Supporting Information

Asymmetric Nitroaldol Reaction Catalyzed by a C_2-Symmetric Bisoxazolidine Ligand

Shuanglong Liu and Christian Wolf*
Department of Chemistry, Georgetown University, Washington, DC 20057
cw27@georgetown.edu

1. Synthetic Procedure

General nitroaldol reaction procedure

To a solution of the chiral ligand (28 mg, 0.075 mmol, 8 mol%) in toluene (1 ml) was added dimethylzinc (3 mmol, 1 M in heptane) at -50 ^{o}C under N_2. After 10 min, $MeNO_2$ (570 mg, 9.4 mmol) was added in one portion followed by the aldehyde (0.94 mmol). After 1 hour, the reaction temperature was warmed to -15 ^{o}C and saturated NH_4Cl solution was added upon completion of the reaction. The mixture was warmed to room temperature, extracted with $CHCl_3$ and the combined organic phases were dried over anhydrous $MgSO_4$. The solvents were removed under reduced pressure and the residue was purified by flash column chromatography (15% EtOAc in hexanes).

2. Characterization of Nitroaldol Products

All commercially available reagents and solvents were used without further purification. NMR spectra were obtained at 300 MHz (^{1}H NMR) and 75 MHz (^{13}C NMR). Chemical shifts are reported in ppm relative to TMS. Reaction products were purified by column chromatography on silica gel (particle size 32-63 μm) as indicated below.

2-Nitro-1-phenylethanol[1]

Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 144 mg (0.86 mmol, 92%, 92% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.98 (b, 1H), 4.49 (dd, *J* = 2.9 Hz, 13.2 Hz, 1H), 4.59 (dd, *J* = 9.5 Hz, 13.2 Hz, 1H), 5.43 (dd, *J* = 2.9 Hz, 9.5 Hz, 1H), 7.34-7.40 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 71.6, 81.9, 126.6, 129.6, 129.7, 138.8. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (8:92) as the mobile phase, t_1 = 13.1 min, t_2 = 15.3, min, α = 1.18.

2-Nitro-1-(1-naphthyl)ethanol[2]

Chromatographic purification (hexanes:ethyl acetate = 85: 15) gave 187 mg (0.86 mmol, 92%, 90% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.29 (d, *J* = 4.2 Hz, 1H), 4.46 (d, *J* = 5.5 Hz, 2H), 6.12 (m, 1H), 7.37 (dd, *J* = 8.1 Hz, 8.1 Hz, 1H), 7.38-7.49 (m, 2H), 7.58 (d, *J* = 7.1 Hz, 1H), 7.73 (d, *J* = 8.3 Hz, 1H), 7.80 (dd, *J* = 2.2 Hz, 7.1 Hz, 1H), 7.90 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 68.7, 81.2, 122.4, 124.3, 126.0, 126.6, 127.5, 129.7, 129.8, 130.0, 134.1. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (20:80) as the mobile phase, t_1 = 7.8 min, t_2 = 10.2, min, α = 1.33.

2-Nitro-1-(2-naphthyl)ethanol[3]

Chromatographic purification (hexanes:ethyl acetate = 85: 15) gave 194 mg (0.89 mmol, 95%, 92% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.18 (d, *J* = 3.9 Hz, 1H), 4.48 (dd, *J* = 2.7 Hz, 13.2 Hz, 1H), 4.59 (dd, *J* = 9.3 Hz, 13.2 Hz, 1H), 5.51 (ddd, *J* = 2.7 Hz, 3.9 Hz, 9.3 Hz, 1H), 7.37 (dd, *J* = 1.7 Hz, 8.3 Hz, 1H), 7.45-7.51 (m, 2H), 7.76-7.81 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 71.7, 81.7, 123.8, 125.9, 127.3, 127.4, 128.4, 128.7, 129.5, 133.7, 134.0, 136.0. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (25:75) as the mobile phase, t_1 = 8.9 min, t_2 = 11.4, min, α = 1.29.

2-Nitro-1-(4-bromophenyl)ethanol[4]
Chromatographic purification (hexanes:ethyl acetate = 95: 5) gave 221 mg (0.90 mmol, 96%, 94% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.30 (d, *J* = 3.9 Hz, 1H), 4.47 (dd, *J* = 3.4 Hz, 13.2 Hz, 1H), 4.54 (dd, *J* = 9.0 Hz, 13.2 Hz, 1H), 5.38 (ddd, *J* = 3.4 Hz, 3.9 Hz, 9.0 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 70.9, 81.5, 123.5, 128.3, 132.7, 137.7. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (8:92) as the mobile phase, t_1 = 14.5 min, t_2 = 17.1, min, α = 1.20.

2-Nitro-1-(4-nitrophenyl)ethanol[1]
Chromatographic purification (hexanes:ethyl acetate = 95: 5) gave 175 mg (0.83 mmol, 88%, 95% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 4.62-4.64 (m 2H), 4.64 (dd, *J* = 5.1 Hz, 7.6 Hz, 1H), 7.64 (d, *J* = 8.6 Hz, 2H), 8.24 (d, *J* = 8.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 70.6, 81.3, 124.7, 127.6, 145.9, 148.6. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (15:85) as the mobile phase, t_1 = 11.2 min, t_2 = 12.7, min, α = 1.19.

2-Nitro-1-(4-cyanophenyl)ethanol[5]
Chromatographic purification (hexanes:ethyl acetate = 95: 5) gave 159 mg (0.83 mmol, 88%, 95% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.19 (d, *J* = 4.2Hz, 1H), 4.54 (dd, *J* = 2.0 Hz, 11.5 Hz, 1H), 4.60 (dd, *J* = 6.1 Hz, 11.5 Hz, 1H), 5.55 (ddd, *J* = 2.0 Hz, 4.2 Hz, 6.1 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.71 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 70.8, 81.3, 113.4, 118.9, 127.4, 133.5, 143.8. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (8:92) as the mobile phase, t_1 = 24.1 min, t_2 = 27.4, min, α = 1.14.

