

Supporting Information for

Asymmetric Nitroallylation of Arylboronic Acids with Nitroallyl Acetates Catalyzed by Chiral Rhodium Complexes and Its Application in a Concise Total Synthesis of Optically Pure (+)- γ -Lycorane

Lin Dong, Yan-Jun Xu, Lin-Feng Cun, Xin Cui, Ai-Qiao Mi, Yao-Zhong Jiang, and Liu-Zhu Gong*

General. All manipulations were carried out under an argon atmosphere. NMR spectra were recorded on a Bruker-300MHz spectrometer. Mass spectra were recorded on VG 7070E. Elemental analysis was recorded on Italy CARCOERBA 1106. HR-MS (ESI) spectra were recorded on Bruker BioTOF Q.

Materials. THF and dioxane were dried with sodium/ benzophenone. Diethyl ether, petroleum ether (PE) and ethyl acetate for column chromatography were distilled before use.

General procedure for the asymmetric nitroallylation of arylboronic acids with nitroallyl acetates catalyzed by chiral rhodium complexes: Under Argon atmosphere, $[\text{Rh}(\text{OH})(\text{COD})]_2$ ¹ (3.4 mg, 15 μmol , 5 mol%), (S)-BINAP (11.2 mg, 18 μmol , 6 mol%) and phenylboronic acid (183 mg, 1.5 mmol, 5 equiv) in 1,4-dioxane(1 mL) was stirred at 50 $^\circ\text{C}$ for 2 h. Substrate **1a**² (0.3 mmol) in 1,4-dioxane (1 mL) was added to the above mixture, and followed by addition of water (200 μL). The reaction mixture was stirred at 50 $^\circ\text{C}$ for 20 h. After evaporation of the solvent, the residue was purified by column chromatography on silica (Diethyl ether/ petroleum ether =1/20) to yield 1-nitro-6-phenylcyclohexene (**2a**) as a white solid (34 mg, 56% yield). m.p. 71.3-72.0 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = +92.5$ ($c = 1.07$, CHCl_3); IR (KBr): γ 3456, 3023, 2946, 2920, 2867, 1663, 1510, 1491, 1451, 1413, 1333, 1077, 938 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz): δ (ppm) 1.55-1.62 (m, 2H), 1.89-1.92 (m, 1H), 2.01-2.03 (m, 1H), 2.40 - 2.49 (m, 2H), 4.29 (brs, 1H), 7.14-7.34 (m, 5H), 7.57 (t, $J = 4.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz): δ (ppm) 16.5, 25.0, 31.5, 39.5, 126.7, 127.1, 128.5, 136.2, 141.9, 150.9; HRMS for $\text{C}_{12}\text{H}_{13}\text{NO}_2$, calcd: 203.0940, found: 203.0928. Anal. calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_2$: C, 70.93; H, 6.41; N, 6.89. Found: C, 70.84; H, 6.44; N, 6.84. Enantiomeric excess: 97%, determined

by chiral GC analysis (CP-Cyclodextrin- 2, 3, 6 -M-19, 0.25 mm×25 m, column temperature = 168 °C (isothermal), inject temperature = 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

Crystal data of **2a**: C₁₂H₁₃NO₂, MW= 203.23, Orthorhombic, space group P2₁2₁2₁, *a*= 6.1206(5), *b*= 12.592(1), *c*= 14.007(1) Å, α = 90°, β = 90°, γ = 90°, *U*= 1079.57(12) Å³, *T*= 290(2) K, *Z*= 4, *D_c*= 1.250 mg/m³, μ = 0.085 mm⁻¹, λ = 0.71073 Å, *F*(000) 432, crystal size: 0.56 x 0.50 x 0.42 mm³, 1483 reflections collected, 1380 [R(int) = 0.0122]; Refinement method: Full-matrix least-squares on *F*²; goodness-of-fit on *F*²= 0.961, final R indices [*I*> 2σ(*I*)] R1 = 0.0327, wR2 = 0.0652.

1-Nitro-6-(4-methylphenyl)cyclohexene (2b), White solid (41 mg, 63% yield). m.p. 54.4-55.9 °C; [α]_D²⁰ = +84.3 (*c*= 0.86, CHCl₃); IR (KBr): γ 3447, 3022, 2950, 2872, 1660, 1506, 1336, 809 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.57(m, 2H), 1.89(m, 1H), 2.00 (m, 1H), 2.32 (s, 3H), 2.39 (m, 2H), 4.26 (brs, 1H), 7.04 (d, *J*= 8.0 Hz, 2H), 7.14 (d, *J*= 8.0 Hz, 2H), 7.55 (t, *J*= 4.0 Hz, 1H); ¹³C NMR(CDCl₃, 75 MHz): δ (ppm) 16.4, 20.8, 24.9, 31.5, 39.0, 126.9, 129.1, 135.9, 136.2, 138.8, 151.0; HRMS for C₁₃H₁₅NO₂, calcd: 217.1097, found: 217.1095. Enantiomeric excess: 95%, determined by chiral GC analysis (CP-Cyclodextrin-2, 3, 6 – M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature= 240 °C, detector temperature= 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(4-methoxyphenyl)cyclohexene (2c), White solid (40.5 mg, 58% yield). m.p. 56.8-57.9 °C; [α]_D²⁰ = +93.4 (*c*=1.06, CHCl₃); IR (KBr): γ 3494, 2996, 2859, 2936, 2833, 1658, 1613, 1507, 1460, 1448, 1336, 1258, 1177, 1031, 823, 815 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.54-1.61 (m, 2H), 1.82-1.88 (m, 1H), 1.96-2.00 (m, 1H), 2.39 -2.47 (m, 2H), 3.77 (s, 3H), 4.23 (brs, 1H), 6.81 (d, *J*= 11.5 Hz, 2H), 7.04 (d, *J*= 11.5 Hz, 2H), 7.52 (t, *J*= 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.5, 25.0, 31.6, 38.7, 55.2, 113.9, 128.1, 134.0, 135.8, 151.2, 158.3; HRMS for C₁₃H₁₅NO₃, calcd: 233.1046, found: 233.1033. Enantiomeric excess: 96%, determined by chiral HPLC (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0mL / min, *R*-isomer, *t_R*= 12.4 min, *S*-isomer, *t_R*= 14.5 min).

1-Nitro-6-(4-chlorophenyl)cyclohexene (2d), colorless oil (45 mg, 64% yield); [α]_D²⁰ = +102.2 (*c*= 1.15, CHCl₃); IR (neat): γ 2943, 2867, 1662, 1517, 1488, 1336, 1091, 1013, 819; ¹H NMR

(CDCl₃, 300 MHz): δ (ppm) 1.25-1.62 (m, 2H), 1.84-1.87 (m, 1H), 2.03 (m, 1H), 2.41-2.48 (m, 2H), 4.24 (brs, 1H), 7.06 (d, J = 11.0 Hz, 2H), 7.24 (d, J = 11.0 Hz, 2H), 7.56 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.5, 24.9, 31.4, 39.0, 128.4, 128.7, 132.5, 136.6, 140.5, 150.5; HRMS for C₁₂H₁₂ClNO₂, calcd: 237.0551, found: 237.0569. Enantiomeric excess: 99%; determined by chiral GC analysis (CP-Cyclodextrin -2, 3, 6 -M-19, 0.25 mm×25 m, column temperature= 168 °C (iso thermal), inject temperature= 240 °C, detector temperature= 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(4-fluorophenyl)cyclohexene (2e), colorless oil (40 mg, 61% yield); $[\alpha]_D^{20}$ = +87.3 (c = 1.22, CHCl₃); IR (neat): γ 3069, 2945, 2868, 1662, 1602, 1509, 1336, 1222, 826, 816 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.52-1.61 (m, 2H), 1.84-1.87 (m, 1H), 2.02 (m, 1H), 2.39-2.48 (m, 2H), 4.26 (brs, 1H), 6.95-7.01 (m, 2H), 7.10- 7.13 (m, 2H), 7.55 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75MHz): δ (ppm) 16.4, 24.9, 31.5, 38.8, 115.1, 115.4, 128.5, 128.6, 136.4, 137.7, 150.8, 160.0, 163.2; HRMS for C₁₂H₁₂FNO₂, calcd: 221.0846, found: 221.0834. Enantiomeric excess: 98%, determined by Chiral GC analysis (CP -Cyclodextrin-2, 3, 6-M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature = 240 °C, detector temperature= 260 °C, inlet pressure= 10 psi).

1-Nitro-6-(4-*tert*-butylphenyl)cyclohexene (2f), white solid (52 mg, 67% yield). mp: 82.0-83.5 °C; $[\alpha]_D^{20}$ = + 98.2 (c = 0.70, CHCl₃); IR (KBr): γ 2958, 2865, 1659, 1513, 1342, 1330, 1075, 1018, 823 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.28 (s, 9H), 1.57-1.86 (m, 2H), 1.87-2.02 (m, 2H), 2.40-2.48 (m, 2H), 4.25 (brs, 1H), 7.04 (d, J = 8.3 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 7.53 (t, J = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.6, 24.9, 31.2, 31.4, 34.3, 39.0, 125.3, 126.7, 135.8, 138.7, 149.3, 149.3, 151.1; HRMS for C₁₆H₂₁NO₂, calcd: 259.1567, found: 259.1554. Enantiomeric excess: 90%, determined by chiral GC analysis (CP-Cyclodextrin-2, 3, 6-M-19, 0.25 mm×25 m, column temperature= 168 °C (isothermal), inject temperature= 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(4-trifluoromethylphenyl)cyclohexene (2g), colorless oil (42 mg, 52% yield); $[\alpha]_D^{20}$ = +107.2 (c = 0.83, CHCl₃); IR (neat): γ 2943, 2871, 1664, 1618, 1519, 1325, 1163, 1112, 1066, 1017; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.52-1.63 (m, 2H), 1.87-1.90 (m, 1H), 2.08 (m, 1H), 2.44-2.52 (m, 2H), 4.32 (brs, 1H), 7.25 (d, J = 8.2 Hz, 2H), 7.55 (d, J = 8.2 Hz, 2H), 7.62 (t, J = 4.0

Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.5, 24.9, 31.4, 39.5, 122.2, 125.5, 127.4, 128.4, 128.9, 129.3, 129.4, 137.0, 146.1, 150.2; HRMS for C₁₃H₁₂F₃NO₂, calcd: 271.0814, found: 271.0808. Enantiomeric excess: 94%, determined by chiral GC analysis (CP- Cyclodextrin -2, 3, 6-M-19, 0.25 mm×25 m, column temperature = 168 °C (isothermal), inject temperature = 240 °C, detector temperature = 260 °C, inlet pressure = 10 psi).

1-Nitro-6-(3-methoxyphenyl)cyclohexene (2h), white solid (41 mg, 58% yield). m.p.: 81.7-83.6 °C; [α]_D²⁰ = +108.5 (c = 1.29, CHCl₃); IR (KBr): γ 2942, 2866, 2835, 1662, 1600, 1583, 1515, 1487, 1330, 1285, 1263, 1042, 781 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.57-1.62 (m, 2H), 1.88-1.93 (m, 1H), 1.98-2.17 (m, 1H), 2.40-2.48 (m, 2H), 3.78 (s, 3H), 4.24 (brs, 1H), 6.68 (s, 1H), 6.72-6.78 (m, 2H), 7.19 (m, 1H), 7.55 (d, *J* = 8.2 Hz, 2H), 7.62 (t, *J* = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.6, 25.0, 31.4, 39.5, 55.1, 111.6, 113.4, 119.5, 129.5, 136.2, 143.6, 150.8, 159.7; HRMS for C₁₃H₁₅NO₃, calcd: 233.1046, found: 233.1055. Enantiomeric excess: 96%, determined by chiral HPLC analysis (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0 mL / min, *R*-isomer, *t*_R = 22.5 min, *S*-isomer, *t*_R = 25.3 min).

