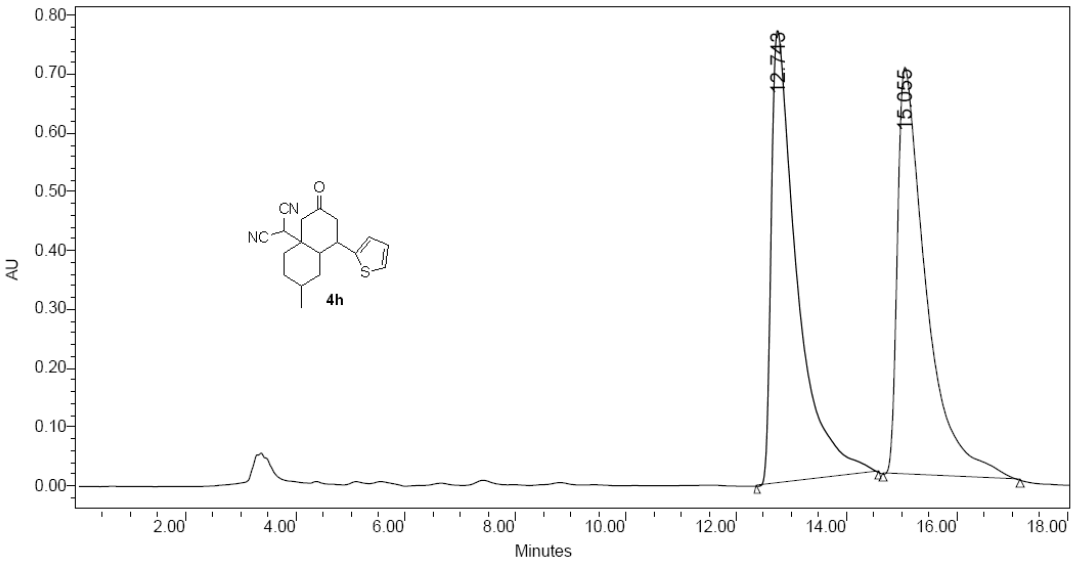
F2 - Acquisition Parameters
Date_ 20080319
Time 2.50
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 6144
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

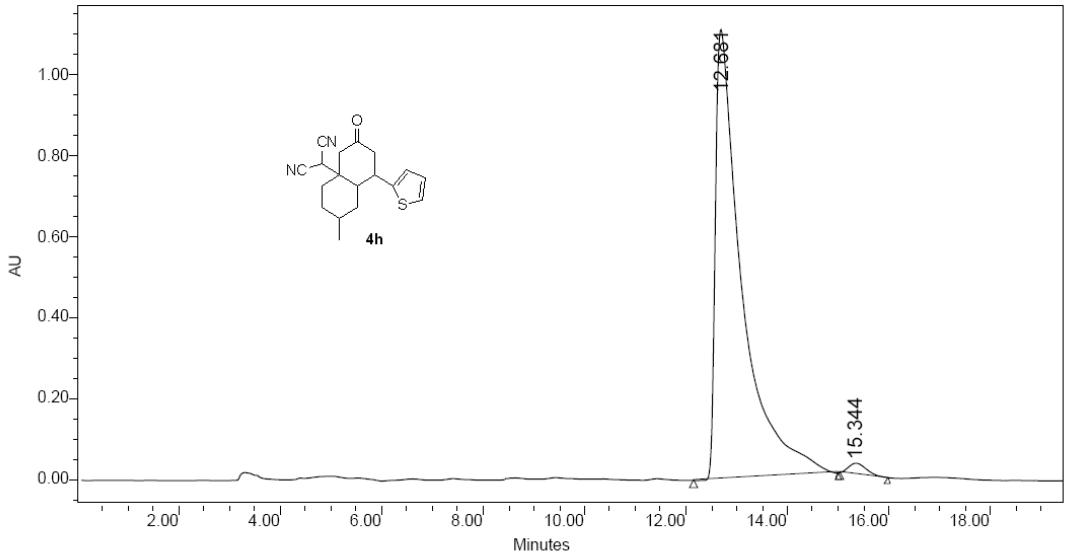
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677509 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

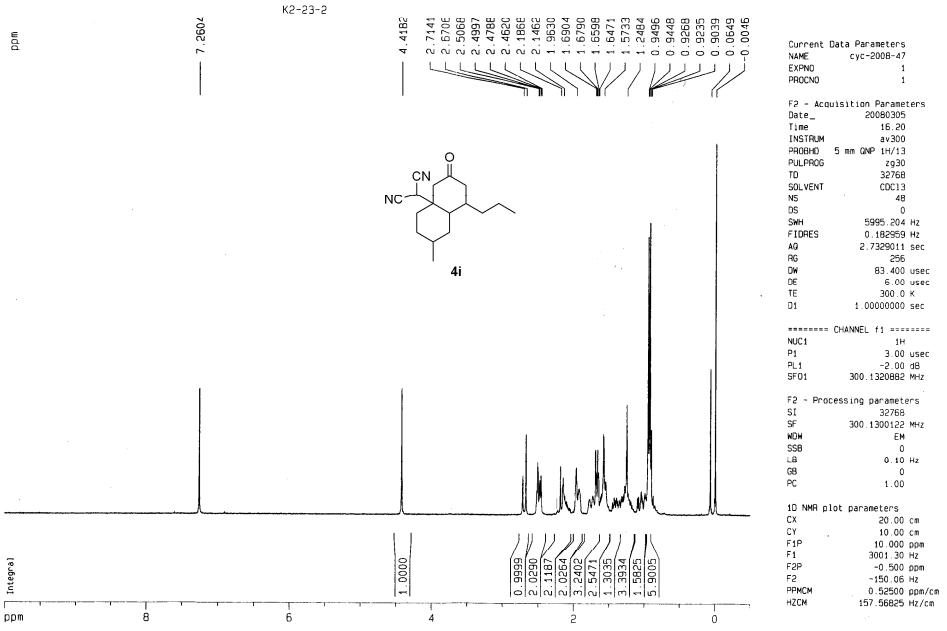
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91864 Hz/cm

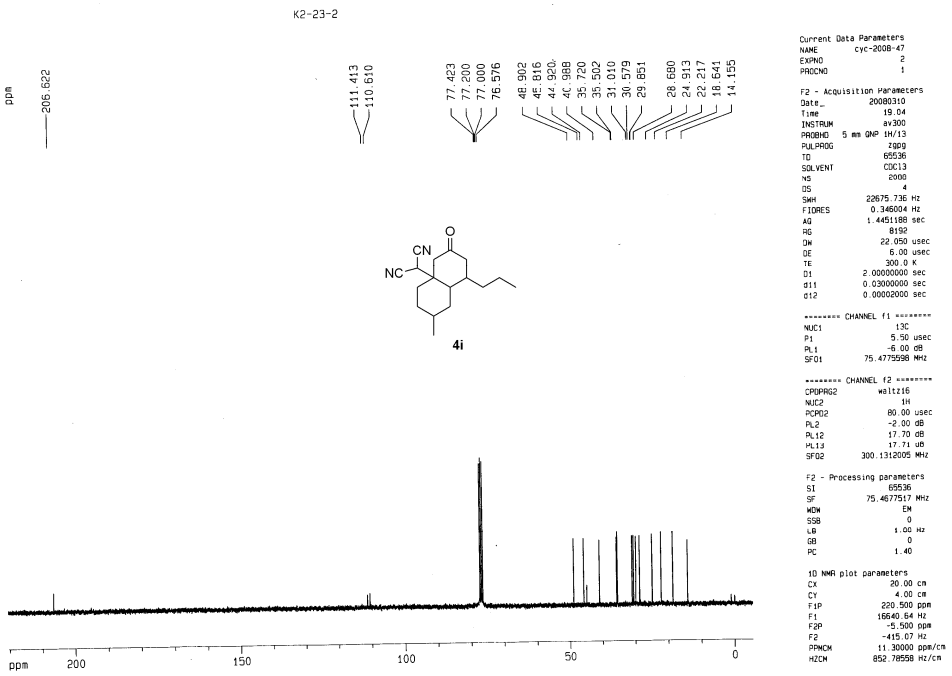


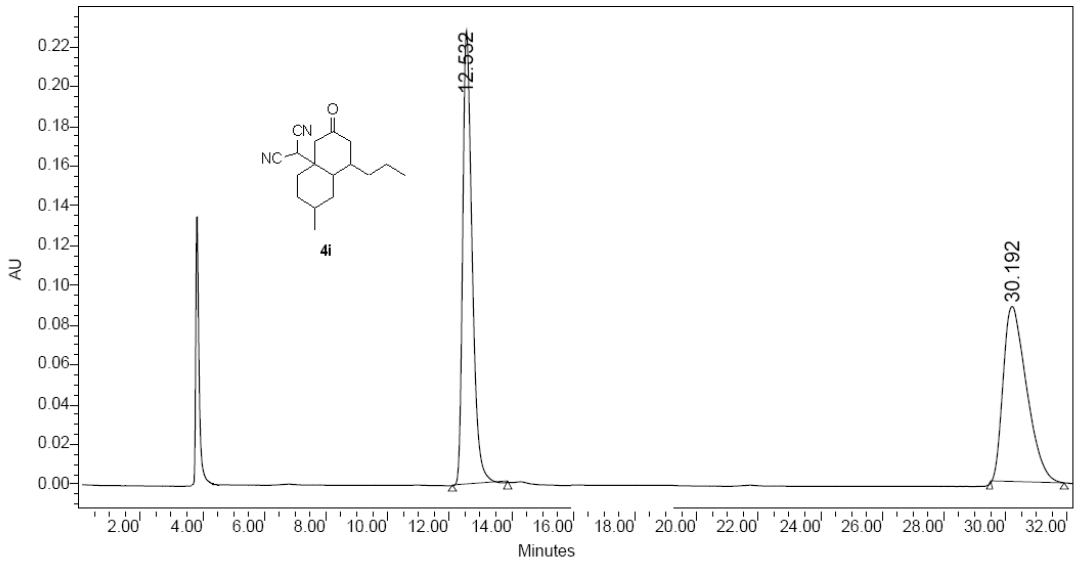
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.743	25211636	50.03	772033	52.67
2	15.055	25182504	49.97	693726	47.33