2-Nitro-1-(4-fluorophenyl)ethanol[2]
Chromatographic purification (hexanes:ethyl acetate = 95: 5) gave 155 mg (0.84 mmol, 89%, 94% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.24 (d, *J* = 3.7 Hz, 1H), 4.46 (dd, *J* = 3.2 Hz, 13.2 Hz, 1H), 4.55 (dd, *J* = 9.3 Hz, 13.2 Hz, 1H), 5.40 (ddd, *J* = 3.2 Hz, 3.7 Hz, 9.3 Hz, 1H), 7.05 (dd, *J* = 8.5 Hz, 8,8 Hz, 2H), 7.34 (dd, *J* = 8.8 Hz, 10.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 70.9, 81.7, 116.6 (d, $J_{C,F}$ = 21.7 Hz), 128.4 (d, $J_{C,F}$ = 8.6 Hz), 134.6 (d, $J_{C,F}$ = 3.0 Hz), 163.5 (d, $J_{C,F}$ = 247.8 Hz). The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (8:92) as the mobile phase, t_1 = 10.7 min, t_2 = 12.5, min, α = 1.17.

2-Nitro-1-(4-methoxyphenyl)ethanol[1]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 180 mg (0.91 mmol, 97%, 95% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.04 (d, *J* = 3.7 Hz, 1H), 3.79 (s, 3H), 4.44 (dd, *J* = 3.2 Hz, 12.9 Hz, 1H), 4.65 (dd, *J* = 9.5 Hz, 12.9 Hz, 1H), 5.34 (ddd, *J* = 3.2 Hz, 3.7 Hz, 9.5 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 2H), 7.51 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 56.0, 71.2, 81.8, 115.0, 127.9, 130.9, 160.6. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (10:90) as the mobile phase, t_1 = 13.7 min, t_2 = 16.2, min, α = 1.18.

2-Nitro-1-(2,6-dimethylphenyl)ethanol
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 154 mg (0.79 mmol, 84%, 88% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.46 (s, 6H), 2.71 (b, 1H)), 4.36 (dd, *J* = 2.9 Hz, 13.4 Hz, 1H), 4.90 (dd, *J* = 10.5 Hz, 13.4 Hz, 1H), 5.90 (d, *J* = 10.5 Hz, 1H), 7.01 (d, *J* = 7.6 Hz, 2H), 7.13 (dd, *J* = 7.6 Hz, 7.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 21.3, 69.2, 79.1, 129.2, 130.4, 134.1, 137.2. Anal. calcd. for $C_{10}H_{13}NO_3$: C, 61.53; H, 6.71; N, 7.18. Found: C, 61.87; H, 6.79; N, 7.15. The ee was determined by HPLC on Chiralcel OD using EtOH:hexanes (10:90) as the mobile phase, t_1 = 8.5 min, t_2 = 10.5, min, α = 1.31.

2-Nitro-1-(3-thienyl)ethanol
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 156 mg (0.90 mmol, 96%, 91% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.15 (d, *J* = 3.7 Hz, 1H), 4.54 (dd, *J* = 3.7 Hz, 13.4 Hz, 1H), 4.63 (dd, *J* = 9.0 Hz, 13.4 Hz, 1H), 5.52 (ddd, *J* = 3.7 Hz, 3.7 Hz, 9.0 Hz, 1H), 7.06 (dd, *J* = 1.2 Hz, 4.9 Hz, 1H), 7.26 (m, 1H), 7.35 (dd, *J* = 3.2 Hz, 4.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 68.0, 81.2, 123.3, 125.7, 127.9, 140.0. Anal. calcd. for $C_6H_7NO_3S$: C, 41.61; H, 4.07; N, 8.09. Found: C, 41.53; H, 3.87; N, 8.08. The ee was determined by HPLC on Chiralcel OD using 2-propanol:hexanes (10:90) as the mobile phase, t_1 = 16.1 min, t_2 = 20.2, min, α = 1.29.

1-Nitro-4-phenyl-2-butanol[2]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 174 mg (0.89 mmol, 95%, 85% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 1.75-1.89 (m, 2 H), 2.64-2.88(m, 3H), .4.27 (m, 1H), 4.37 (d, *J* = 5.8 Hz, 1 H), 7.18-7.38 (m, 5 H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 31.9, 35.8, 68.5, 81.2, 126.9, 129.0, 129.3, 141.3. The ee was determined by HPLC on Chiralpak AD using 2-propanol:hexanes (10:90) as the mobile phase, t_1 = 9.9 min, t_2 = 12.2, min, α = 1.25.

1-Benzyloxy-3-nitro-2-propanol[6]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 184 mg (0.87 mmol, 93%, 75% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.99 (d, *J* = 5.4 Hz, 1H), 3.53 (d, *J* = 4.6 Hz, 1H), 4.43-4.49 (m, 2H), 4.53 (d, *J* = 2.2 Hz, 1H), 7.28-7.36 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 68.3, 71.0, 74.1, 78.7, 128.4, 128.6, 129.1, 137.8. The ee was determined by HPLC on Chiralcel OD using 2-propanol:hexanes (10:90) as the mobile phase, t_1 = 18.7 min, t_2 = 21.4, min, α = 1.18.