1-Nitro-6-(3,4-methylenedioxy-phenyl)cyclohexene (2i), colorless oil (48 mg, 65% yield); [α]_D²⁰ = +98.2 (c = 1.40, CHCl₃); IR (neat): γ 2942, 2897, 1661, 1607, 1513, 1486, 1439, 1333, 1249, 1231, 1037, 934, 809 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.55-1.62 (m, 2H), 1.81-1.87 (m, 1H), 1.95-2.00 (m, 1H), 2.38-2.47 (m, 2H), 4.19 (brs, 1H), 5.92 (s, 2H), 6.57-6.84 (m, 3H), 7.52 (t, *J* = 4.0 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 16.4, 24.9, 31.5, 39.1, 100.9, 107.7, 108.1, 120.0, 135.8, 136.1, 146.3, 147.774, 150.9; HRMS for C₁₃H₁₃NO₄, calcd: 247.0839, found: 247.0852. Enantiomeric excess: 98%, determined by chiral HPLC analysis (Daicel Chiralcel OD; hexane: isopropanol = 99:1, flow = 1.0 mL / min, *R*-isomer, *t*_R = 15.3 min, *S*-isomer, *t*_R = 21.5 min).

Methyl 1,2-cis-2,3-cis-2-nitro-3-benzo[1,3]dioxol-5-ylcyclohexylacetate (8). Colorless oil (138 mg, 72% yield); IR (KBr): 2939, 2864, 1733, 1543, 1504, 1440; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.48-1.78 (m, 4H), 2.03 (m, 1H), 2.28 (m, 2H), 2.39- 2.45 (m, 2H), 2.45-2.99 (m, 1H), 3.68 (s, 3H), 4.98 (t, *J* = 4.0 Hz, 1H), 5.91 (s, 2H), 6.63 -6.71 (m, 2H), 6.74 (d, *J* = 8.1 Hz, 1H); ¹³C NMR (CDCl₃, 75MHz): δ (ppm) 24.1, 24.9, 25.3, 36.7, 36.9, 45.5, 51.8, 91.1, 101.0, 107.6, 108.3, 120.4, 133.7, 146.7, 147.8, 171.8; HRMS for C₁₆H₁₉NO₆, calcd: 321.1207, found: 321.1206.

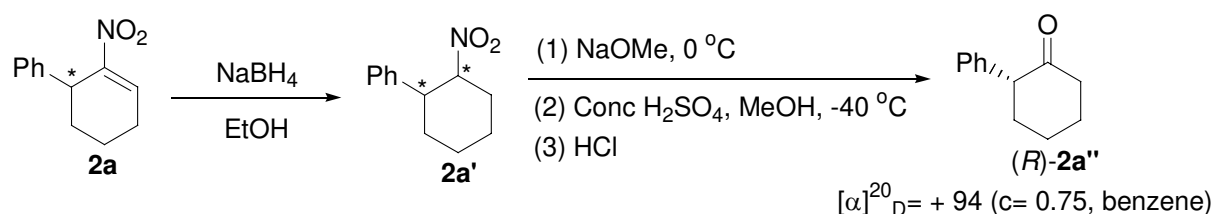
3a, 7a-cis-7, 7a-cis-7-benzo [1, 3] dioxol-5-yl-octahydroindol-2-one (9): To a solution of **8** (96mg, 0.3mmol) in absolute ethanol (1 mL) in autoclave was added Raney-nickel (5 mol%) previously rinsed with absolute ethanol. Hydrogen was introduced into the autoclave until the pressure reached 80 atm. The autoclave was warmed up to 55 °C and was maintained at the temperature for 24 h. After the autoclave was cooled down to room temperature, the mixture was filtered and the filtrate was concentrated. Recrystallization from CHCl₃ / hexane gave **9** as a white solid (74 mg, 95% yield); [α]_D²⁰ = 112.3 (*c* = 1.30, CHCl₃); m.p.: 185.9-187.2 °C; IR (KBr): γ 3252, 2927, 2915, 2853, 1681, 1502, 1488, 1242, 1230, 1039; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.25-1.33 (m, 2H), 1.68-1.75 (m, 3H), 1.82(m, 1H), 1.96 (d, *J*=16.0 Hz, 1H), 2.47 (m, 1H), 2.52 (m, 1H), 2.78-2.82 (m, 1H), 3.84 (t, *J* = 4.2 Hz, 1H), 5.33 (brs, 1H), 5.95 (s, 2H), 6.63 (d, *J*= 1.5 Hz, 1H), 6.66 (dd, *J*= 1.5 Hz, 7.9 Hz, 1H), 6.75 (d, *J*= 7.9 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 24.2, 27.5, 35.1, 40.2, 43.7, 58.8, 101.0, 107.5, 108.5, 119.7, 136.2, 146.4, 148.0, 177.9; HRMS for C₁₅H₁₇NO₃, calcd: 259.1203, found: 259.1203. Enantiomeric excess: 98%, determined by chiral HPLC (Daicel Chiralcel OJ-H; hexane: isopropanol = 70:30, flow = 1.0 mL / min, *R*- isomer, *t*_R = 8.1 min, *S*-isomer, *t*_R = 13.3 min).

Preparation and characterization of compound 10: To a solution of **9** (52 mg, 0.20 mmol) in anhydrous Cl(CH₂)₂Cl (5 mL) was added, sequentially, paraformaldehyde (23.1 mg, 0.77 mmol) and CF₃CO₂H (0.19 mL, 2.5 mmol) at room temperature. The reaction mixture was stirred at room temperature for 24 h, and then quenched with saturated aqueous NaHCO₃ (10 mL) followed by addition of CH₂Cl₂ (40 mL). The organic layer was separated, and the aqueous phases were washed by brine, and dried over NaSO₄. After removal of the solvent under reduced pressure, the residue was purified by column chromatography on silica gel (AcOEt / PET = 3/1) to give **10** (47 mg, 88% yield) as a white solid. m.p.: 144.0-147.7 °C; IR (KBr): γ 2927, 2854, 1676, 1503, 1483, 1440, 1418; ¹H NMR (CDCl₃, 300 MHz): δ (ppm) 1.12 -1.32 (m, 3H), 1.68 (m, 3H), 2.04 (d, *J*= 16.0 Hz, 1H), 2.39 (m, 1H), 2.51 (dd, *J* = 16.0 Hz, 6.8 Hz, 1H), 2.69 (m, 1H), 3.73 (t, *J* = 4.5 Hz, 1H), 3.76 (d, *J*= 17.3 Hz, 1H), 4.49 (d, *J*= 17.3 Hz, 1H), 5.89 (m, 2H), 6.57 (s, 1H), 6.59 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 23.5, 27.8, 30.2, 32.9, 39.7, 40.2, 42.6, 55.6, 100.9, 106.6, 108.4, 123.2, 131.5, 146.5, 146.6, 175.6; HRMS for C₁₆H₁₇NO₃, calcd: 271.1203, found: 271.1193.

(+)- γ -Lycorane: A mixture of **10** (40 mg, 0.15 mmol) and LiAlH₄ (17.1 mg, 0.45 mmol) in THF (5

mL) was stirred at reflux for 18 h. NaSO₄ was added, and the precipitate was removed by filtration. The filtrate was concentrated, and the residue was purified by flash column chromatography (Et₂O) to give (+)- γ -Lycorane as a colorless oil (37 mg, 98% yield); $[\alpha]_D^{20} = +17.3$ ($c = 0.98$, EtOH), [lit.³ $[\alpha]_D^{20} +17.1$ ($c = 0.25$, EtOH)]; IR (KBr): γ 2928, 2848, 1505, 1483, 1375, 1318, 1245, 1226, 1038; ¹HNMR (CDCl₃, 300 MHz): δ (ppm) 1.25-1.35 (m, 5H), 1.42 (m, 3H), 1.69-2.20 (m, 3H), 2.36 (t, $J = 4.5$ Hz, 1H), 2.70 (m, 1H), 3.18 (d, $J = 14.4$ Hz, 1H), 3.23 (m, 1H), 3.99 (d, $J = 14.4$ Hz, 1H), 5.87 (m, 2H), 6.49 (s, 1H), 6.61 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ (ppm) 25.2, 29.2, 30.3, 31.7, 37.3, 39.4, 53.7, 57.1, 62.8, 100.6, 106.2, 108.3, 127.3, 133.1, 145.6, 146.0; HRMS for C₁₆H₂₀NO₂, $[M+H]^+$ calcd: 258.1489, found: 258.1496. The spectral data are in agreement with those reported in the literature.⁴

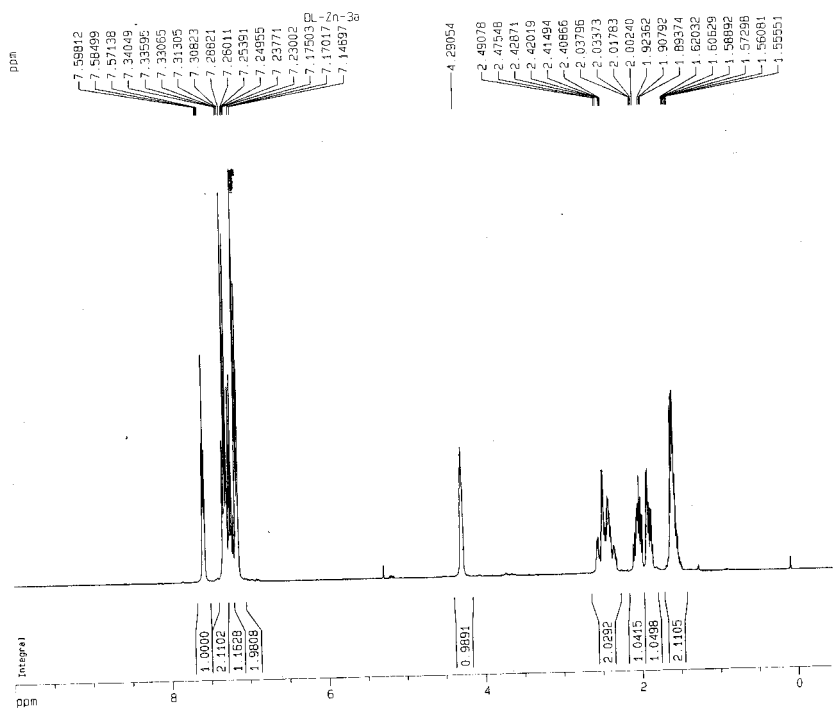
Determination of the absolute configuration of compound **2a**:



To a solution of the nitro olefin **2a** (40.6 mg, 0.2 mmol) in ethanol (10 mL) at 0 °C was added NaBH₄ (15.2 mg, 0.4 mmol) over 20 min. After stirring for 2 h at r.t., the ethanol was removed and 2N HCl (5 mL) was added. The product was extracted with CH₂Cl₂ (3 \times 10 mL) and combined organic layers were dried (NaSO₄), and the CH₂Cl₂ was removed to give the crude nitro **2a'** (40 mg, 98% yield).⁵ To a solution of **2a'** in methanol (0.2 mL) was added sodium methoxide (13.2 mg, 0.24 mmol) in methanol (0.2 mL) at 0 °C and the mixture was stirred for 0.5 h. To the nitronate solution was added a mixture of concentrated sulfuric acid (40 μ L) and methanol (0.2 mL). During the addition, the mixture was kept at -40 °C and after additional stirring at -20 °C for 1 h, it was poured into dichloromethane (50 mL). The organic layer was washed with water (20 mL) and was extracted with dichloromethane (20 mL). The combined organic layers were dried over NaSO₄, and the CH₂Cl₂ was removed. Dichloromethane (20 mL) and hydrochloric acid (2M, 20 mL) were added, and the mixture was stirred at r.t for 2 h. The aqueous layer was extracted with dichloromethane, and the combined organic layers were dried over NaSO₄. After the CH₂Cl₂ was removed, the residue was purified by column chromatography on silica gel to give 2-phenylcyclohexanone (**R**)-**2a''**: $[\alpha]_D^{20} = +94$ ($c = 0.75$, benzene).⁶ The reported specific rotation of (**2S**)-**2a''** is $[\alpha]_D^{20} -114$ ($c = 0.60$, benzene).⁷