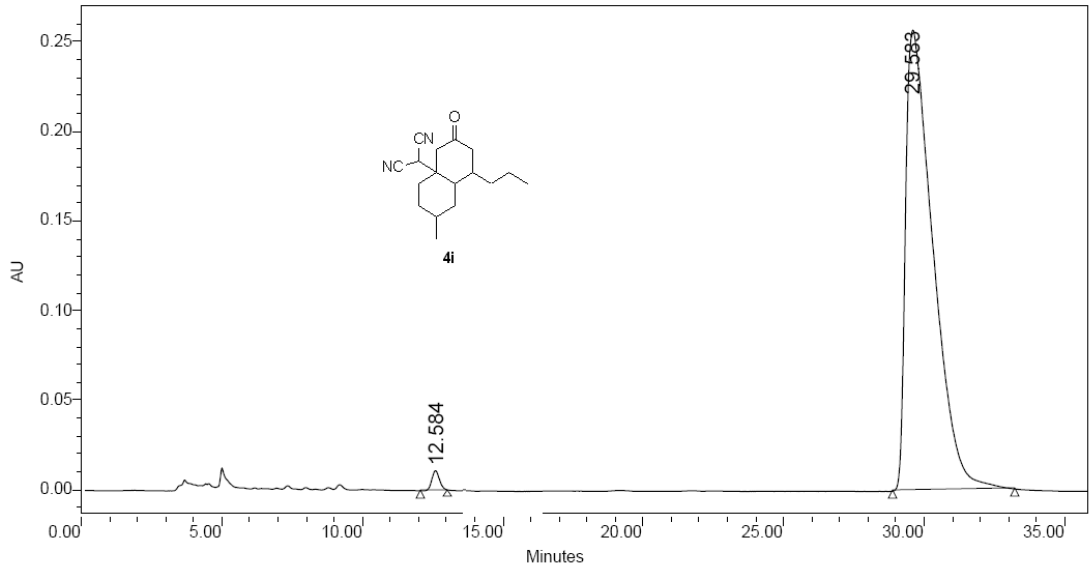
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.681	40242363	98.36	1111046	97.60
2	15.344	669303	1.64	27371	2.40


K2-23-2
ppm
7.2604
4.4186
2.7141
2.6706
2.5066
2.4997
2.4788
2.4626
2.1965
2.1462
1.9930
1.6904
1.6790
1.6598
1.6471
1.5733
1.2484
0.9496
0.9448
0.9268
0.9235
0.9039
0.0549
-0.0045
CN
NC
O
4i
Integral
1.0000
0.9999
2.0290
2.1187
2.0264
3.2402
2.5471
1.3035
3.3934
1.5825
5.9005
ppm
8
6
4
2
0
Current Data Parameters
NAME cyc-2008-47
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080305
Time 16.20
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 48
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 300.1300122 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

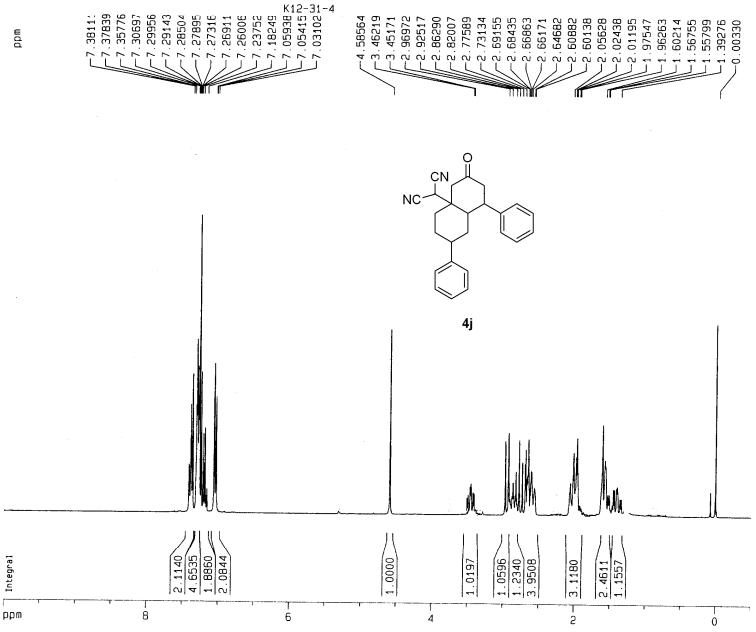

K2-23-2
ppm
206.622
111.413
110.610
77.423
77.200
77.000
76.576
48.902
45.816
44.920
40.988
35.720
35.502
31.010
30.579
29.851
28.680
24.913
22.217
18.641
14.155
CN
NC
O
4i
ppm
200
150
100
50
0
Current Data Parameters
NAME cyc-2008-47
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080310
Time 19.04
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2000
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 75.4677517 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 220.500 ppm
F1 16640.64 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 11.30000 ppm/cm
HZCM 852.78558 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.532	4838848	50.65	228339	72.03
2	30.192	4715155	49.35	88674	27.97



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	12.584	207333	1.17	10970	4.10
2	29.583	17454122	98.83	256750	95.90




```
Current Data Parameters
NAME                  cyc-2008-15
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_               20080108
Time                      20.05
INSTRUM                   av300
PROBHD   5 mm QNP 1H/13
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                         128
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               300.1320884 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                       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
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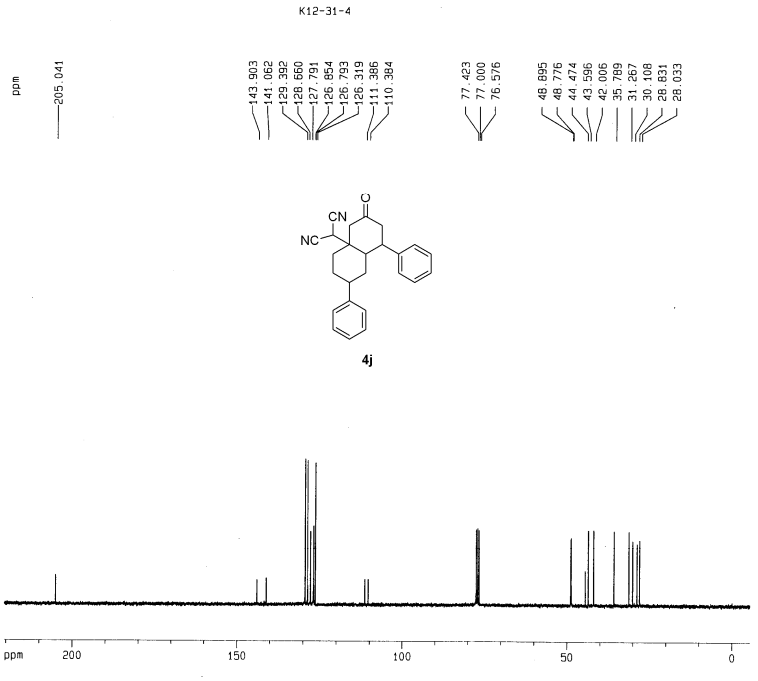


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Current Data Parameters
NAME                  cyc-2008-15
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_               20080110
Time                      13.21
INSTRUM                   av300
PROBHD   5 mm QNP 1H/13
PULPROG                   zgpg
TD                       65536
SOLVENT                  CDCl3
NS                         512
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                 75.4677548 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                   220.500 ppm
F1                   16640.64 Hz
F2P                    -5.500 ppm
F2                    -415.07 Hz
PPMCM                11.30000 ppm/cm
HZCM                852.78564 Hz/cm
```
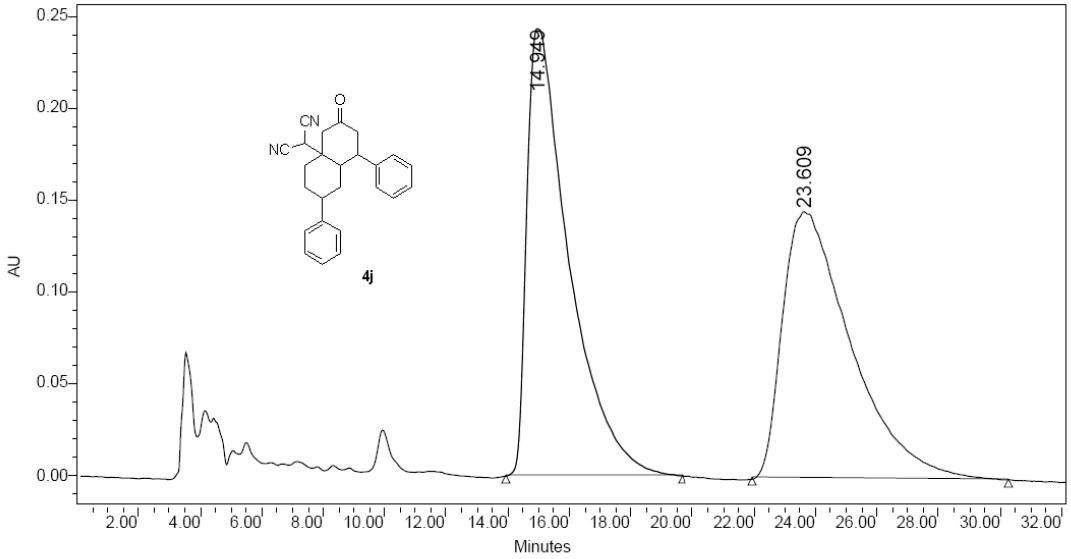
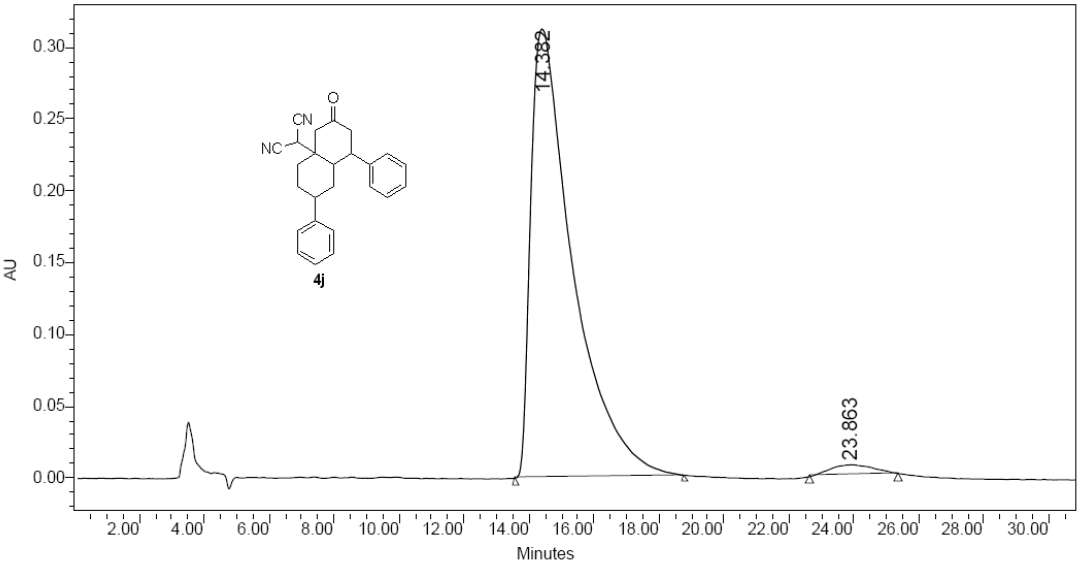
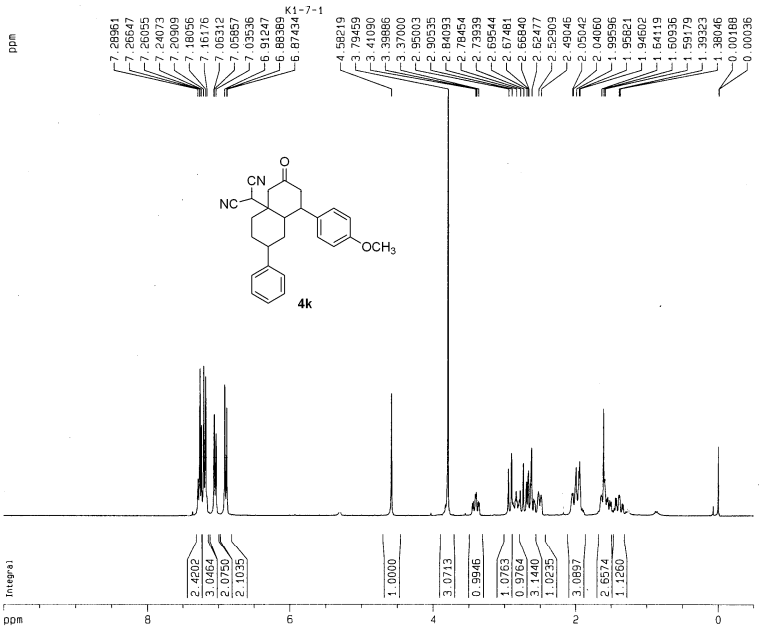