1-Nitro-2-heptanol[3]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 136 mg (0.85 mmol, 90%, 84% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.89 (t, *J* = 6.8 Hz, 3H), 1.28-1.40 (m, 5H), 1.41-1.53 (m, 3H), 2.80 (d, *J* = 4.9 Hz, 1H), 4.22-4.26 (m, 1H), 4.39 (dd, *J* = 8.3 Hz, 13.2 Hz, 1H), 4.42 (dd, *J* = 2.9 Hz, 13.2 Hz, 1H), ^{13}C NMR (75 MHz, $CDCl_3$) δ = 14.6, 23.1, 25.5, 32.1, 34.3, 69.4, 81.3. The ee was determined by GC on octakis(6-*O*-methyl-2,3-di-*O*-pentyl)-γ-cyclodextrin, 75 ^{o}C, t_1 = 34.2 min, t_2 = 36.7 min, α = 1.04.

1-Nitro-4-phenyl-but-3-en-2-ol[5]

Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 179 mg (0.93 mmol, 98%, 82% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.92 (b, 1H); 4.48 (d, *J* = 6.1 Hz, 2H); 5.02 (m, 1H); 6.10 (dd, *J* = 6.4 Hz, 15.9 Hz, 1H), 6.77 (d, *J* = 15.9 Hz, 1H), 7.24-7.39 (5H, m). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 70.2, 80.5, 125.6, 127.4, 129.1, 129.4, 134.2, 136.2. The ee was determined by HPLC on Chiralpak AD using EtOH:hexanes (10:90) as the mobile phase, t_1 = 18.7 min, t_2 = 22.2, min, α = 1.18.

3,3-Dimethyl-1-nitro-2-butanol[2]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 127 mg (0.86 mmol, 92%, 86% ee) of a colorless oil.^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.96 (s, 9H), 4.01 (ddd, *J* = 2.2 Hz, 5.1 Hz, 10.3 Hz, 1H), 4.36 (dd, *J* = 10.3 Hz, 12.9 Hz, 1H), 4.53 (dd, *J* = 2.2 Hz, 12.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 26.1, 34.9, 76.9, 78.9. The ee was determined by GC on octakis(6-*O*-methyl-2,3-di-*O*-pentyl)-γ-cyclodextrin, 70 ^{o}C, t_1 = 25.3 min, t_2 = 27.6.4 min, α = 1.09.

1-Cyclohexyl-2-nitro-ethanol[1]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 152 mg (0.88 mmol, 94%, 83% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 1.02-1.32 (m, 5H), 1.48 (m, 1H), 1.61-1.72 (m, 2H), 1.78-1.89 (m, 3H), 2.68 (d, *J* = 5.1 Hz, 1H), 4.11 (m, 1H), 4.40 (dd, *J* = 8.8 Hz, 12.9 Hz, 1H), 4.50 (dd, *J* = 3.1 Hz, 12.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 26.4, 26.5, 26.7, 28.6, 29.4, 42.1, 73.5, 80.0. The ee was determined by GC on octakis(6-*O*-methyl-2,3-di-*O*-pentyl)-γ-cyclodextrin, 80 ^{o}C, t_1 = 19.5 min, t_2 = 21.5 min, α = 1.11.

3-Methyl-1-nitro-2-butanol[2]
Chromatographic purification (hexanes:ethyl acetate = 90: 10) gave 111 mg (0.85 mmol, 90%, 81% ee) of a white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.97 (d, *J* = 4.4 Hz, 3 H), 1.02 (d, *J* = 4.1 Hz, 3H), 1.81 (m, 1 H), 2.60 (d, *J* = 4.9 Hz, 1H), 4.11 (ddd, *J* = 3.2 Hz, 5.8 Hz, 8.5 Hz, 1 H), 4.39 (dd, *J* = 8.5 Hz, 13.2 Hz, 1 H), 4.47 (dd, *J* = 3.2 Hz, 13.2 Hz, 1 H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 18.1, 19.1, 32.4, 74.0, 79.9. The ee was determined by GC on octakis(6-*O*-methyl-2,3-di-*O*-pentyl)-γ-cyclodextrin, 65 ^{o}C, t_1 = 28.6 min, t_2 = 30.1 min, α = 1.05.

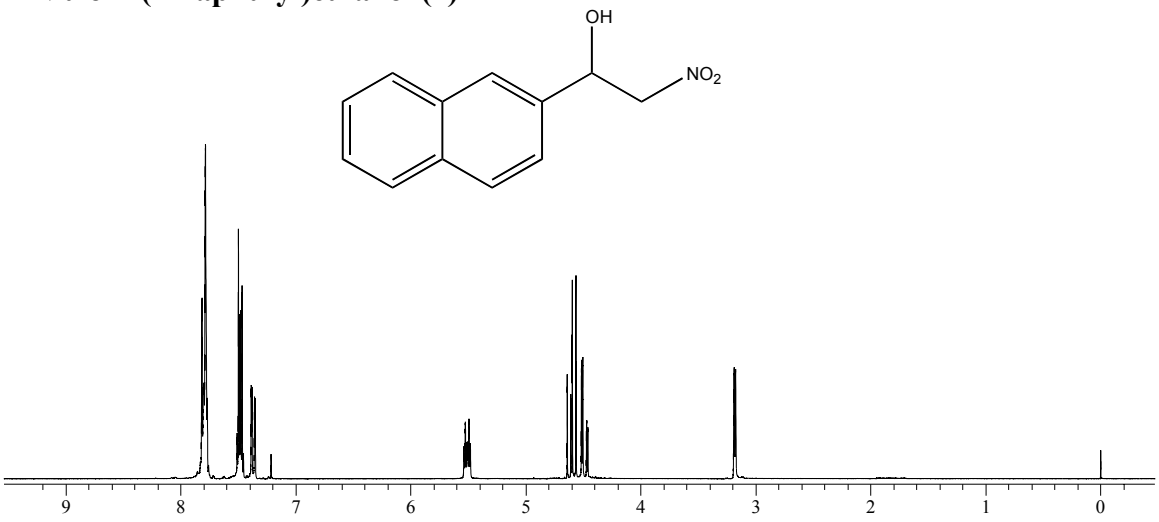
***Anti* 2-nitro-1-phenyl-1-propanol**[7]
Chromatographic purification (hexanes:ethyl acetate = 98: 2) gave 18.7 mg (0.10 mmol, 11%, 91% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 1.50 (d, *J* = 6.8 Hz,3H), 2.70 (d, *J* = 3.7 Hz, 1H), 4.69 (dq, *J* = 3.4 Hz, 6.8 Hz, 1H), 5.40 (dd, *J* = 3.4 Hz, 3.7 Hz, 1H), 7.35-7.42 (m,5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 12.7, 74.6, 88.1, 126.6, 129.2, 129.4, 139.1. The ee was determined by HPLC on Chiralpak AD using EtOH:hexanes (10:90) as the mobile phase, t_1 = 10.4 min, t_2 = 11.5, min, α = 1.12.