References

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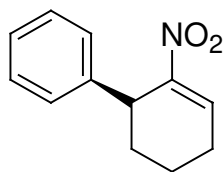
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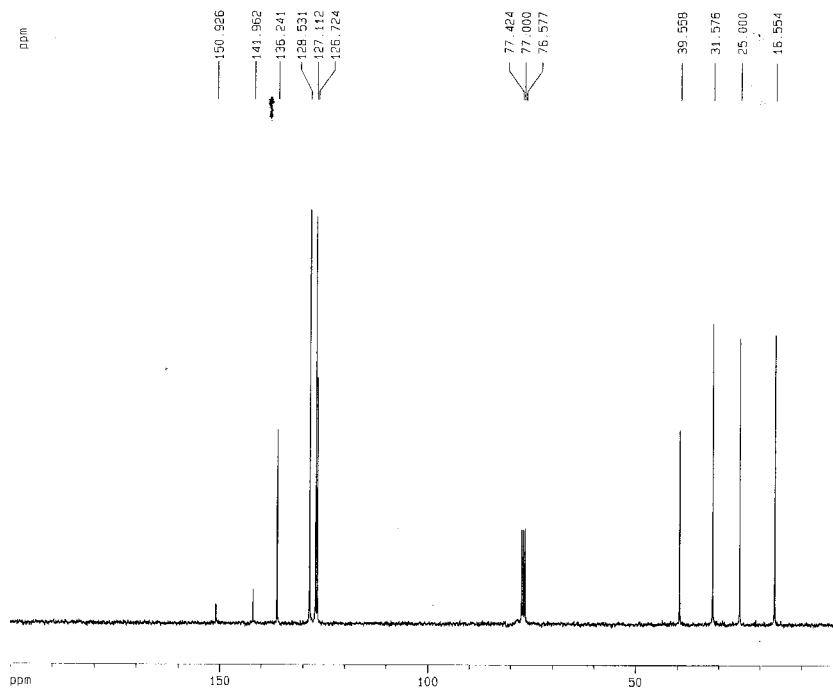
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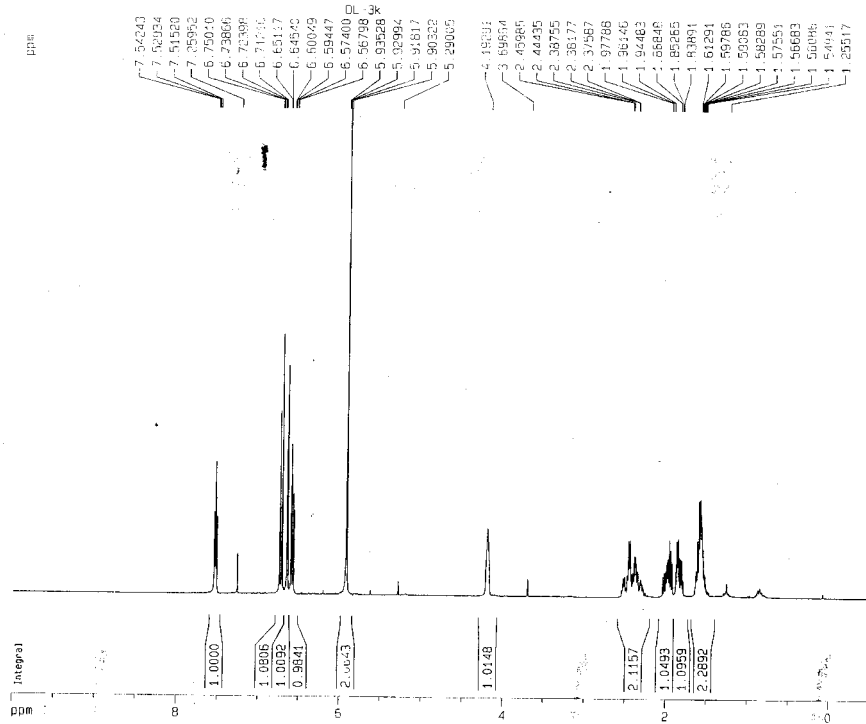
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 SFG2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677563 MHz
 WDW EN
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 150.93 Hz
 F1 75477.56 Hz
 F2P -0.38 Hz
 F2 10.00050 ppm/cm
 HZCM 754.71533 Hz/cm



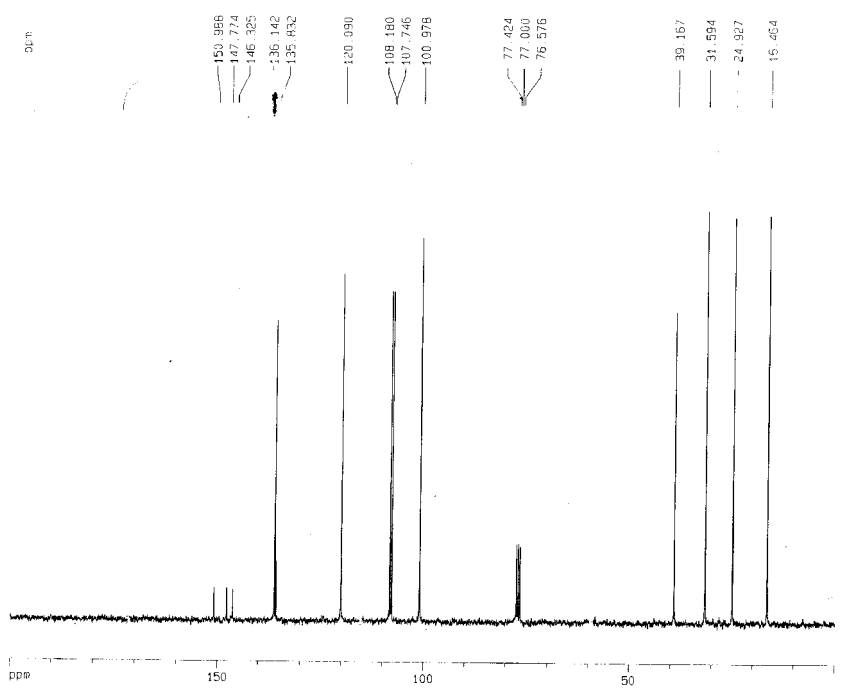
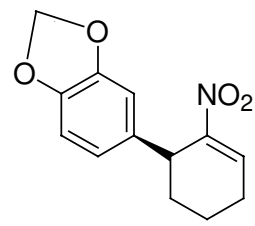
Current Data Parameters
 NAME 1367-01-569
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050330
 Time 11.57
 INSTRUM av300
 PROBHD 5 mm GNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 256
 DN 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 DT 1.00000000 sec

***** CHANNEL f1 *****
 NU1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SF01 300.1320882 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300128 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 20.00 cm
 FAP 10.000 ppm
 F1 3001.30 Hz
 F2 -150.05 Hz
 PPMCM 0.52900 ppm/cm
 HZCM 157.56825 Hz/cm



Current Data Parameters
 NAME 1367-01-569
 EXNO 2
 PROCNO 1

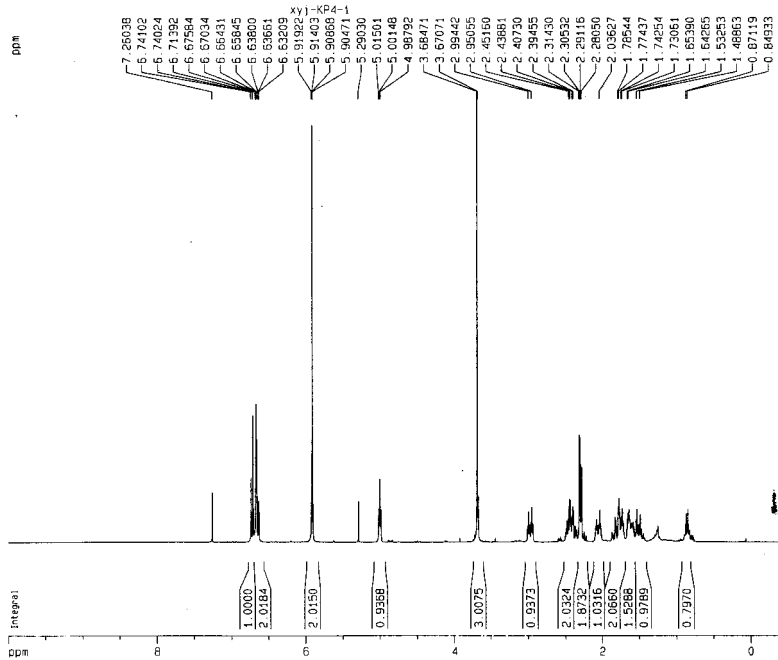
F2 - Acquisition Parameters
 Date_ 20050330
 Time 12.00
 INSTRUM av300
 PROBHD 5 mm GNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.4451888 sec
 RG 8192
 DN 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 DT 2.00000000 sec
 D13 0.00000000 sec
 D12 0.00000000 sec

***** CHANNEL f1 *****
 NU1 13C
 P1 6.50 usec
 PL1 -6.00 dB
 SF01 75.4775998 MHz

***** CHANNEL f2 *****
 CDPRG2 waltz16
 NU2 1H
 P2 80.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SF02 300.1312008 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4673758 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 FAP 200.005 ppm
 F1 15093.93 Hz
 F2 -0.005 ppm
 F3 -0.38 Hz
 PPMCM 10.00050 ppm/cm
 HZCM 754.71933 Hz/cm



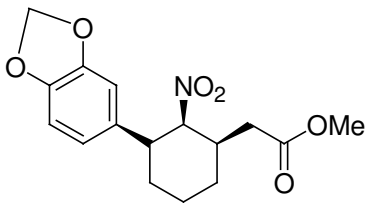
Current Data Parameters
 NAME 1367-xyj-116
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050426
 Time 15.12
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 599.5264 Hz
 FIDRES 0.182959 Hz
 AQ 2.7326011 sec
 RG 64
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

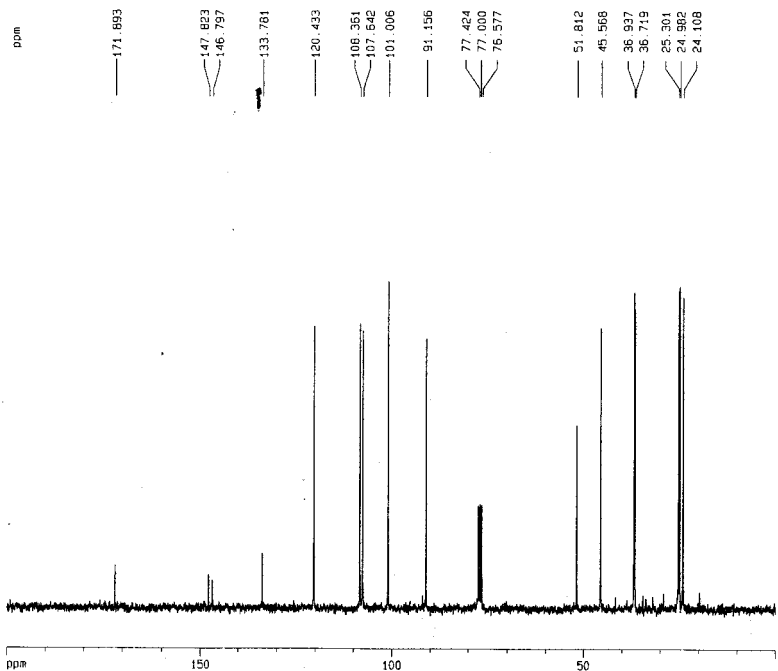
***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.132682 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300132 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