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.949	23264927	50.79	243390	62.60
2	23.609	22537990	49.21	145401	37.40



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.382	28033192	97.65	312214	97.75
2	23.863	674434	2.35	7175	2.25



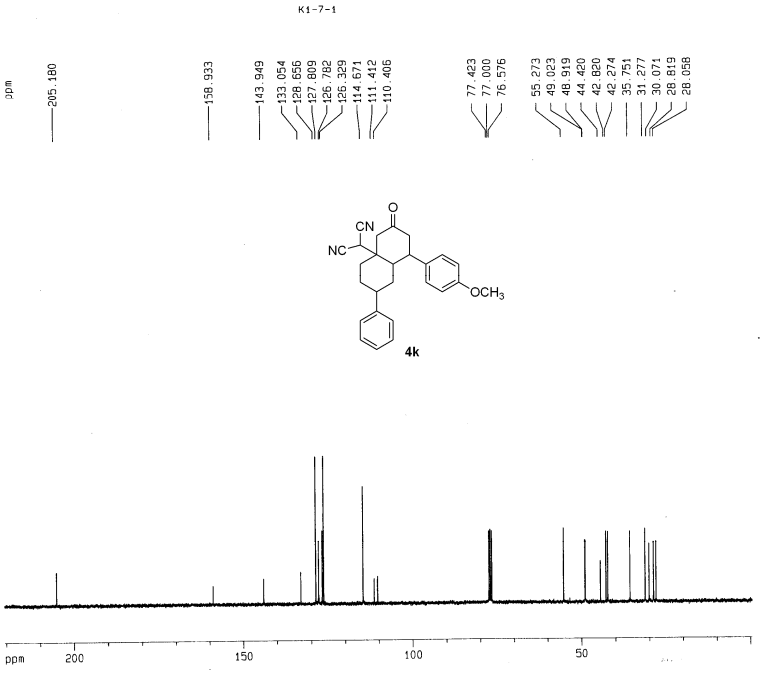
Current Data Parameters
NAME cyc-2008-24
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080116
Time 17.56
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
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 14.00 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-2008-24
EXPNO 2
PROCNO 1

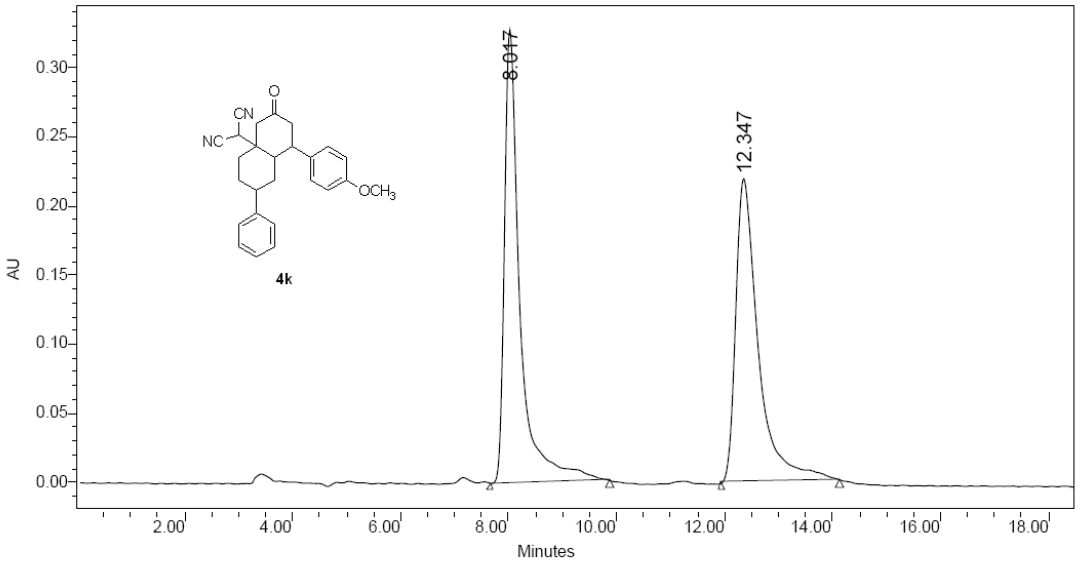
F2 - Acquisition Parameters
Date_ 20080116
Time 19.06
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 636
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

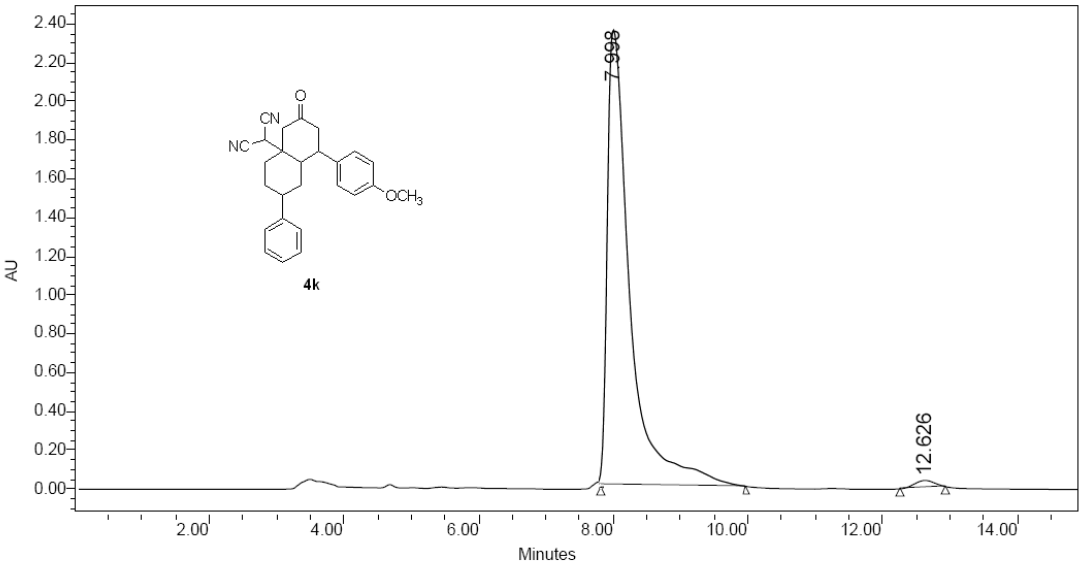
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

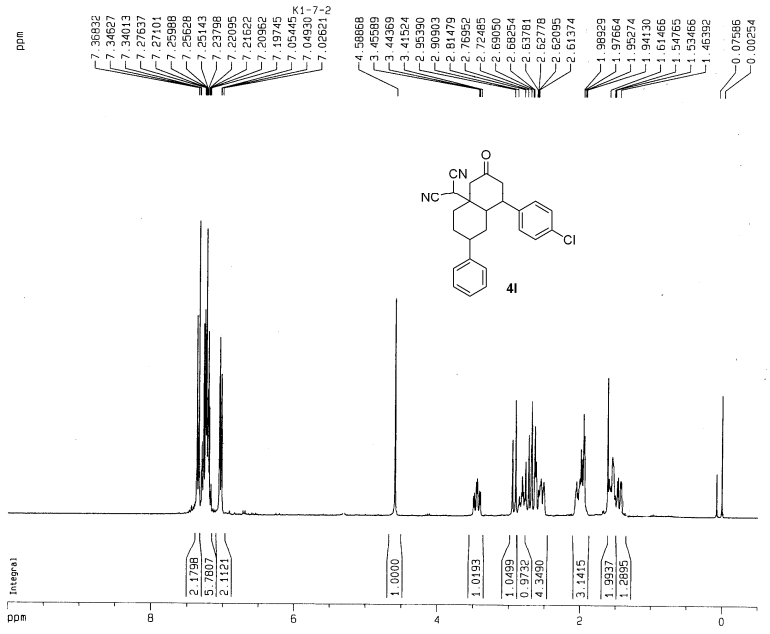
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91870 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	8.017	6610728	50.77	327761	59.88
2	12.347	6410322	49.23	219587	40.12



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.998	54938576	98.58	2346411	98.41
2	12.626	791760	1.42	37869	1.59



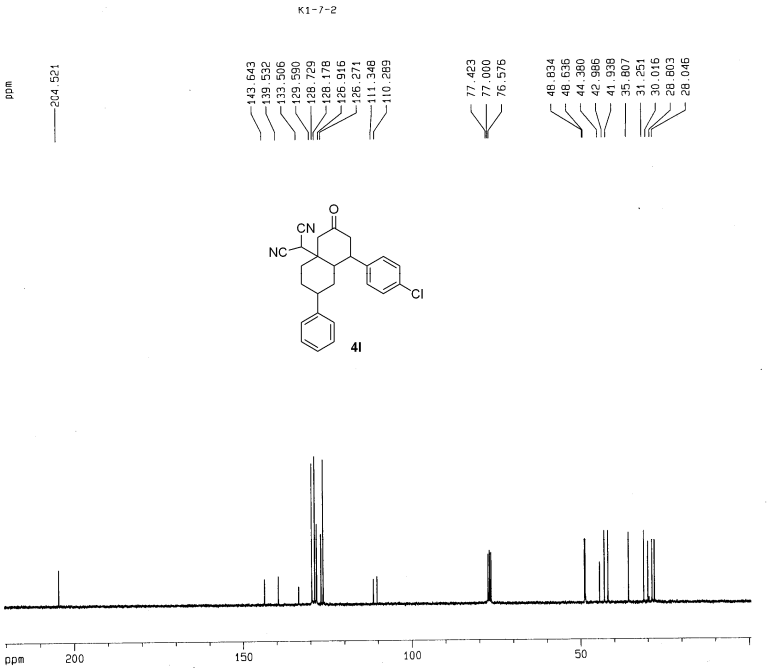
Current Data Parameters
NAME cyc-2008-25
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080116
Time 18.00
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
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 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-2008-25
EXPNO 2
PROCNO 1