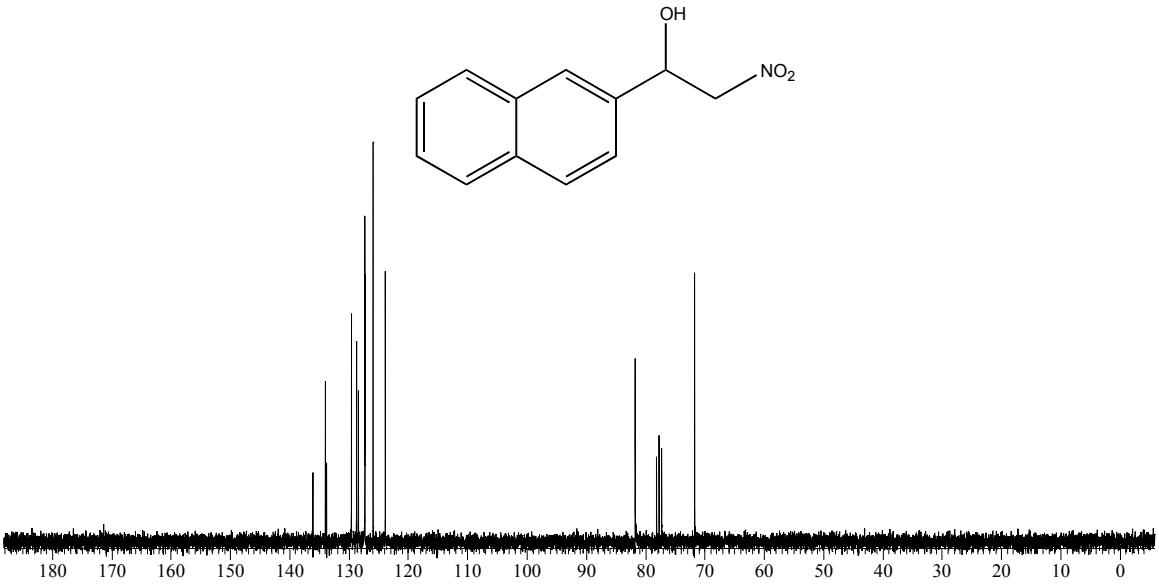
***Syn* 2-nitro-1-phenyl-1-propanol**7
Chromatographic purification (hexanes:ethyl acetate = 98: 2) gave 130 mg (0.72 mmol, 0.77%, 92% ee) of a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ = 1.31 (d, *J* = 6.8 Hz, 3H), 2.67 (d, *J* = 3.9 Hz, 1H), 4.76 (dq, *J* = 6.8 Hz, *J* = 9.0 Hz, 1H), 5.01 (dd, *J*= 3.9 Hz, 9.0 Hz, 1H), 7.34-7.42 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 17.1, 76.9, 89.1, 127.6,

129.7, 129.9, 139.0. The ee was determined by HPLC on Chiralpak AD using EtOH:hexanes (10:90) as the mobile phase, t_1 = 11.1 min, t_2 = 13.2, min, α = 1.22.