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



xyj-KP4-1



Current Data Parameters
 NAME 1367-xyj-116
 EXPNO 2
 PROCNO 1

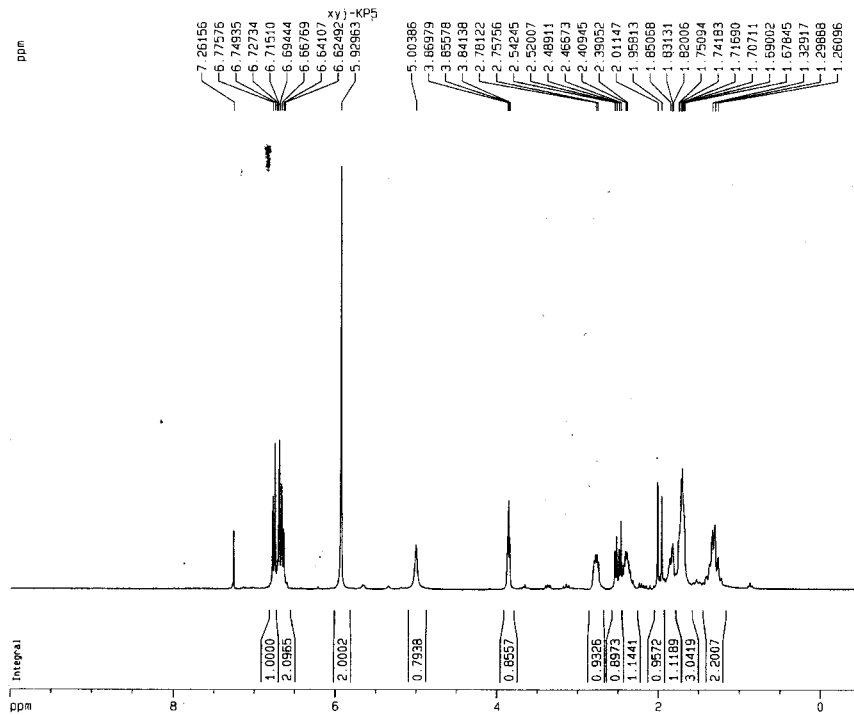
F2 - Acquisition Parameters
 Date_ 20050426
 Time 15.48
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 276
 DS 4
 SMH 22675.736 Hz
 FIDRES 0.346304 Hz
 AQ 1.4451188 sec
 RG 8192
 DM 22.090 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.00 usec
 PL1 -8.00 dB
 SFO1 75.4775898 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 60.00 usec
 PL2 -2.00 dB
 PL12 17.70 dB
 PL13 17.71 dB
 SFO2 300.1310015 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677536 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 300.095 ppm
 F1 15083.81 Hz
 F2P -0.805 ppm
 F2 -0.36 Hz
 WINDX 10.00000 ppm/cm
 HZCX 754.71527 Hz/cm



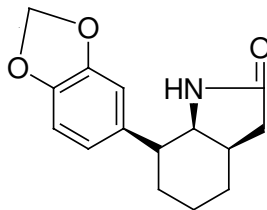
Current Data Parameters
 NAME 1367-xyj-111
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050419
 Time 18.00
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 ID 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.162953 Hz
 AQ 2.7329011 sec
 RG 256
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

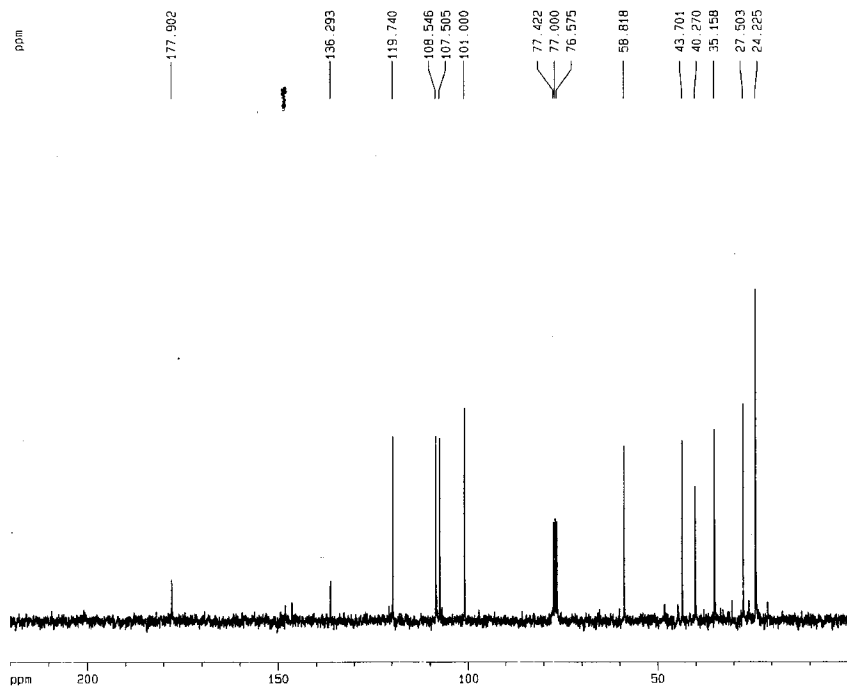
***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.1320882 MHz

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 10.900 ppm
 F1 3001.30 Hz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PPMCH 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



xyj-Kp5



Current Data Parameters
 NAME 1367-xyj-111
 EXPNO 2
 PROCNO 1

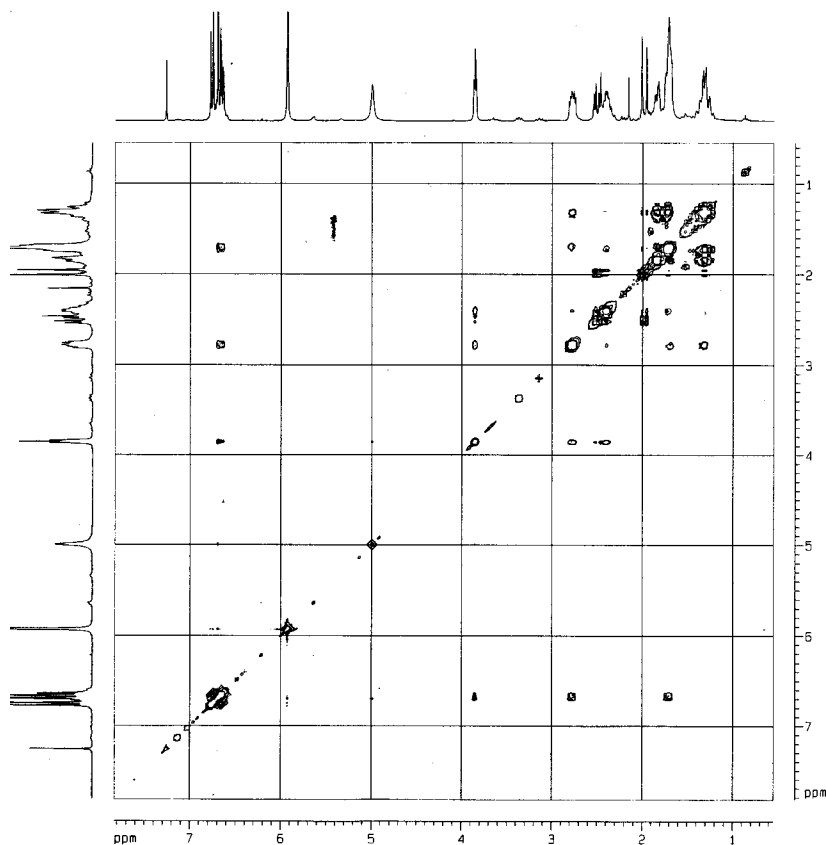
F2 - Acquisition Parameters
 Date_ 20050419
 Time 18.05
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg
 ID 65536
 SOLVENT CDCl3
 NS 120
 DS 1
 SWH 22675.736 Hz
 FIDRES 0.345004 Hz
 AQ 1.4491188 sec
 RG 8192
 DW 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

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

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 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 75.4677551 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 220.005 ppm
 F1 15603.29 Hz
 F2P -0.000 ppm
 F2 -0.38 Hz
 PPMCH 11.00050 ppm/cm
 HZCM 830.18311 Hz/cm