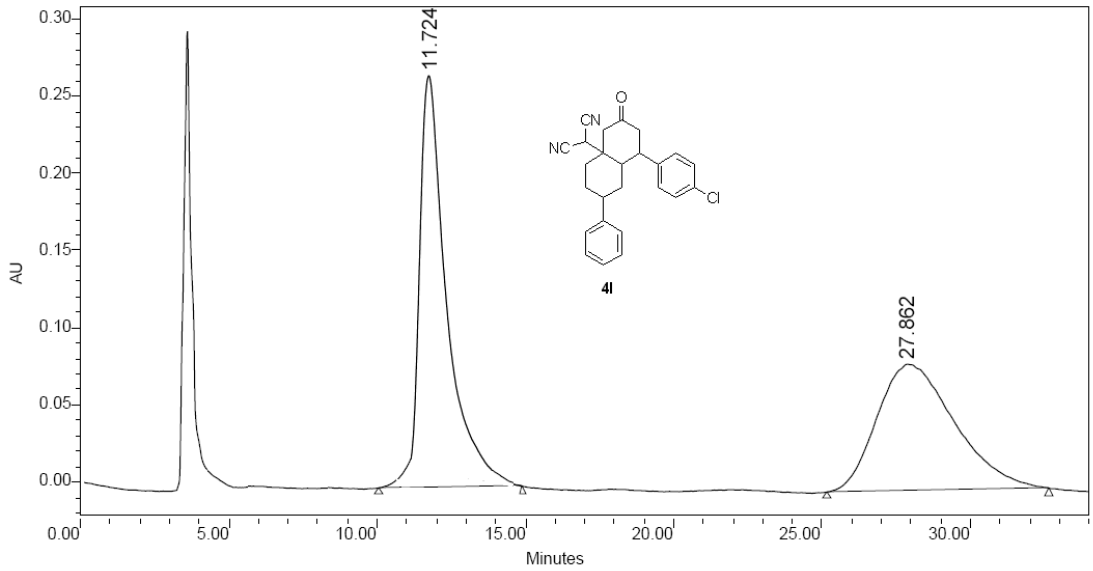
F2 - Acquisition Parameters
Date_ 20080116
Time 20.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 898
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451088 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

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

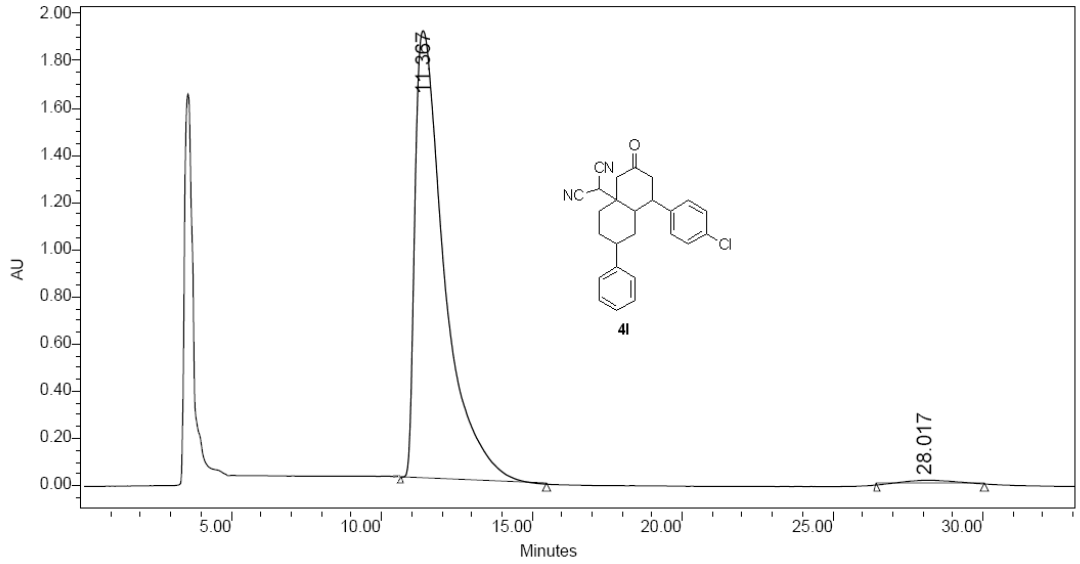
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677598 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

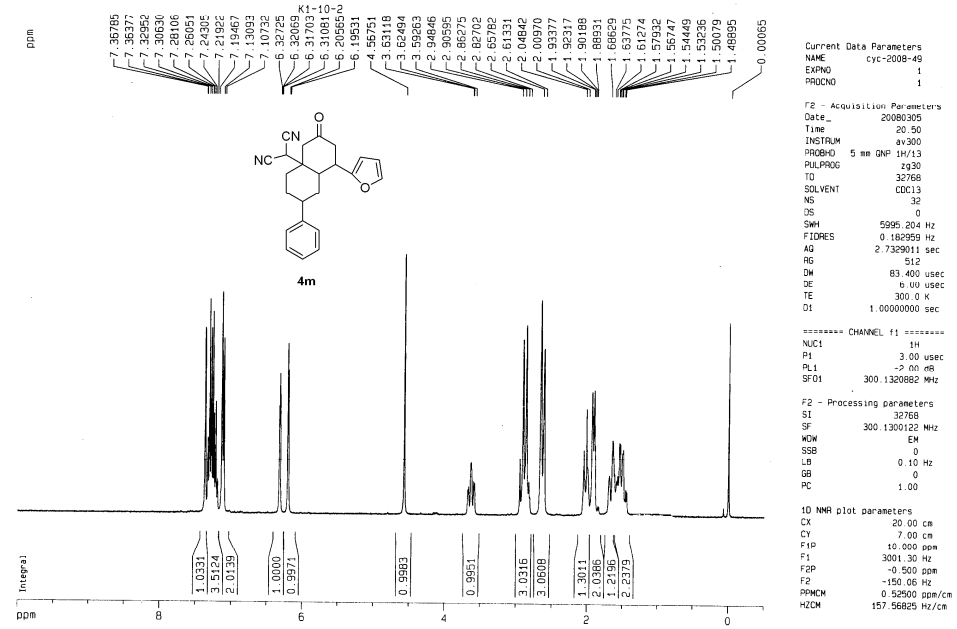
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91870 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.724	15572619	50.99	257507	75.85
2	27.862	14965554	49.01	82008	24.15



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	11.367	128918883	98.33	1893936	99.08
2	28.017	2193458	1.67	17576	0.92



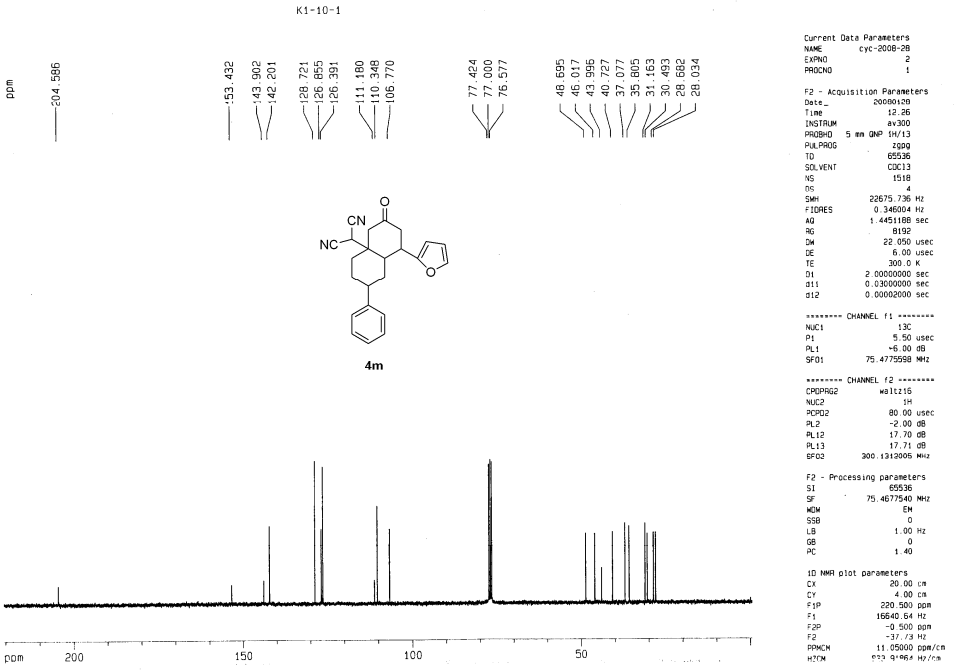
Current Data Parameters
NAME cyc-2008-49
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080305
Time 20.50
INSTRUM av300
PROBHD 5 mm GNP 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 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 7.00 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-2008-28
EXPNO 2
PROCNO 1

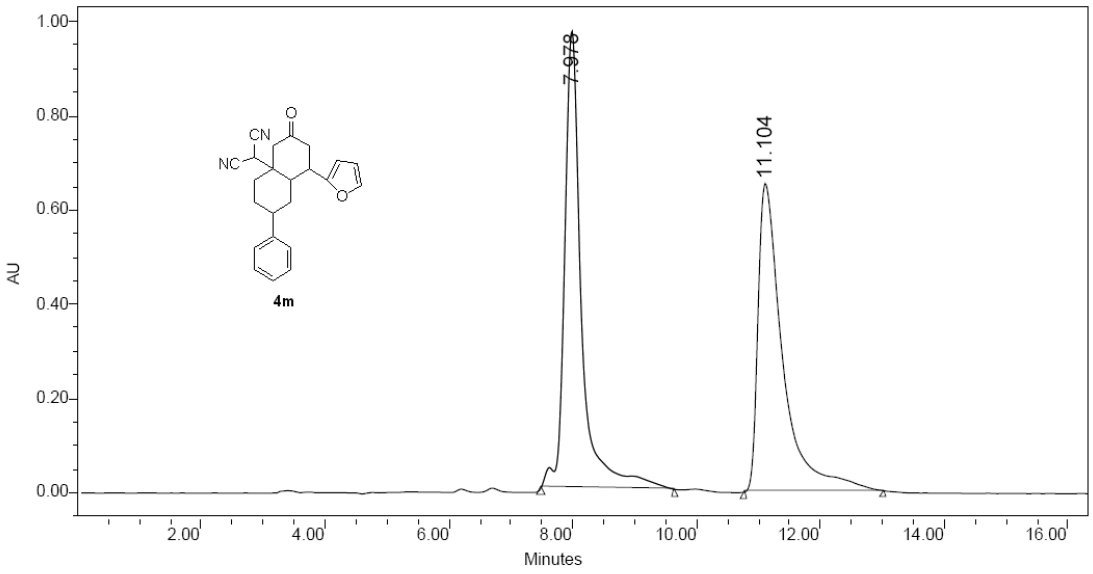
F2 - Acquisition Parameters
Date_ 20080128
Time 12.26
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1518
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