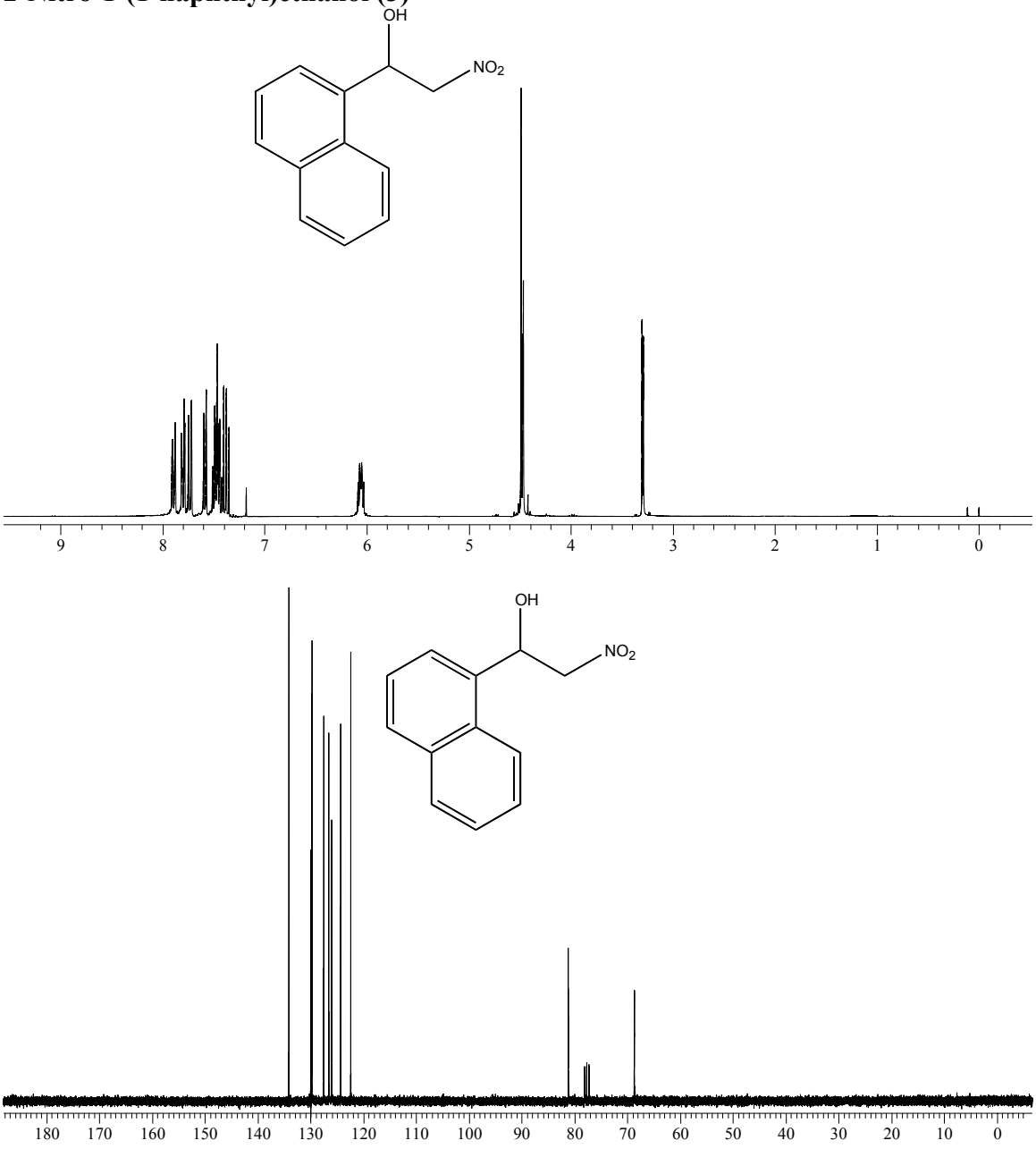
3. NMR Spectra·of Nitroaldol Products

2-Nitro-1-(2-naphthyl)ethanol (4)