xyj-KP5 NOESY

```

Current Data Parameters
NAME      1367-xyj-111
EXPNO    8
PROCNO   1

F2 - Acquisition Parameters
Date_    20050420
Time     18.48
INSTRUM  av300
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD        131074
SOLVENT  DMS-D6
NS        32
DS        4
SWH       2180.310 Hz
FIDRES    0.134898 Hz
AQ         0.2343412 sec
RG         32
DM         228.800 usec
DE         6.00 usec
TE         300.2 K
DQ         0.0000000 sec
DI         2.0000000 sec
DB         1.0000000 sec
JRG        0.0000000 sec
HCNRES1   0.0000000 sec
MWRFR     1.0000000 sec
ST1CHW    250

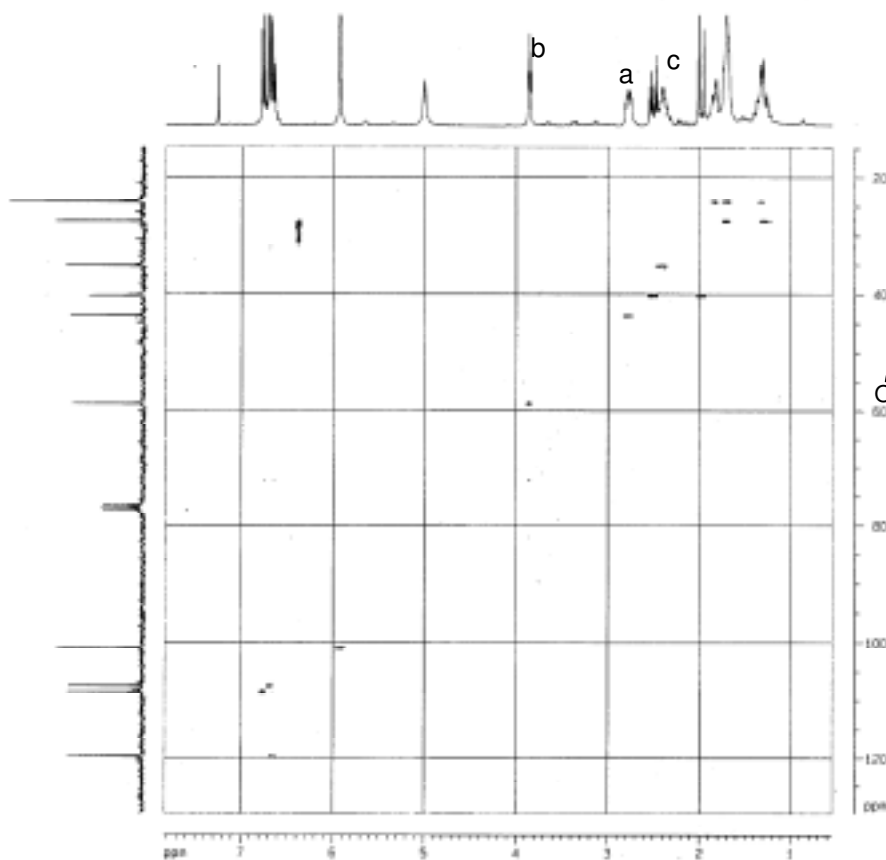
***** CHANNEL f1 *****
NUC1      13C
P1         7.00 usec
PL1        2.00 dB
SFO1      300.1312689 MHz

F1 - Acquisition parameters
AQ         1.0000000 sec
DI         2.0000000 sec
DB         1.0000000 sec
JRG        0.0000000 sec
MWRFR     1.0000000 sec
ST1CHW    250

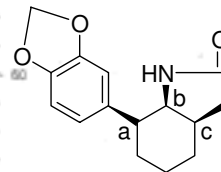
F2 - Processing parameters
SI         1024
SF         300.1301020 MHz
WDW        HANNING
SSB         0
LB          0.00 Hz
GB          0
PC          1.00

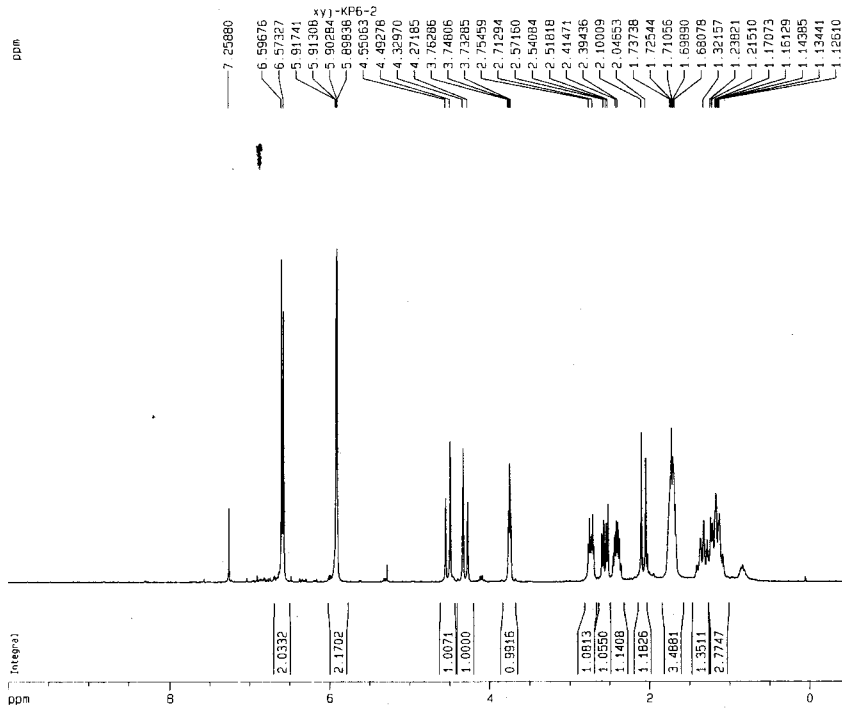
F1 - Processing parameters
SI         1024
MC2        States-IPPI
SF         300.1300122 MHz
WDW        HANNING
SSB         0
LB          0.00 Hz
GB          0

2D NMR plot parameters
CX2        15.00 cm
CY2        15.00 cm
F2PLD      7.828 ppm
F2OL       2340.20 Hz
F2PH1      0.547 ppm
F2PL1      164.04 Hz
F2OL1      2340.20 Hz
F2PH11     0.547 ppm
F2PL11     164.04 Hz
F2PH1CH1  0.48042 ppm/c
F2OLCH1   140.68764 Hz/c
F2PH1CH2  0.48042 ppm/c
F2OLCH2   140.68764 Hz/c
  
```



xyj-KP5 HSQC





Current Data Parameters
 NAME 1367-xyj-128
 EXPNO 1
 PROCNO 1

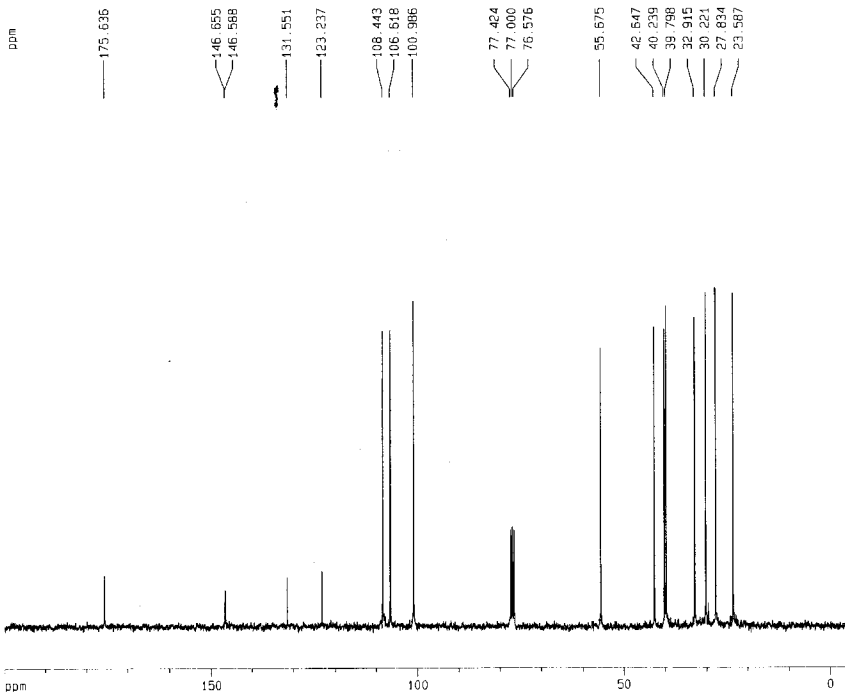
F2 - Acquisition Parameters
 Date_ 20050522
 Time 18.13
 INSTRUM av300
 PROBDH 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 512
 OW 83.400 usec
 OE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.132082 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300126 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 300.130 MHz
 F2P -0.500 ppm
 F2 -150.06 Hz
 PRMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm

xyj-KP6-2



Current Data Parameters
 NAME 1367-xyj-128
 EXPNO 2
 PROCNO 1

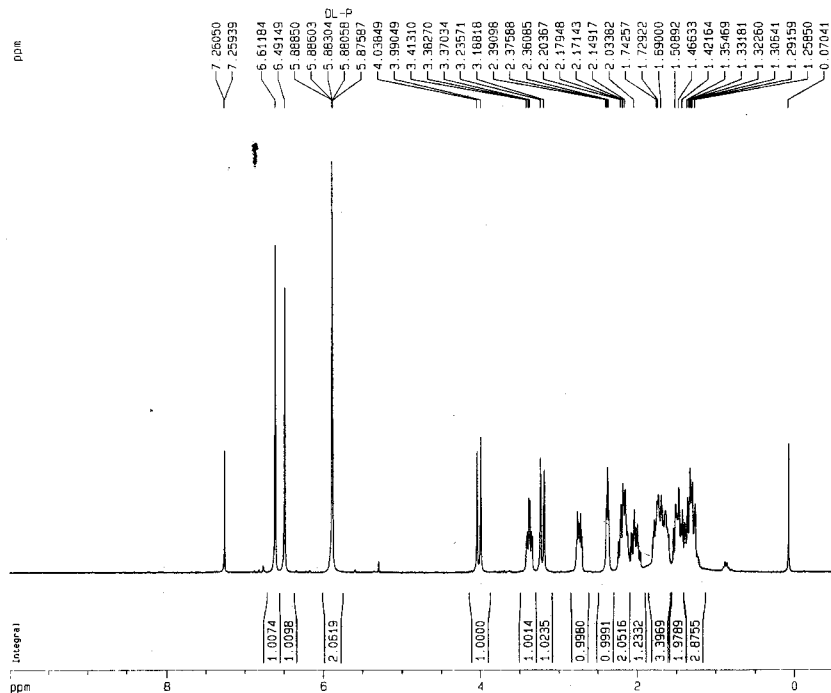
F2 - Acquisition Parameters
 Date_ 20050523
 Time 9.20
 INSTRUM av300
 PROBDH 5 mm QNP 1H/13
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346094 Hz
 AQ 1.4491188 sec
 RG 8152
 OW 22.050 usec
 OE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -6.00 dB
 SFO1 75.4775958 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PULP2 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 75.4677551 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 150.933 MHz
 F2P -377.72 Hz
 F2 10.25050 ppm/cm
 HZCM 773.56828 Hz/cm



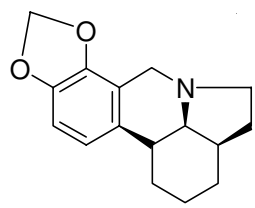
Current Data Parameters
 NAME 1367-d1-605
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050522
 Time 15.09
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.182599 Hz
 AQ 2.7329011 sec
 RG 512
 DW 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

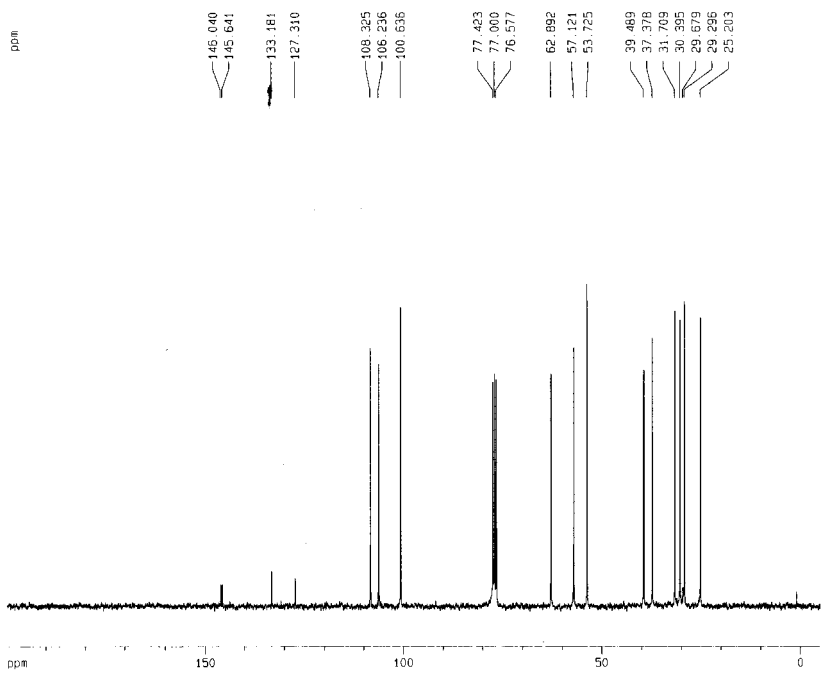
***** CHANNEL f1 *****
 NUC1 1H
 P1 3.00 usec
 PL1 -2.00 dB
 SFO1 300.132082 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300126 MHz
 MHZ EN
 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 -150.00 ppm
 F2 -150.06 Hz
 PPMCM 0.52500 ppm/cm
 HZCM 157.56825 Hz/cm



DL-P



Current Data Parameters
 NAME 1367-d1-605
 EXPNO 2
 PROCNO 1

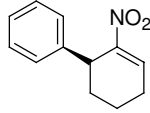
F2 - Acquisition Parameters
 Date_ 20050522
 Time 15.14
 INSTRUM av300
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 292
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.346004 Hz
 AQ 1.445188 sec
 RG 8192
 DN 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00000000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.50 usec
 PL1 -8.00 dB
 SFO1 75.477598 MHz