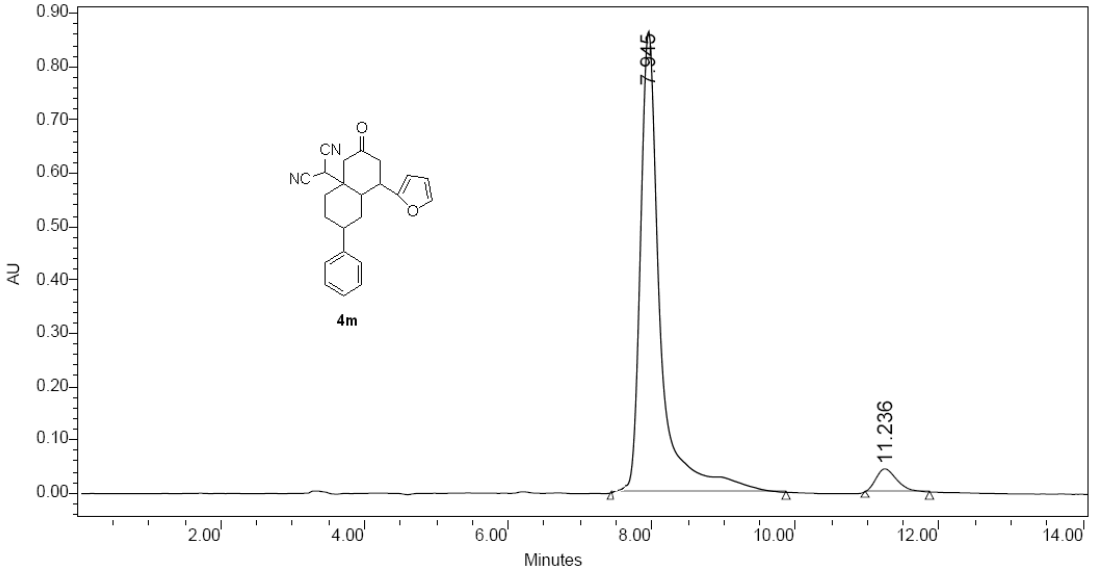
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

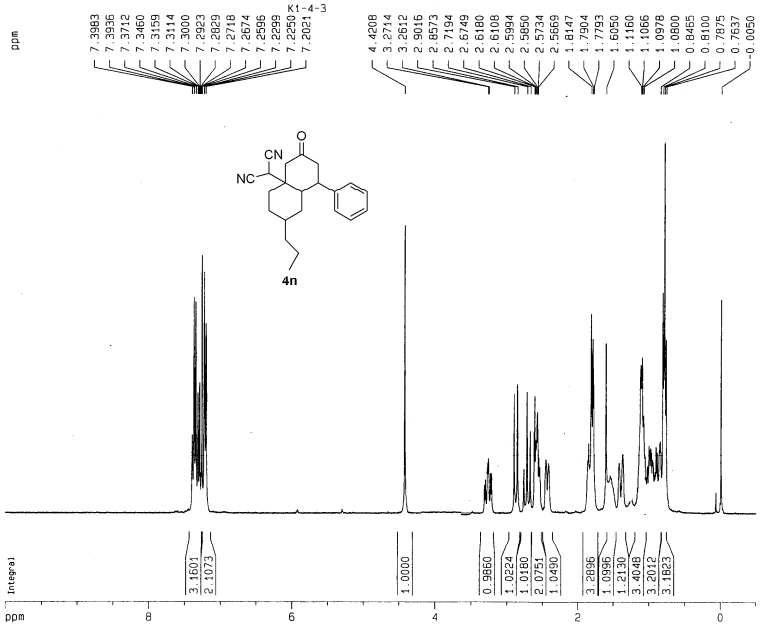
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91962 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.978	18496030	50.30	970561	59.69
2	11.104	18276627	49.70	655309	40.31



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	7.945	16786615	95.09	866043	95.29
2	11.236	867533	4.91	42826	4.71



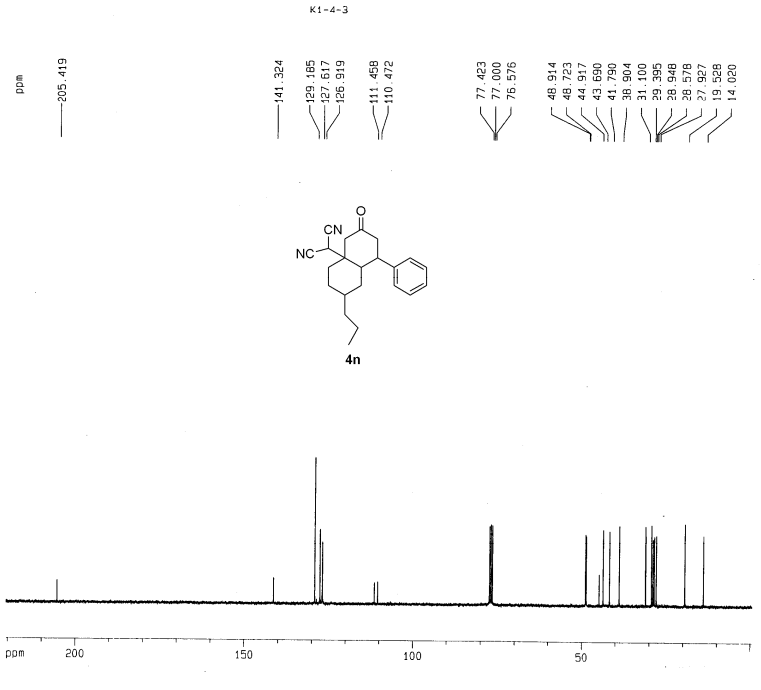
Current Data Parameters
NAME cyc-2008-26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080116
Time 18.08
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
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-2008-26
EXPNO 2
PROCNO 1

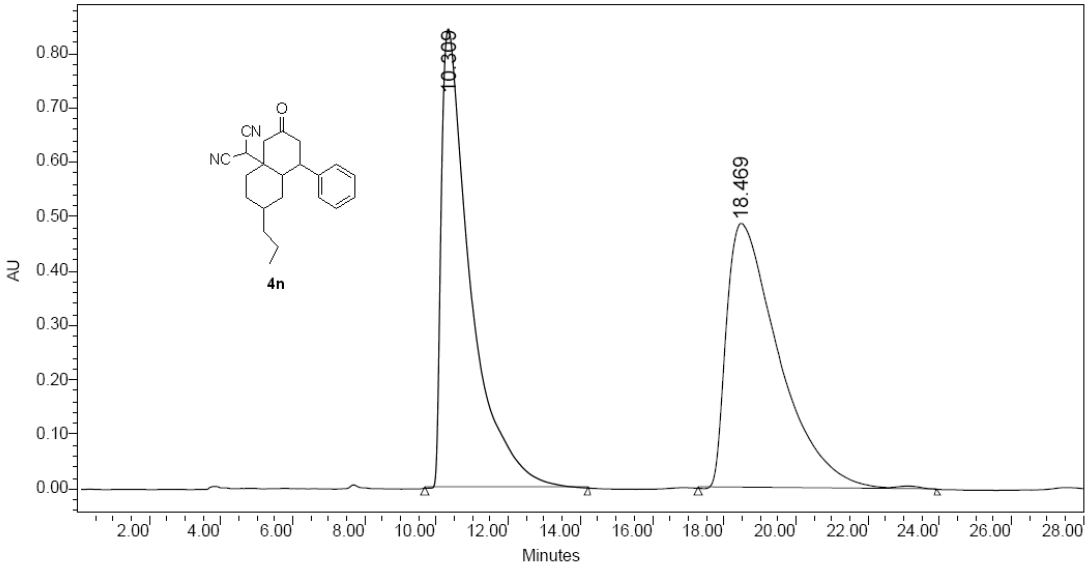
F2 - Acquisition Parameters
Date_ 20080117
Time 9.11
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 882
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775396 MHz

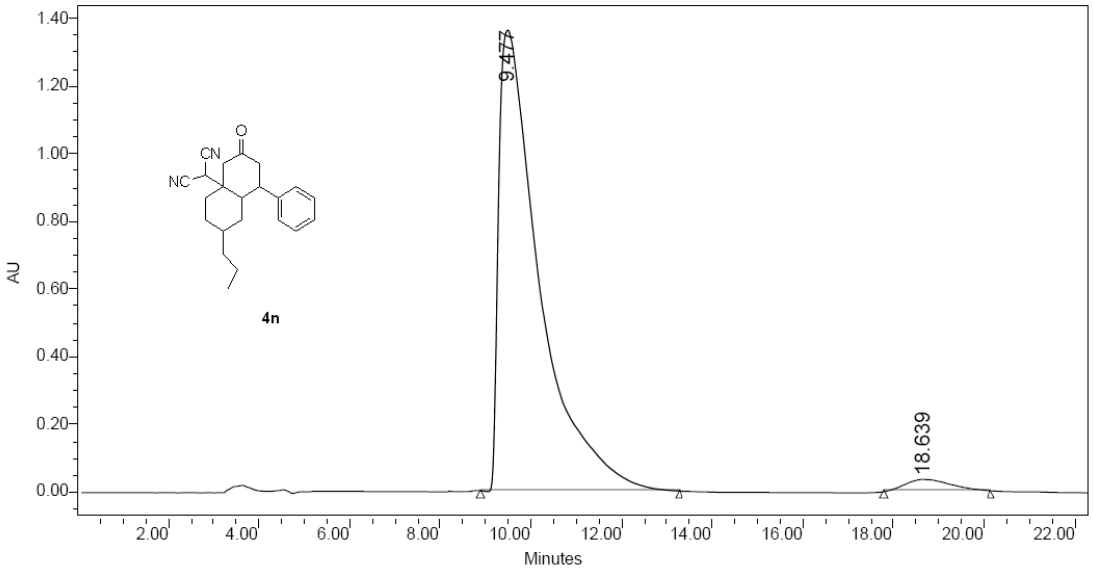
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677549 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

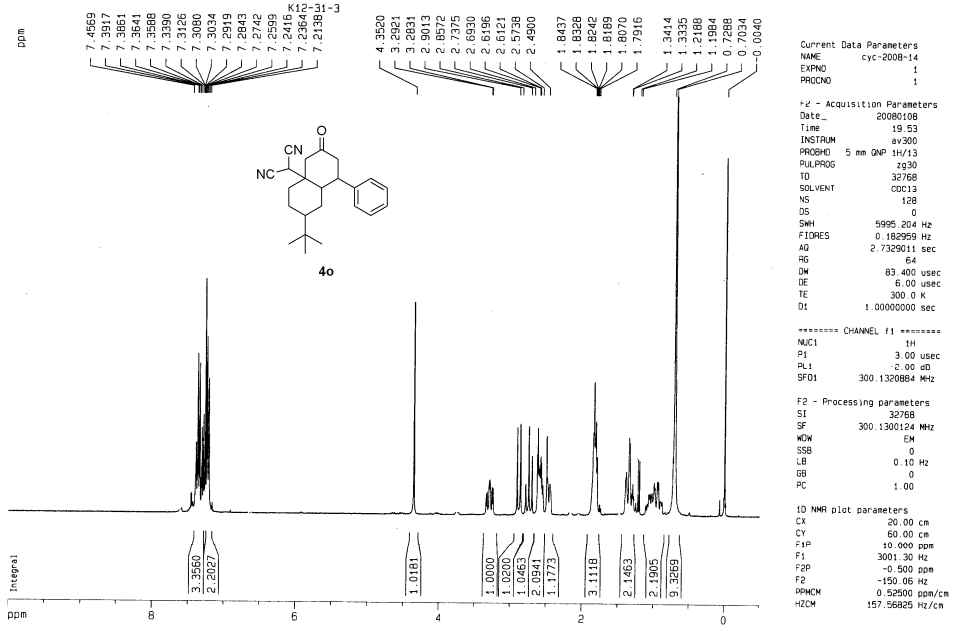
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91870 Hz/cm