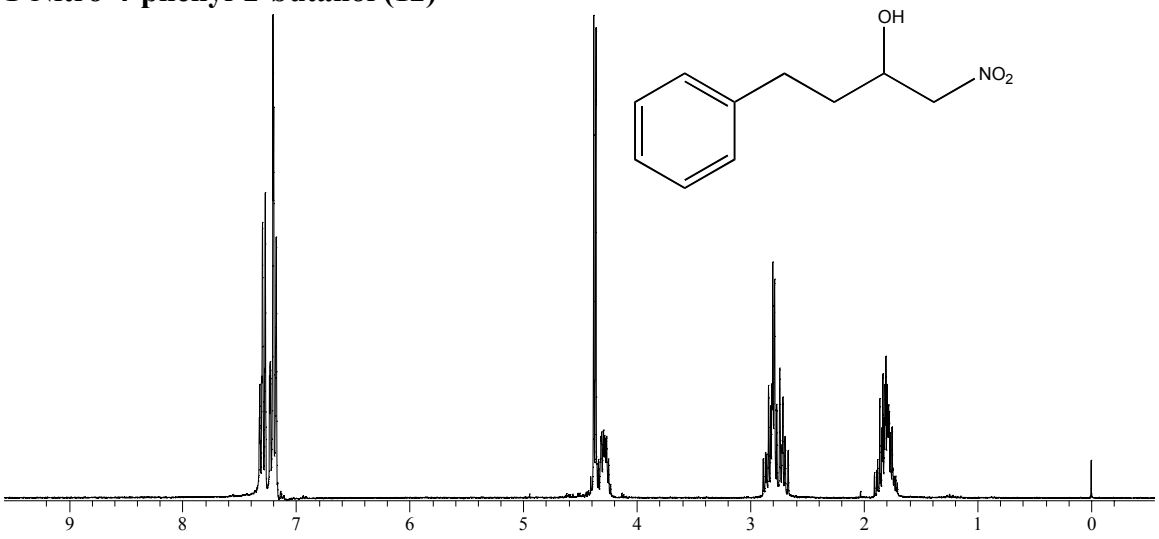


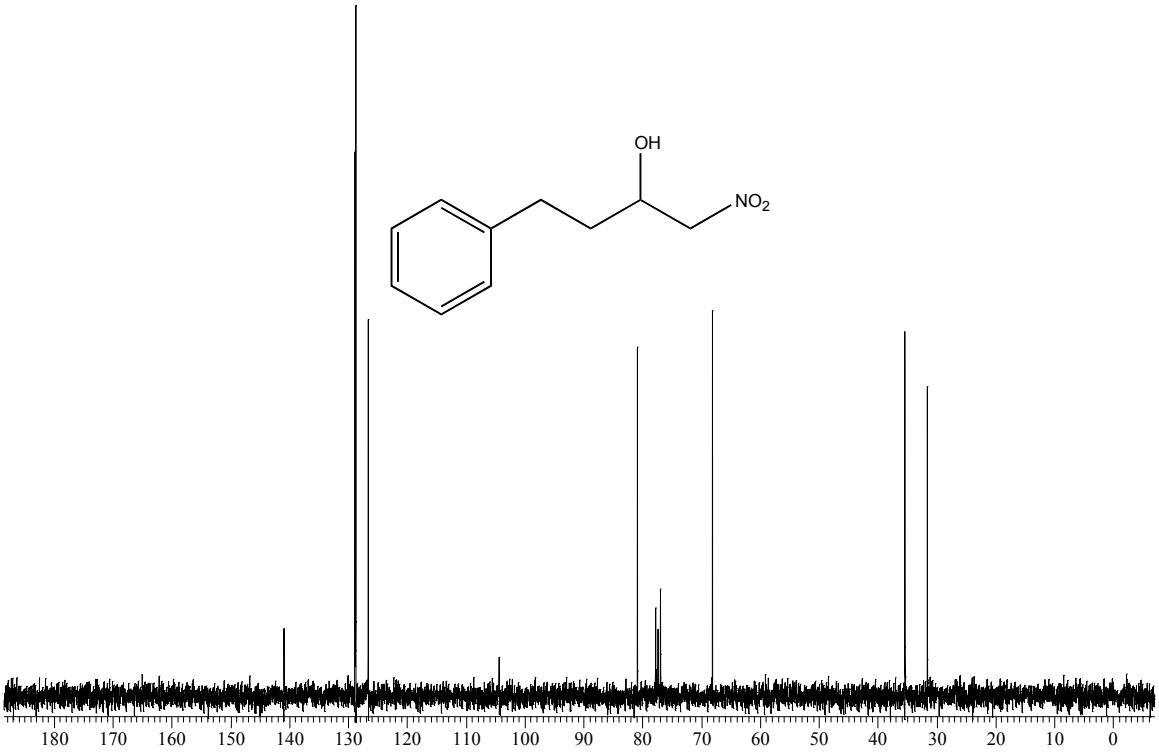


2-Nitro-1-(1-naphthyl)ethanol (3)

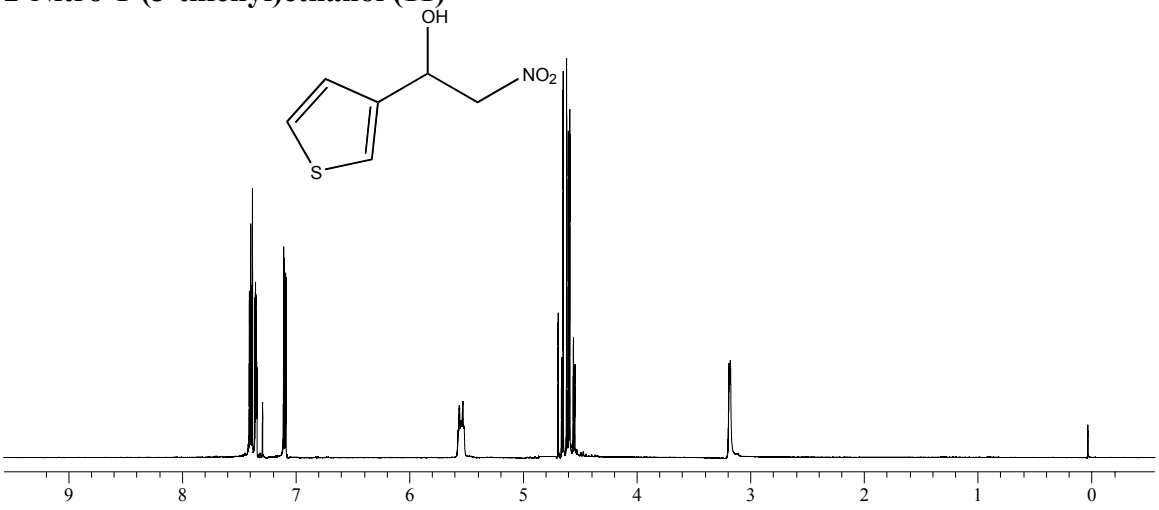


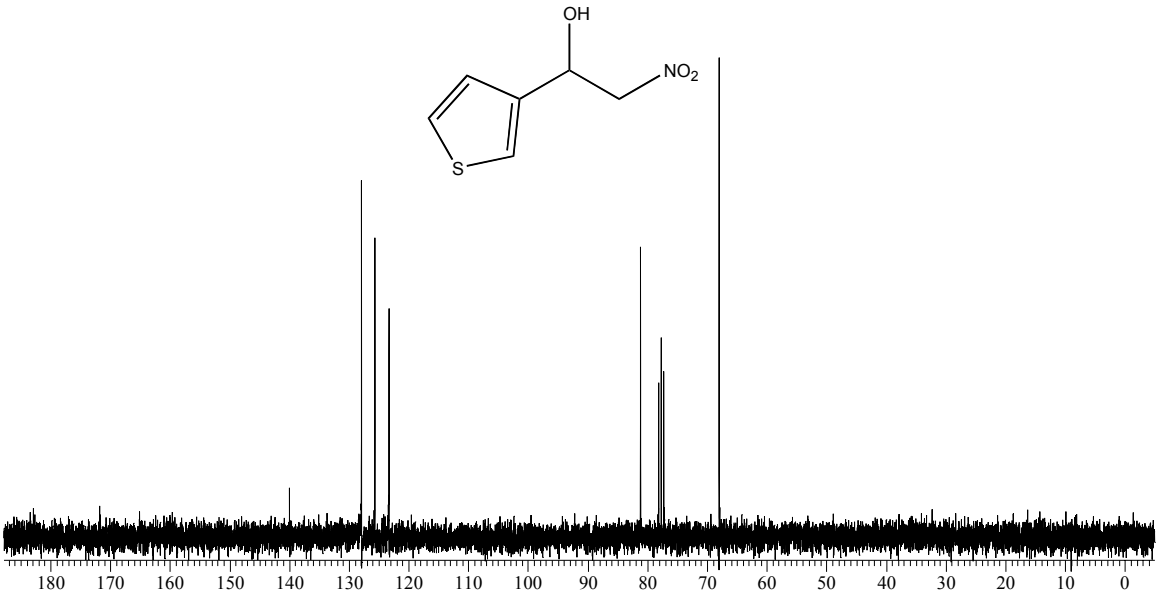
1-Nitro-4-phenyl-2-butanol (12)