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

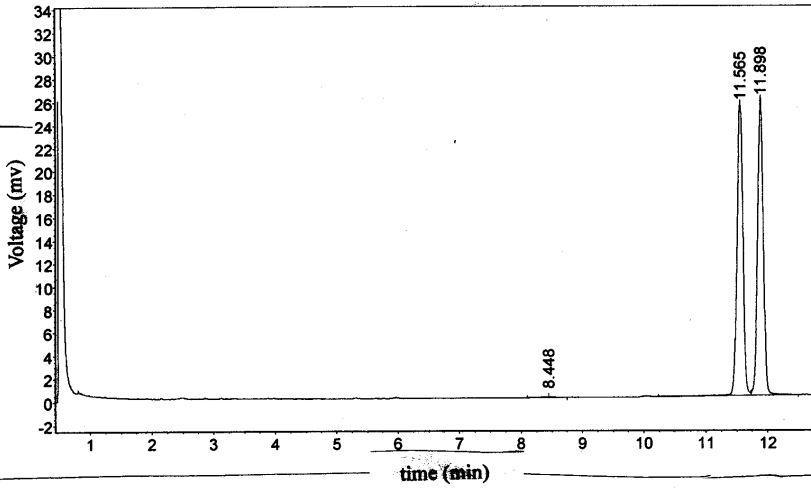
F2 - Processing parameters
 SI 32768
 SF 75.4677518 MHz
 MHZ EN
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 200.005 ppm
 F1 15053.89 Hz
 F2P -15.000 ppm
 F2 -377.72 Hz
 PPMCM 10.25050 ppm/cm
 HZCM 773.58221 Hz/cm



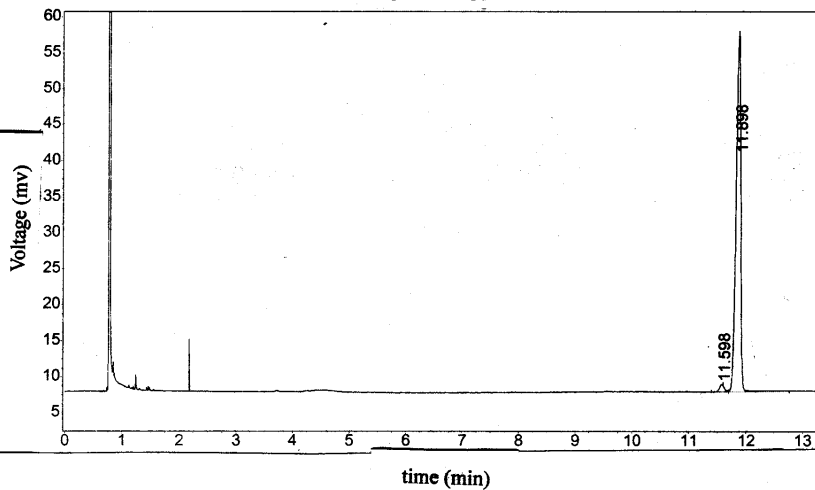
11.898

GC Spectroscopy

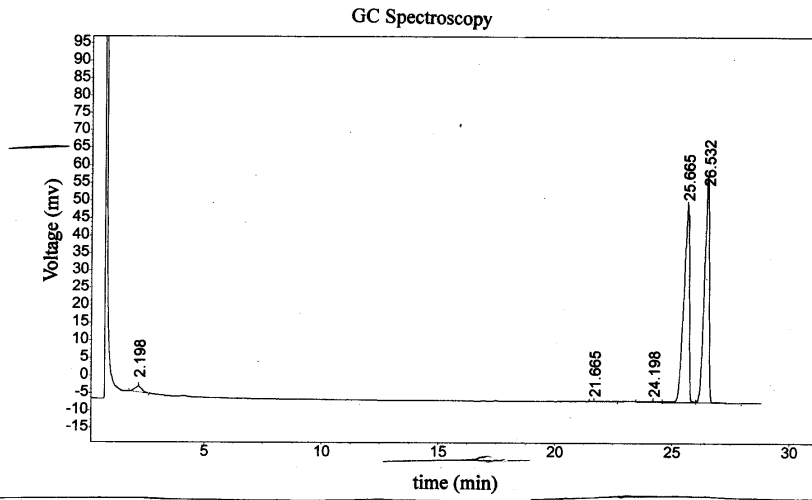
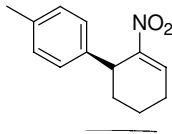


Peak No.	RT(min)	Height	Area	% Area
1	8.448	53.385	1383.000	0.4067
2	11.565	23644.205	169898.766	49.9658
3	11.898	24106.625	168748.172	49.6274
Total		47804.215	340029.938	100.0000

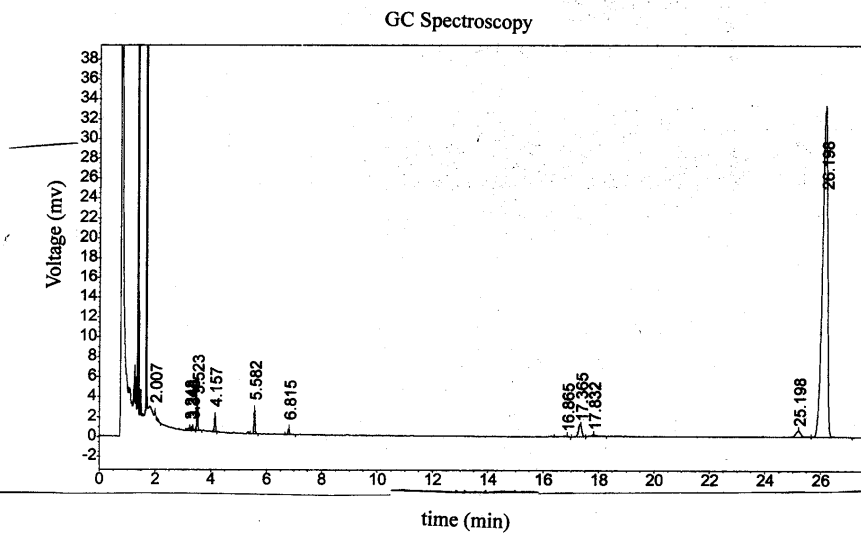
GC Spectroscopy



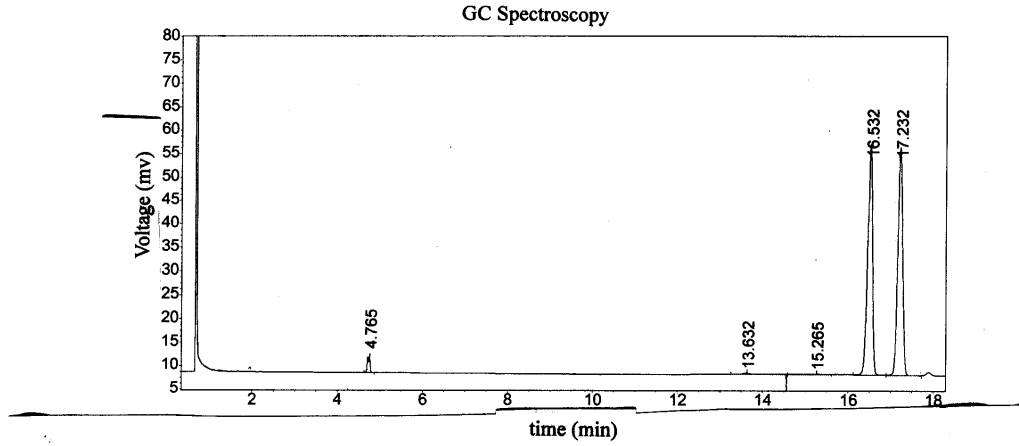
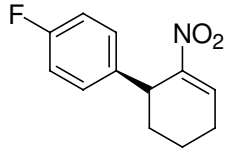
Peak No.	RT(min)	Height	Area	% Area
1	11.598	976.781	5968.438	2.0723
2	11.898	44995.953	282048.281	97.9277
Total		45972.734	288016.719	100.0000



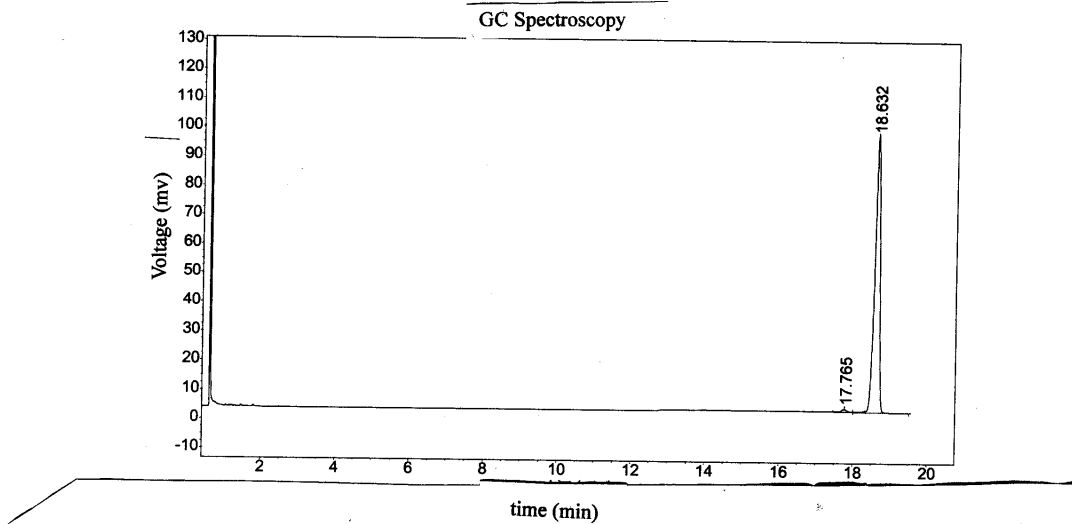
Peak No.	RT(min)	Height	Area	% Area
1	2.198	1688.608	35677.199	1.8732
2	21.665	172.576	10506.529	0.5516
3	24.198	265.953	16151.837	0.8480
4	25.665	53381.750	926318.875	48.6359
5	26.532	63053.223	915943.625	48.0912
Total		118562.110	1904598.065	100.0000



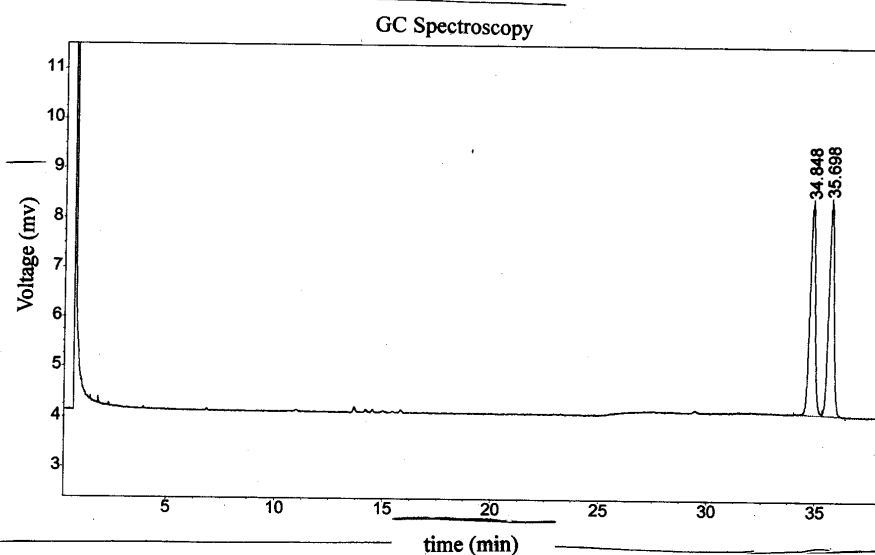
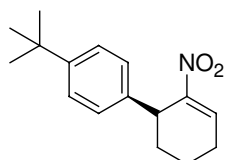
Peak No.	RT(min)	Height	Area	% Area
1	2.007	262.556	1217.450	0.2541
2	3.248	221.652	1912.620	0.3992
3	3.348	266.087	1242.450	0.2593
4	3.523	2807.348	6518.280	1.3604
5	4.157	1671.118	4672.275	0.9751
6	5.582	2452.647	7556.600	1.5770
7	6.815	593.727	2271.700	0.4741
8	16.865	93.558	1294.416	0.2701
9	17.365	1250.140	10707.479	2.2346
10	17.832	200.696	3011.700	0.6285
11	25.198	618.730	11814.473	2.4657
12	26.198	30722.973	426942.094	89.1019
Total		41161.230	479161.537	100.0000