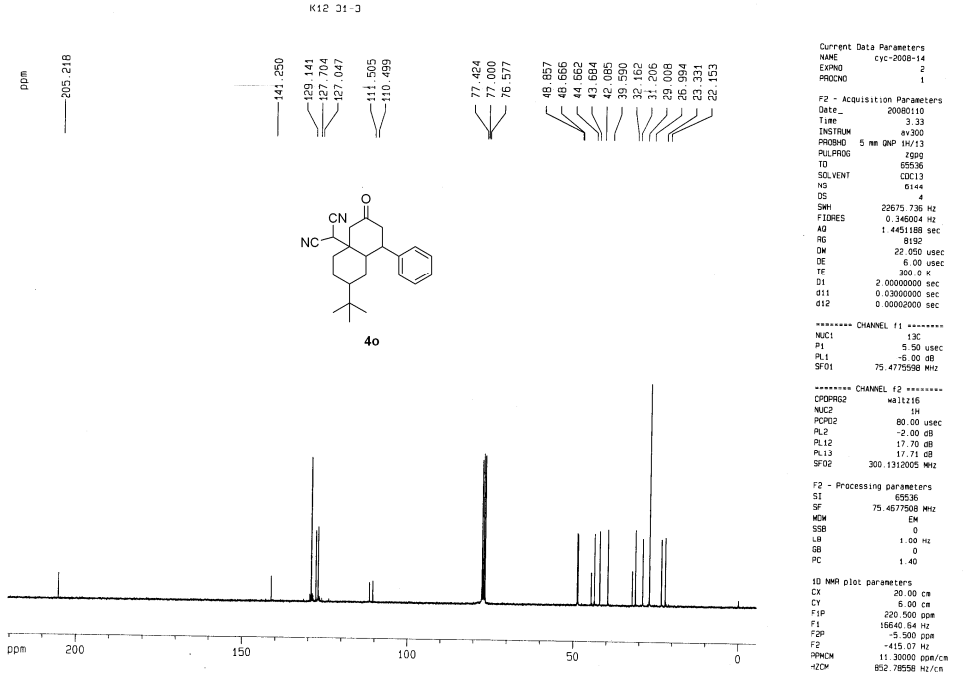


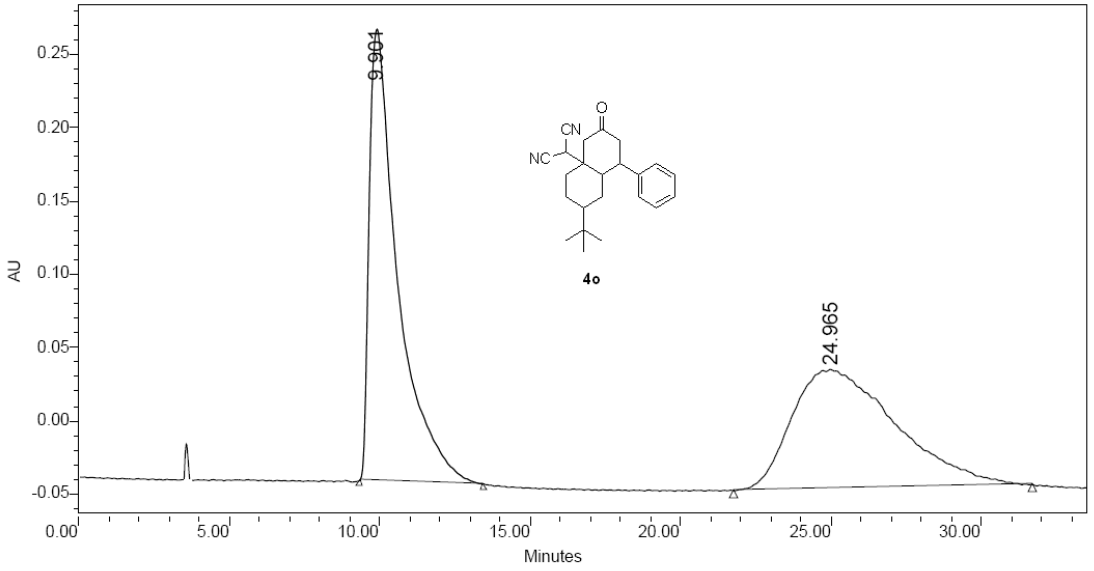
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	10.309	47616241	48.98	843758	63.40
2	18.469	49607848	51.02	487094	36.60



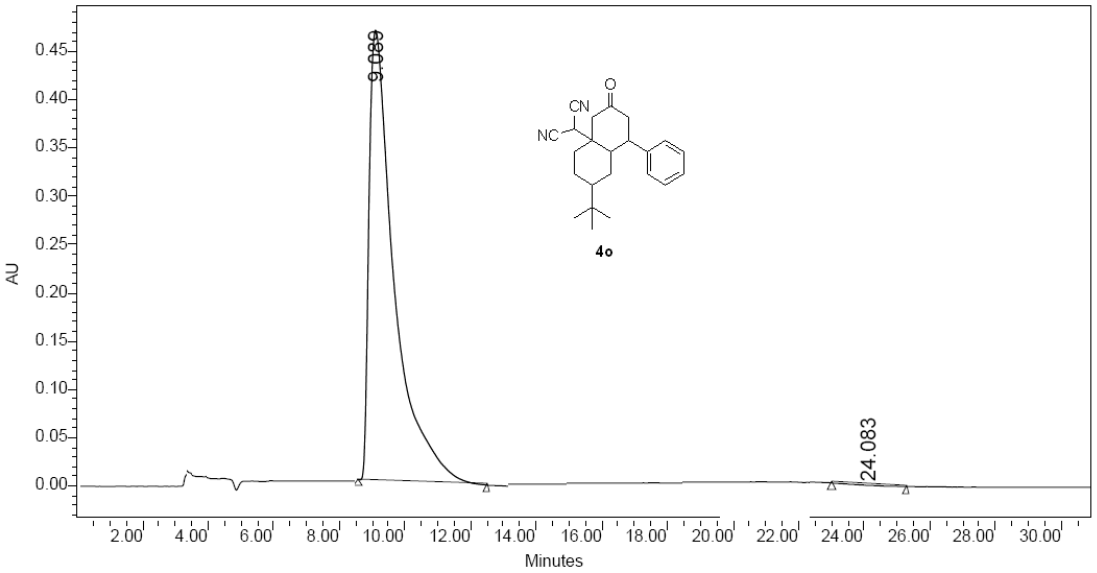
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.477	84958716	97.21	1361432	97.49
2	18.639	2440935	2.79	35012	2.51


K12-31-3
ppm
7.4569
7.3917
7.3861
7.3641
7.3588
7.3390
7.3126
7.3080
7.3034
7.2919
7.2843
7.2742
7.2599
7.2416
7.2364
7.2138
4.3520
3.2921
3.2831
2.9013
2.8572
2.7375
2.6930
2.6196
2.6121
2.5738
2.4900
1.8437
1.8328
1.8242
1.8189
1.8070
1.7916
1.3414
1.3335
1.2188
1.1984
0.7288
0.7034
-0.0040
O
CN
NC
4o
Integral
3.3560
2.2627
1.0181
1.0000
1.0200
1.0463
2.0941
1.1773
3.1118
2.1463
2.1905
9.3269
ppm
8
6
4
2
0
Current Data Parameters
NAME cyc-2008-14
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080108
Time 19.53
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 128
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 300.1320884 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 60.00 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

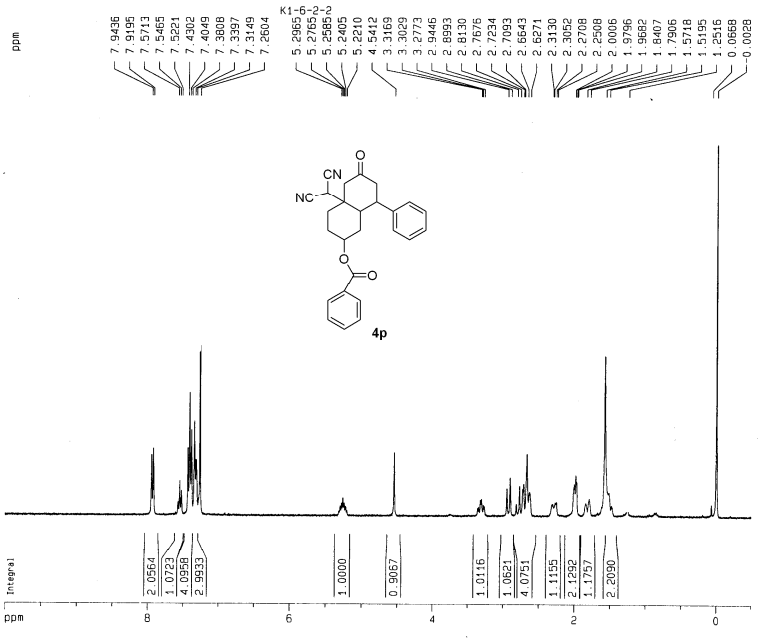

K12 31-3
ppm
205.218
141.250
129.141
127.704
127.047
111.505
110.499
77.424
77.000
76.577
48.857
48.666
44.662
43.684
42.085
39.590
32.152
31.206
29.008
26.994
23.331
22.153
O
CN
NC
4o
ppm
200
150
100
50
0
Current Data Parameters
NAME cyc-2008-14
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080110
Time 3.33
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 6144
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 75.4677508 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 11.30000 ppm/cm
HZCM 852.78558 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.901	20233980	50.74	307450	79.04
2	24.965	19642284	49.26	81509	20.96



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	9.089	24912430	99.74	466384	99.82
2	24.083	64713	0.26	-855	0.18