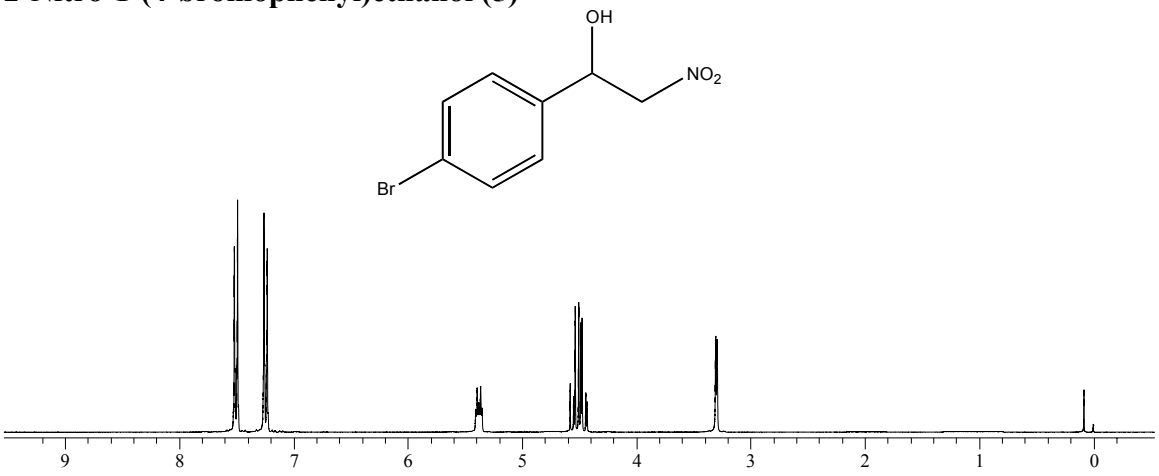


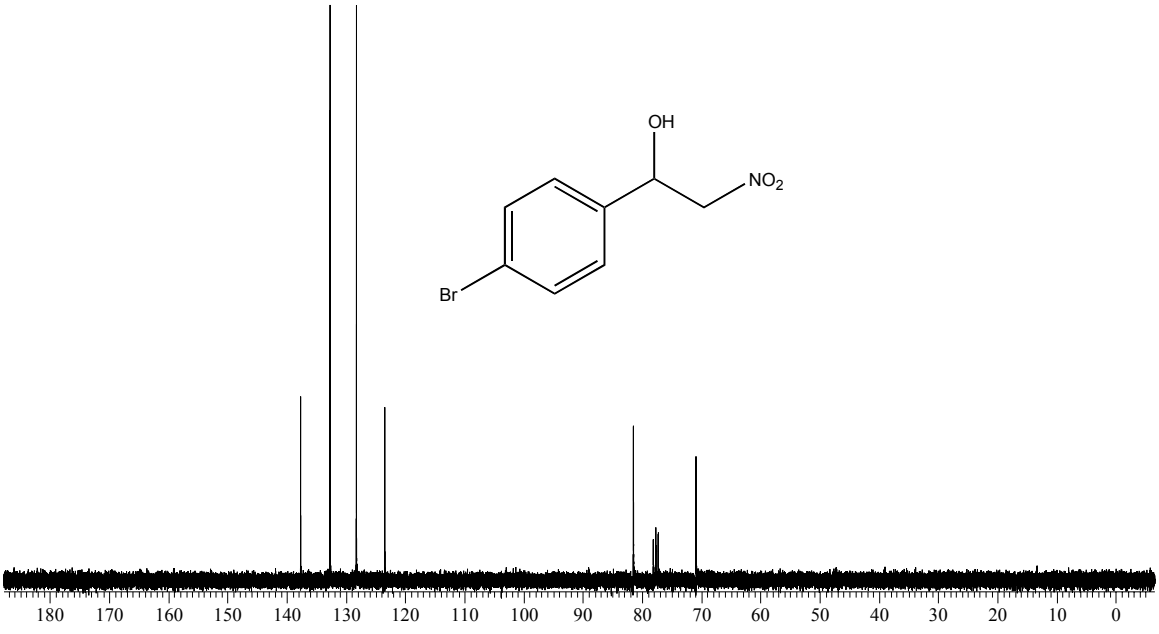
2-Nitro-1-(3-thienyl)ethanol (11)