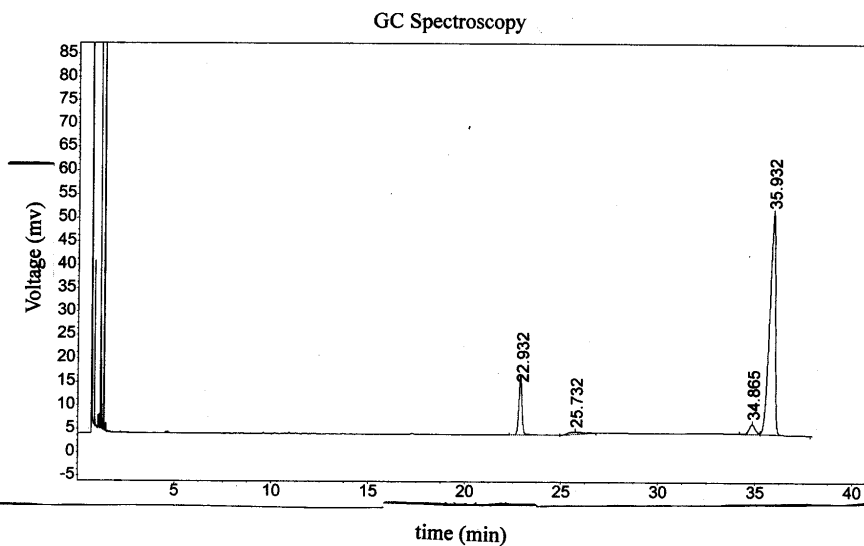
Peak No.	RT(min)	Height	Area	% Area
1	4.765	3365.444	15588.560	1.8827
2	13.632	427.254	18123.023	2.1888
3	15.265	263.422	15125.823	1.8268
4	16.532	47785.301	392320.563	47.3816
5	17.232	47484.051	386844.594	46.7202
Total		99325.472	828002.563	100.0000



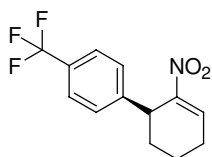
Peak No.	RT(min)	Height	Area	% Area
1	17.765	938.836	9384.417	1.0360
2	18.632	92089.922	896404.500	98.9640
Total		93028.758	905788.917	100.0000



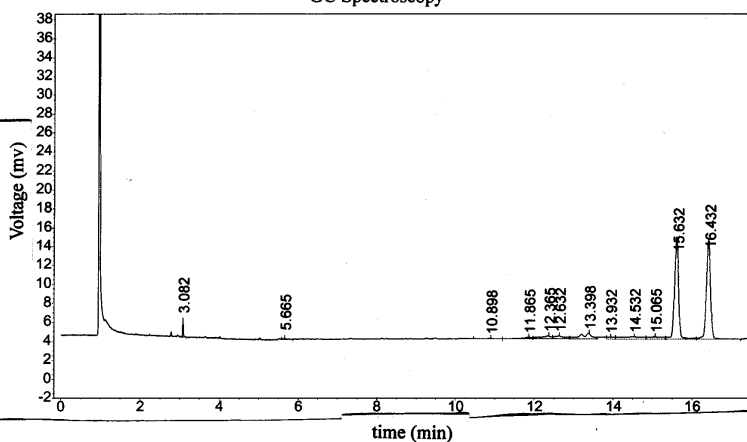
Peak No.	RT(min)	Height	Area	% Area
1	34.848	4252.729	72584.242	50.1869
2	35.698	4299.750	72043.555	49.8131
Total		8552.479	144627.797	100.0000



Peak No.	RT(min)	Height	Area	% Area
1	22.932	11783.612	144855.188	11.8763
2	25.732	532.000	41827.398	3.4293
3	34.865	2161.471	55576.078	4.5565
4	35.932	45424.727	977439.313	80.1378
Total		59901.810	1219697.977	100.0000

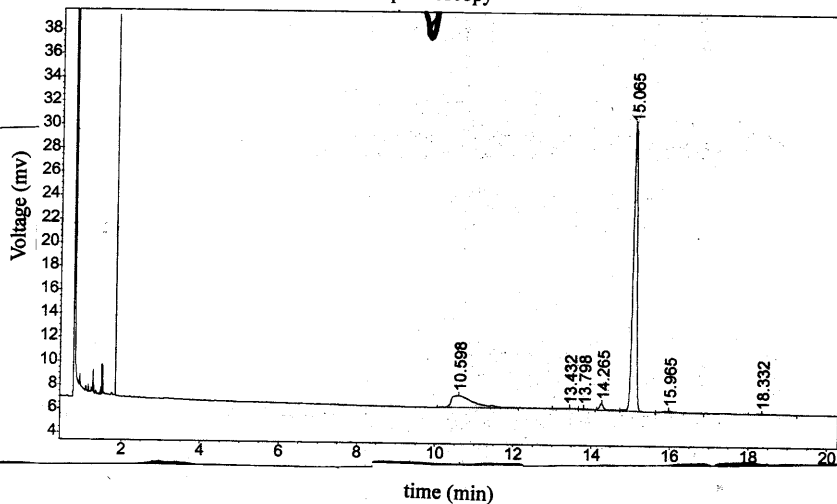


GC Spectroscopy

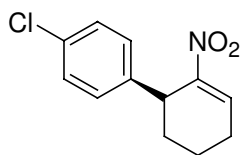


Peak No.	RT(min)	Height	Area	% Area
1	3.082	1479.429	2192.100	1.0952
2	5.665	182.700	1460.251	0.7296
3	10.898	85.461	1277.200	0.6381
4	11.865	174.849	3494.204	1.7458
5	12.365	304.049	6376.963	3.1861
6	12.632	311.156	5923.470	2.9596
7	13.398	580.463	12176.129	6.0836
8	13.932	184.677	2518.174	1.2582
9	14.532	204.918	8630.977	4.3123
10	15.065	198.131	5402.146	2.6991
11	15.632	10154.358	75915.336	37.9299
12	16.432	10053.680	74779.664	37.3624
Total		23913.870	200146.612	100.0000

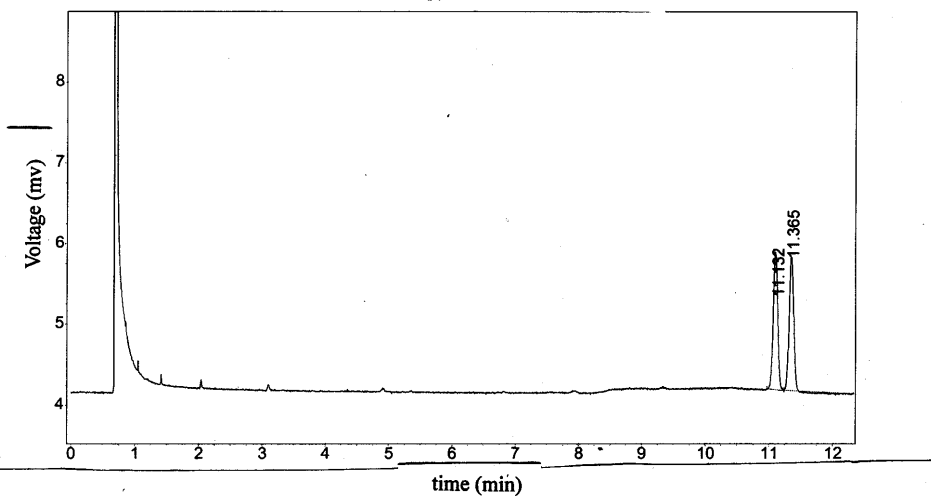
GC Spectroscopy



Peak No.	RT(min)	Height	Area	% Area
1	10.598	993.935	38361.500	17.5286
2	13.432	68.405	1457.452	0.6660
3	13.798	83.671	1782.269	0.8144
4	14.265	564.646	5726.372	2.6166
5	15.065	23493.316	167245.344	76.4198
6	15.965	118.054	2977.100	1.3603
7	18.332	38.378	1300.900	0.5944
Total		25360.406	218850.936	100.0000

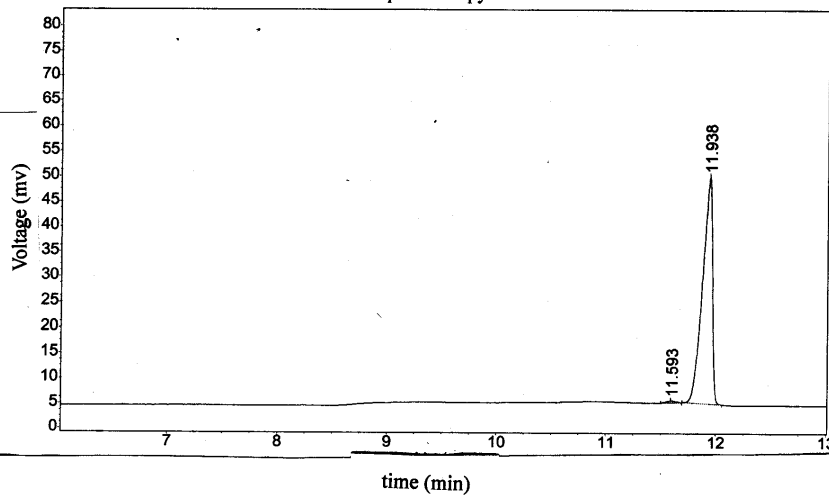


GC Spectroscopy



Peak No.	RT(min)	Height	Area	% Area
1	11.132	1484.923	8422.314	50.7121
2	11.365	1587.015	8185.781	49.2879
Total		3071.938	16608.095	100.0000

GC Spectroscopy



Peak No.	RT(min)	Height	Area	% Area
1	11.593	344.618	1831.899	0.6493
2	11.938	44855.289	280286.281	99.3507
Total		45199.907	282118.180	100.0000

ulas

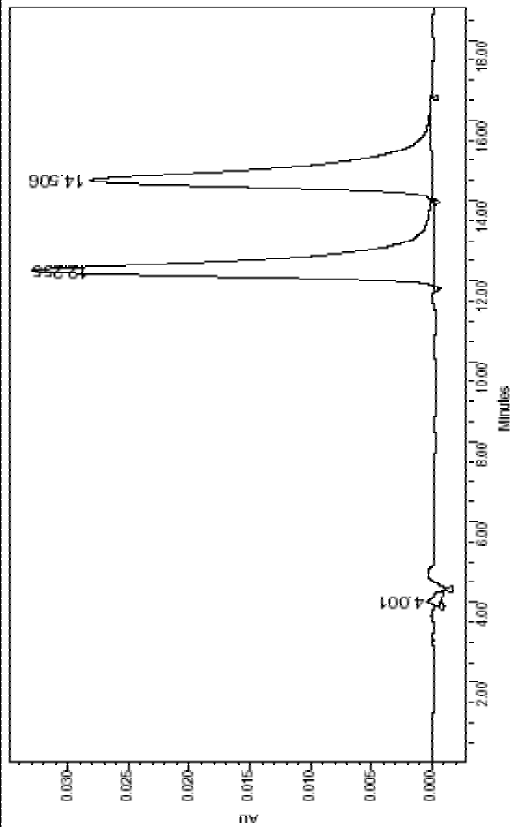
Project Name: Defaults
Reported by User: System