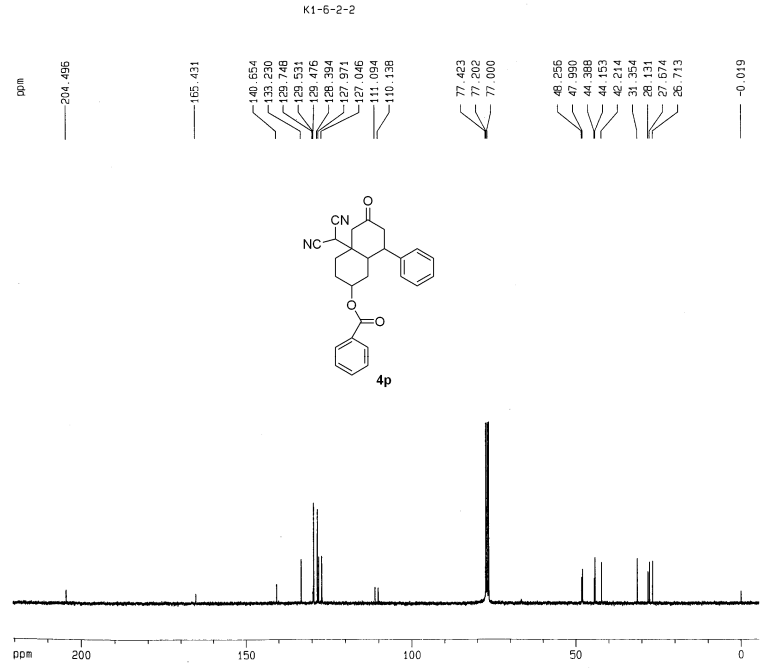
Current Data Parameters
NAME cyc-2008-50
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080305
Time 20.54
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 2.7328011 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 300.1300122 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-2008-50
EXPNO 2
PROCNO 1

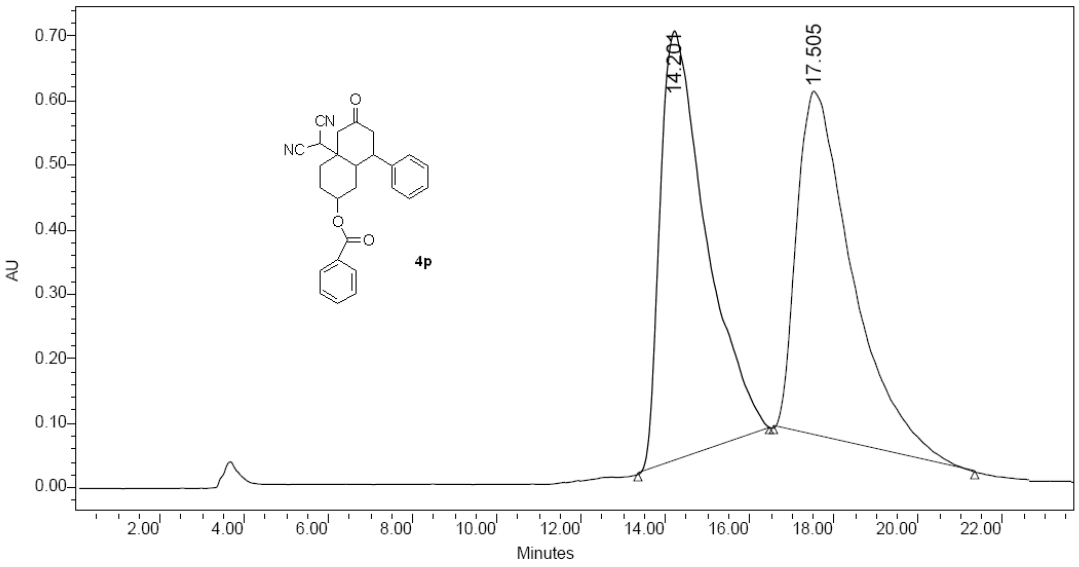
F2 - Acquisition Parameters
Date_ 20080306
Time 22.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 11580
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

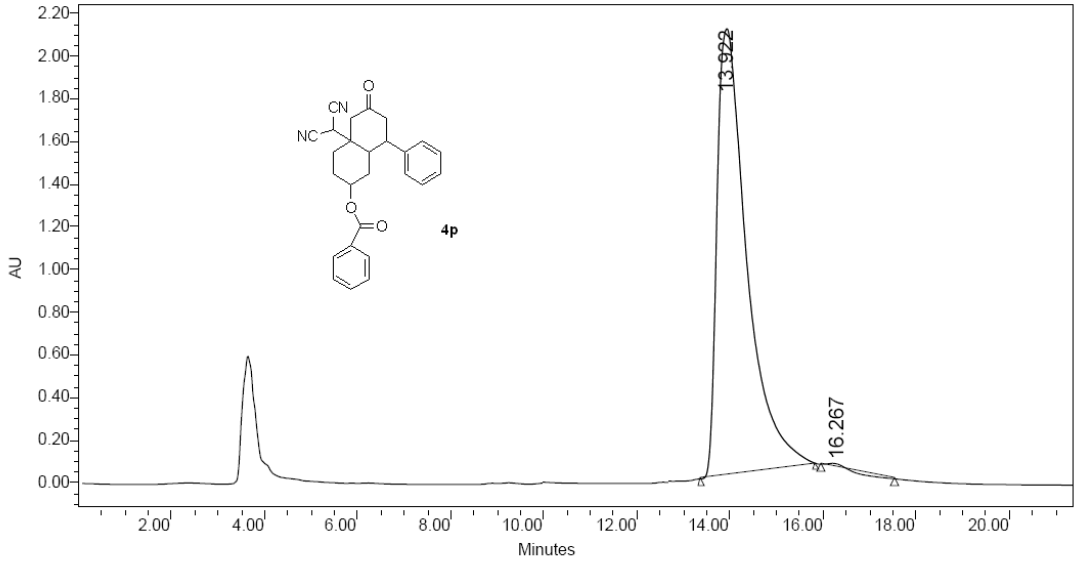
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677504 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

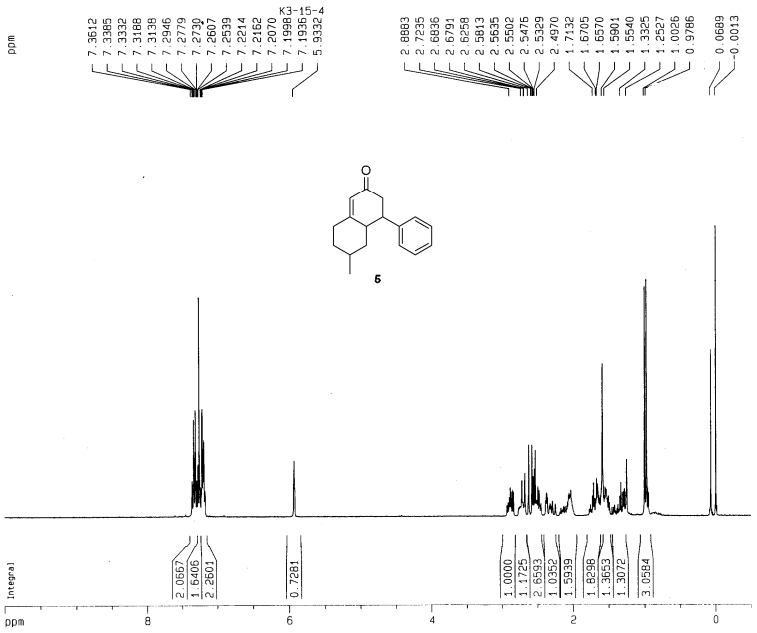
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 11.30000 ppm/cm
HZCM 852.78558 Hz/cm



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	14.201	50472557	50.48	667021	55.53
2	17.505	49505696	49.52	534243	44.47



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	13.922	93224575	99.23	2092995	99.23
2	16.267	725414	0.77	16186	0.77



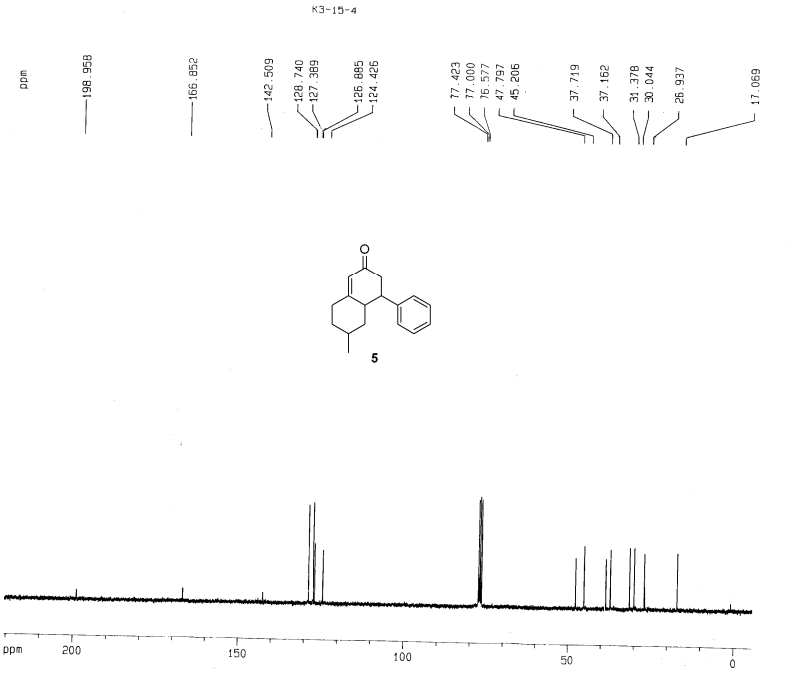
Current Data Parameters
NAME cyc-2008-85
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080414
Time 19.26
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 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 300.1300121 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-2008-85
EXPNO 2
PROCNO 1

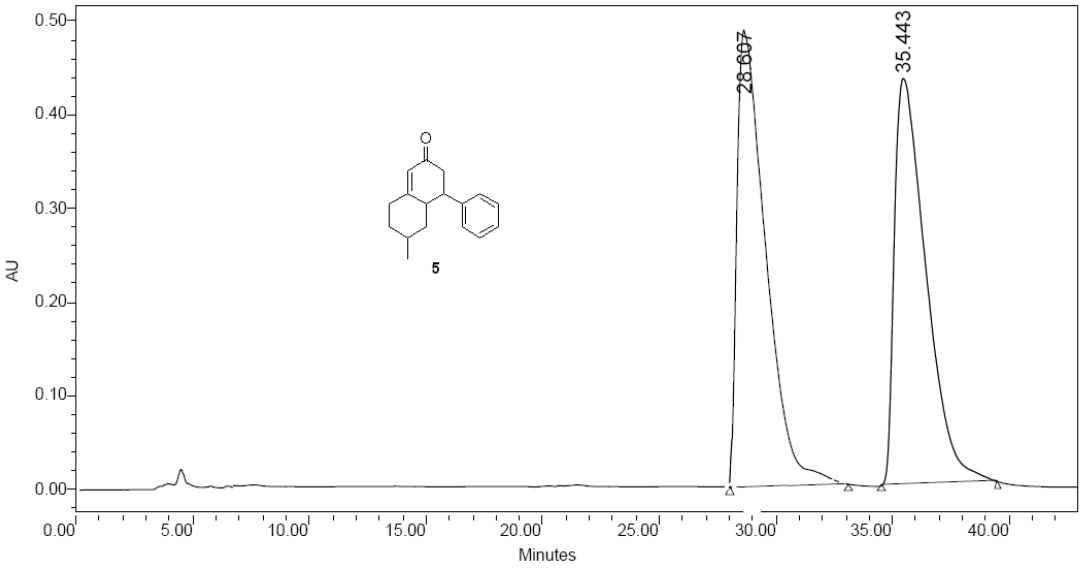
F2 - Acquisition Parameters
Date_ 20080422
Time 13.58
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