SAMPLE INFORMATION

Sample Name: dl-3b-0
Sample Type: Unknown
Vial: 1
Injection #: 4
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes

Acquired By: System
Date Acquired: 3/28/05 5:53:42 PM
Acq. Method: 100A
Date Processed: 3/28/05 6:12:43 PM
Channel Name: 2487Channel 1
Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 4.001	17523	0.93	1119	1.79
2 12.255	99909	49.78	33260	53.07
3 14.506	930073	49.29	28291	45.14

ulas

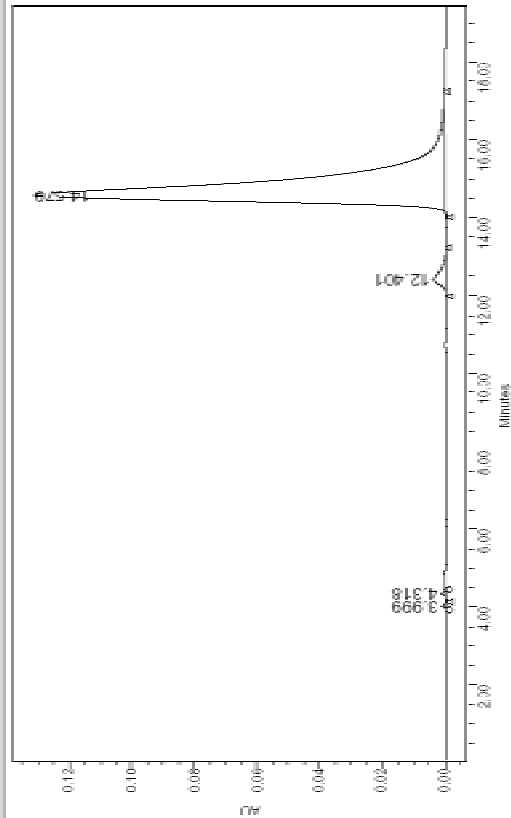
Project Name: Defaults
Reported by User: System



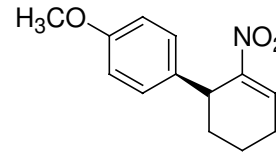
SAMPLE INFORMATION

Sample Name: dl-3b-1
Sample Type: Unknown
Vial: 1
Injection #: 5
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes

Acquired By: System
Date Acquired: 3/28/05 6:13:43 PM
Acq. Method: 100A
Date Processed: 3/28/05 6:33:10 PM
Channel Name: 2487Channel 1
Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.999	7329	0.15	1432	1.04
2 4.318	11521	0.24	1538	1.11
3 12.401	100021	2.08	3844	2.79
4 14.670	4678486	97.52	13155	95.06



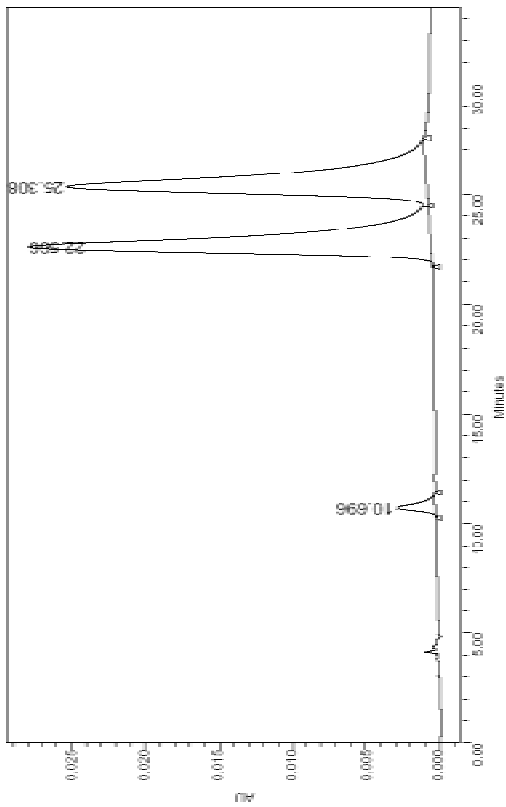
ulas

Project Name: Defaults
Reported by User: System

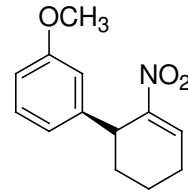


SAMPLE INFORMATION

Sample Name: dl-3c-0 System
Sample Type: Unknown Date Acquired: 3/28/05 4:15:08 PM
Vial: 1 Acq. Method: 100A
Injection #: 1 Date Processed: 3/28/05 4:48:43 PM
Injection Volume: 20.00 ul Channel Name: 2487Channel 1
Run Time: 180.00 Minutes Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 10.696	61478	2.15	2616	4.80
2 25.308	1400030	48.96	27431	50.29
3 26.308	1386273	46.89	24601	44.92



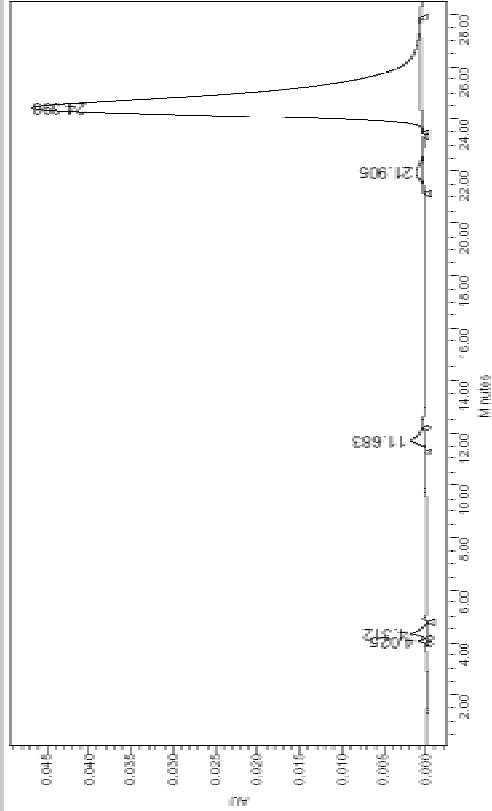
ulas

Project Name: Defaults
Reported by User: System



SAMPLE INFORMATION

Sample Name: dl-3c System
Sample Type: Unknown Date Acquired: 3/28/05 4:49:31 PM
Vial: 1 Acq. Method: 100A
Injection #: 2 Date Processed: 3/28/05 5:18:18 PM
Injection Volume: 20.00 ul Channel Name: 2487Channel 1
Run Time: 180.00 Minutes Sample Set Name:



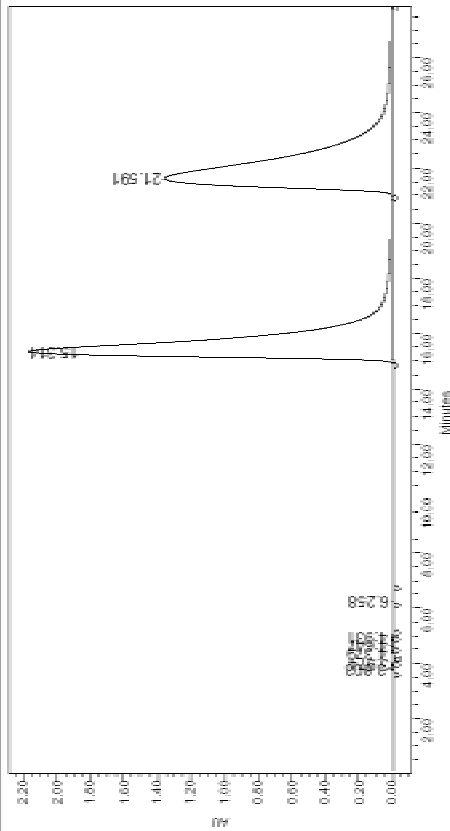
RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 4.02E	7784	0.28	1004	1.93
2 4.312	29202	1.04	1962	3.81
3 11.685	38660	1.37	1583	3.04
4 21.905	42801	1.51	967	1.86
5 24.398	2701142	95.81	46456	89.35



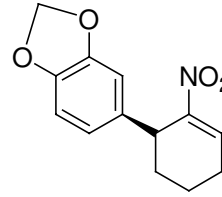
SAMPLE INFORMATION

Sample Name: dlk-0
Sample Type: Unknown
Vial: 1
Injection #: 2
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes

Acquired By: System
Date Acquired: 3/31/05 12:52:33 PM
Acq. Method: 100A
Date Processed: 3/31/05 2:34:45 PM
Channel Name: 2487Channel 1
Sample Set Name:



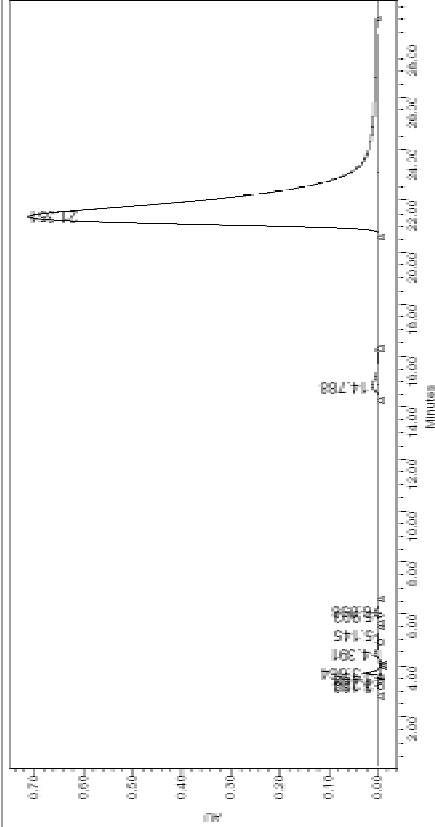
RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.803	248349	0.12	19601	0.64
2 3.974	56806	0.03	8106	0.22
3 4.379	103844	0.05	11704	0.32
4 4.647	346391	0.17	18743	0.62
5 4.931	103887	0.05	11942	0.33
6 6.258	118503	0.06	5155	0.14
7 16.314	101073644	49.70	2173078	60.26
8 21.591	101320312	48.82	1358813	37.87



SAMPLE INFORMATION

Sample Name: dlk-3
Sample Type: Unknown
Vial: 1
Injection #: 3
Injection Volume: 20.00 ul
Run Time: 180.00 Minutes

Acquired By: System
Date Acquired: 4/4/05 10:39:39 AM
Acq. Method: 100A
Date Processed: 4/4/05 11:09:38 AM
Channel Name: 2487Channel 1
Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 3.183	39209	0.08	3803	0.48
2 3.226	50710	0.10	4453	0.56
3 3.484	39320	0.08	4762	0.60
4 3.664	254228	0.62	31962	4.00
5 4.391	290002	0.69	13126	1.64
6 5.145	85784	0.17	4493	0.58
7 5.903	34022	0.07	4160	0.52
8 6.058	85731	0.17	6972	0.87
9 14.788	426787	0.87	12059	1.51
10 21.351	47917977	97.36	712716	89.26