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

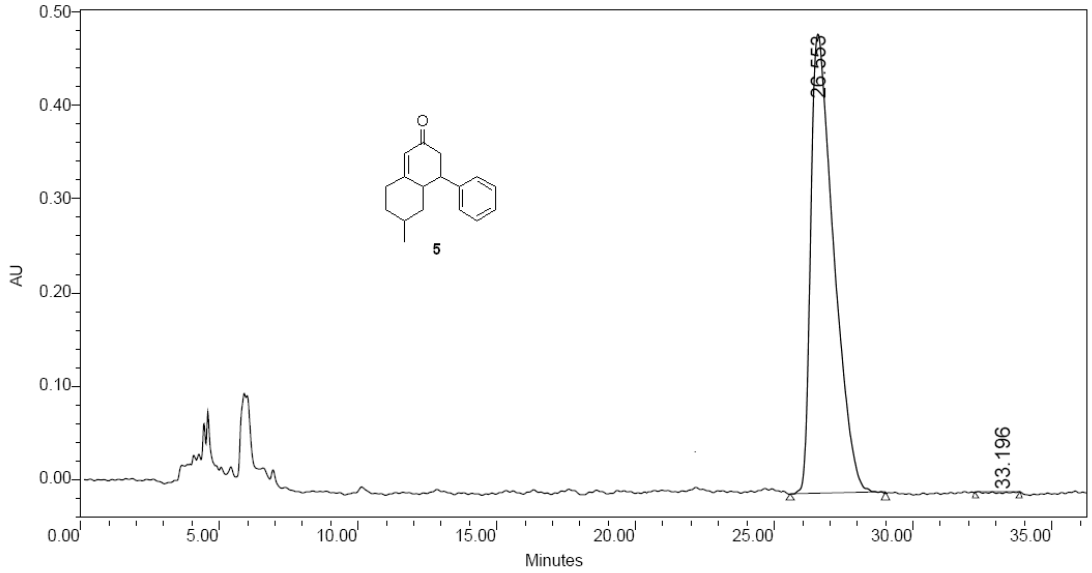
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.76 dB
PL13 17.71 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 65536
SF 75.4677523 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

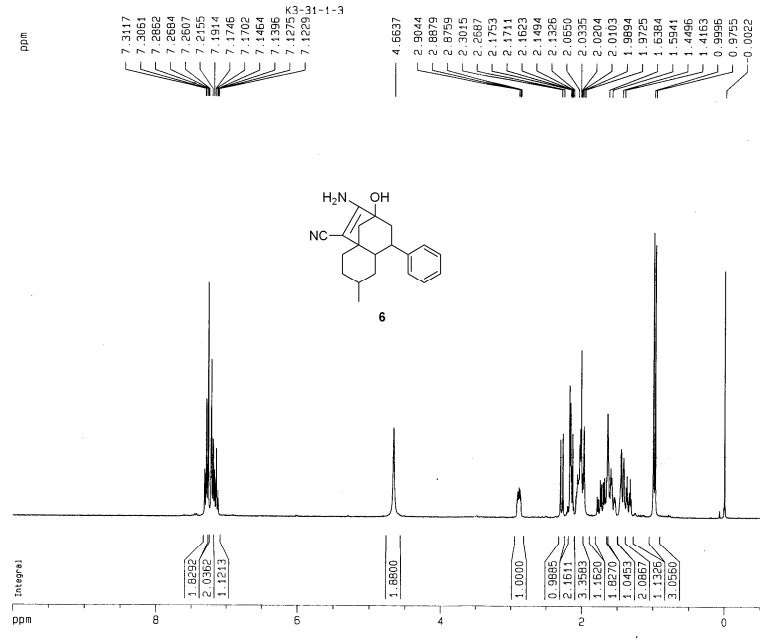
1D NMR plot parameters
CX 20.00 cm
CY 3.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 11.30000 ppm/cm
HZCM 852.78558 Hz/cm



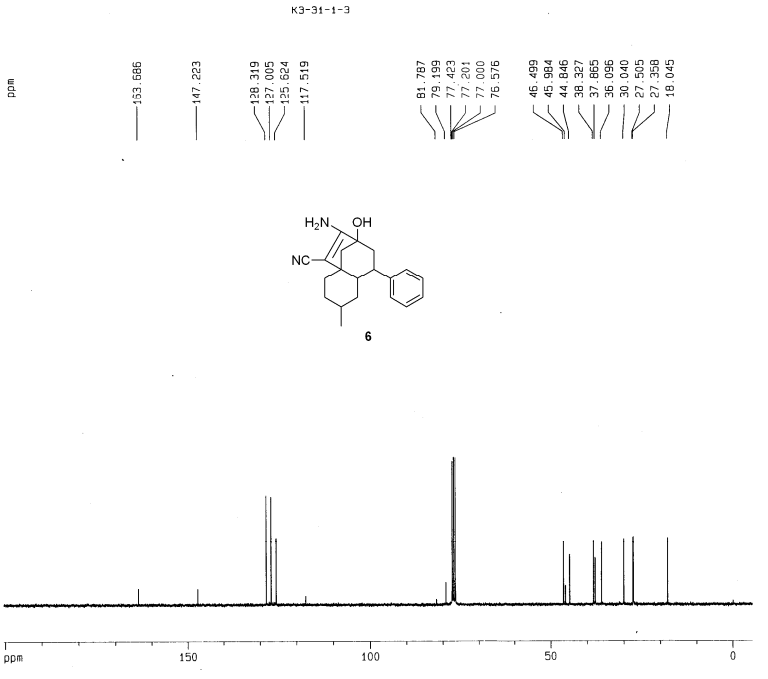
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	28.607	40412122	49.74	478889	52.52
2	35.443	40834607	50.26	433003	47.48



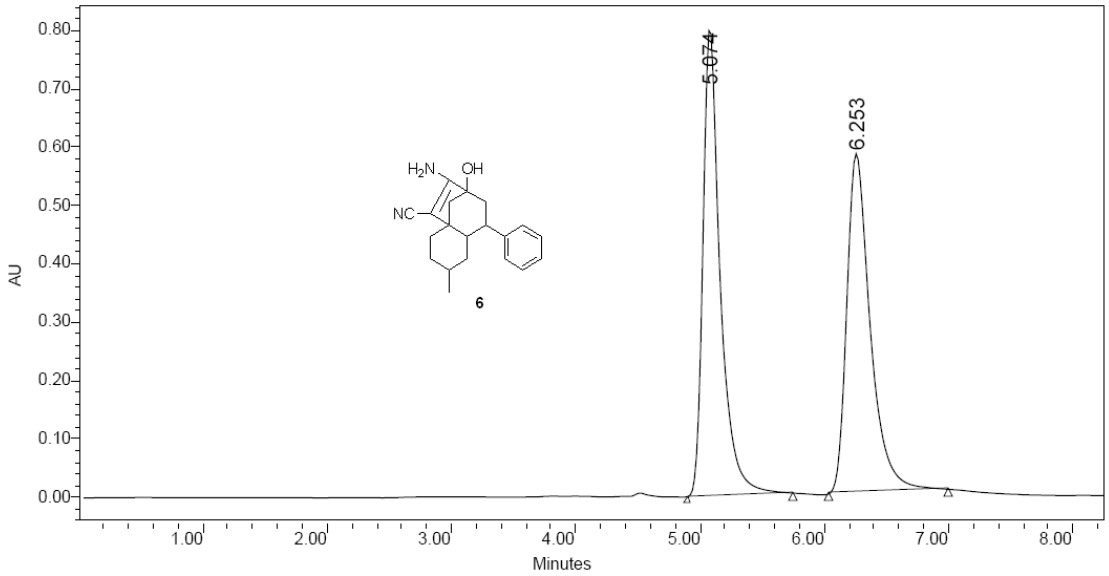
	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	26.553	28448502	99.73	492335	99.65
2	33.196	76860	0.27	-1752	0.35



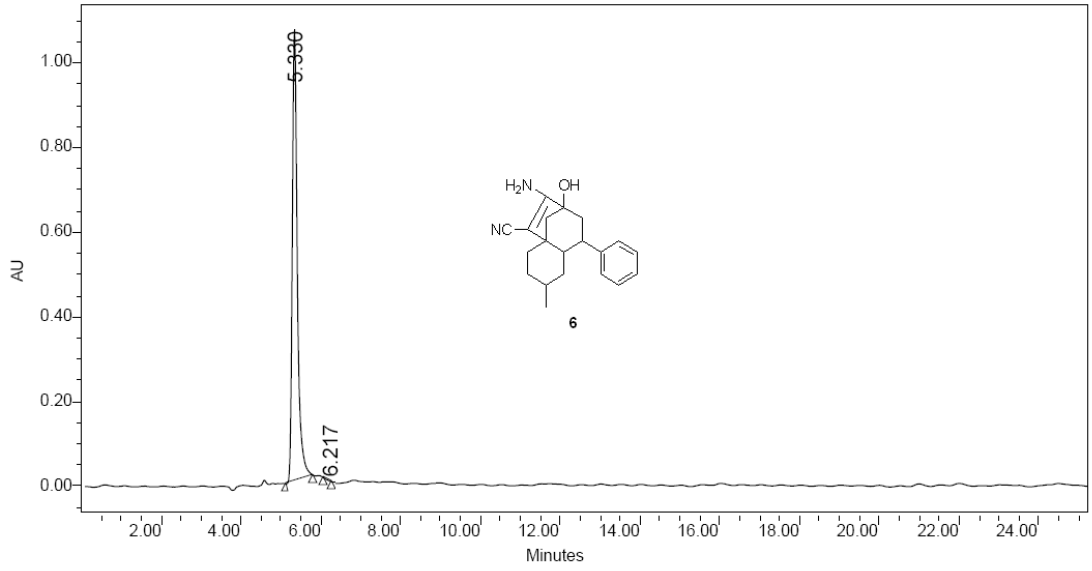

```
Current Data Parameters
NAME                  cyc-2008-74
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_               20080403
Time                      12.11
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                         128
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                 300.1300122 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-2008-74
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_               20080403
Time                      12.40
INSTRUM                   av300
PROBHD     5 mm QNP 1H/13
PULPROG                   zgpg
TD                       65536
SOLVENT                  CDCl3
NS                        2574
DS                           4
SWH                22675.736 Hz
FIDRES              0.346004 Hz
AQ                  1.445188 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                75.4677516 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                   200.500 ppm
F1                   15131.28 Hz
F2P                   -6.500 ppm
F2                    -415.07 Hz
PPMCM                10.30000 ppm/cm
HZCM                777.31787 Hz/cm
```



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.074	7745374	50.31	802899	58.01
2	6.253	7649325	49.69	581220	41.99



	RT (min)	Area (V *sec)	% Area	Height (V)	% Height
1	5.330	9421184	99.91	1068094	99.91
2	6.217	8838	0.09	-919	0.09