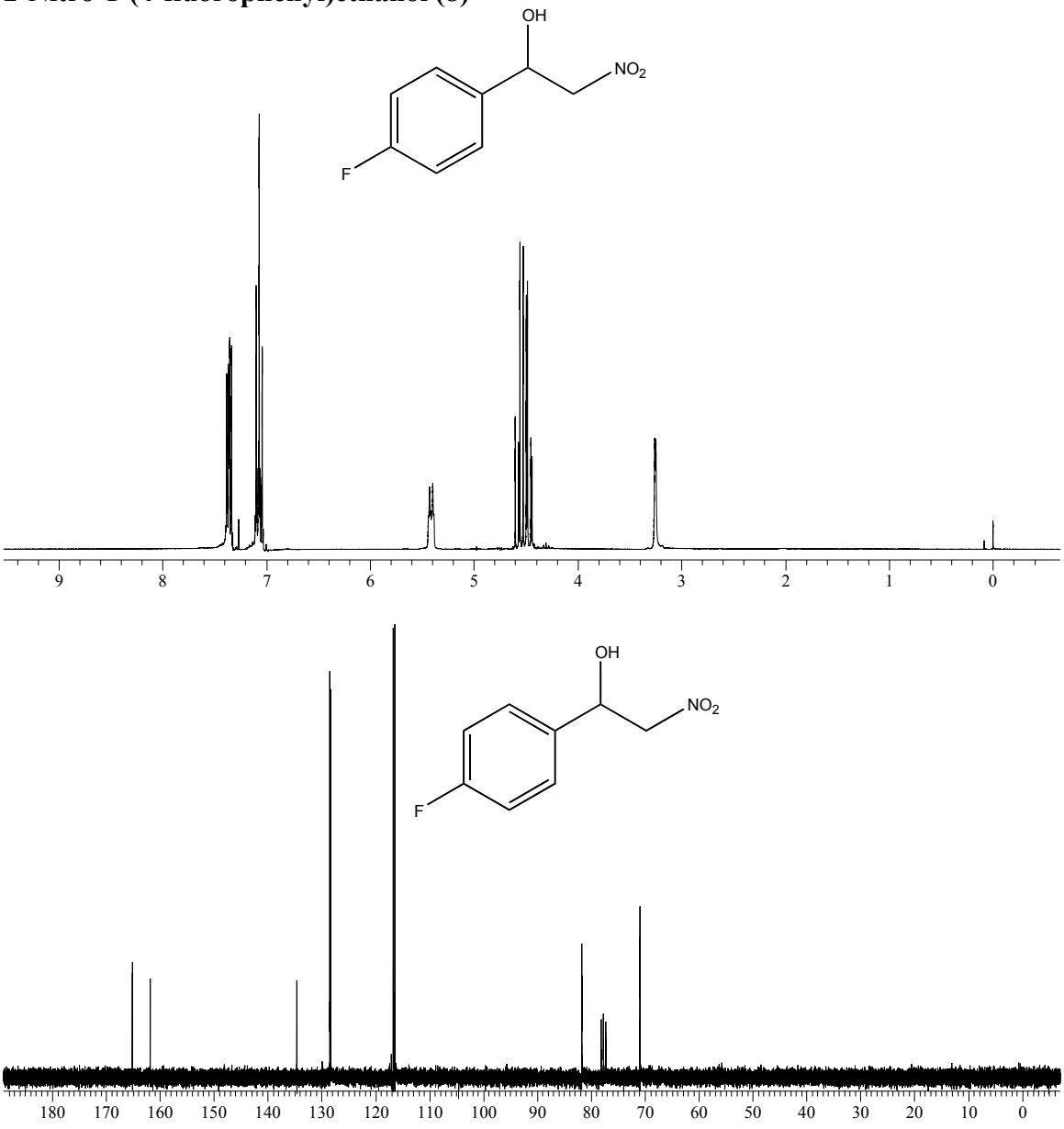


2-Nitro-1-(4-bromophenyl)ethanol (5)

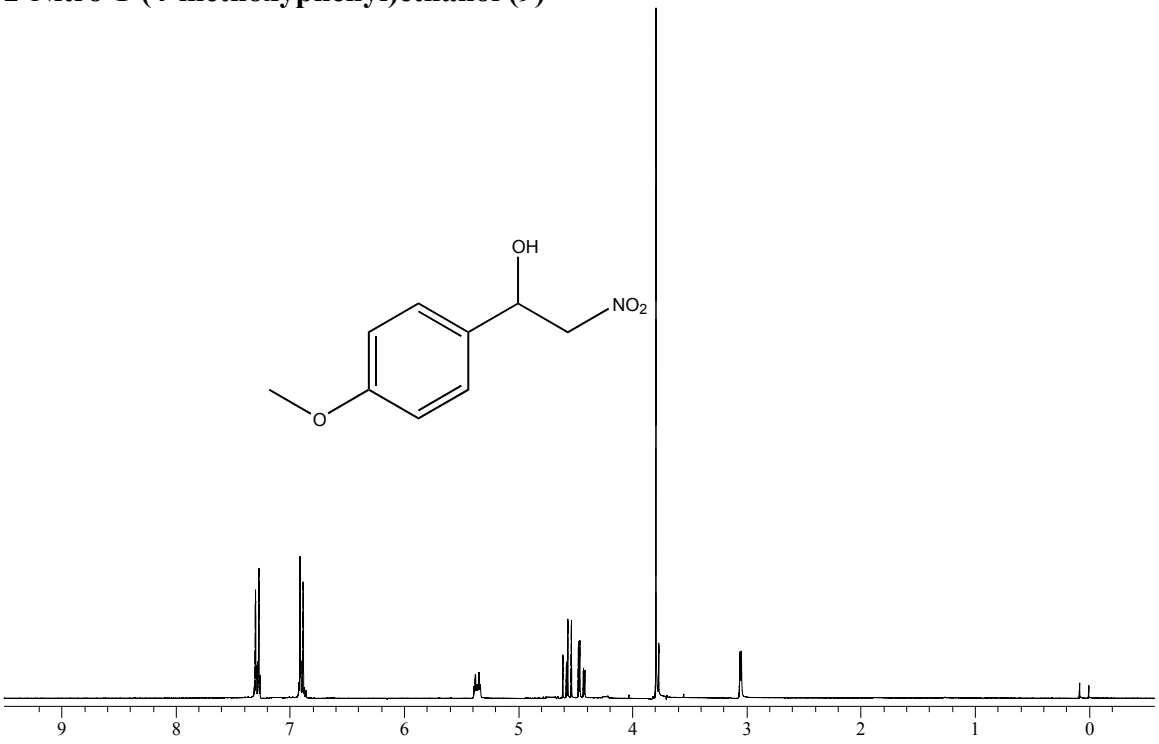


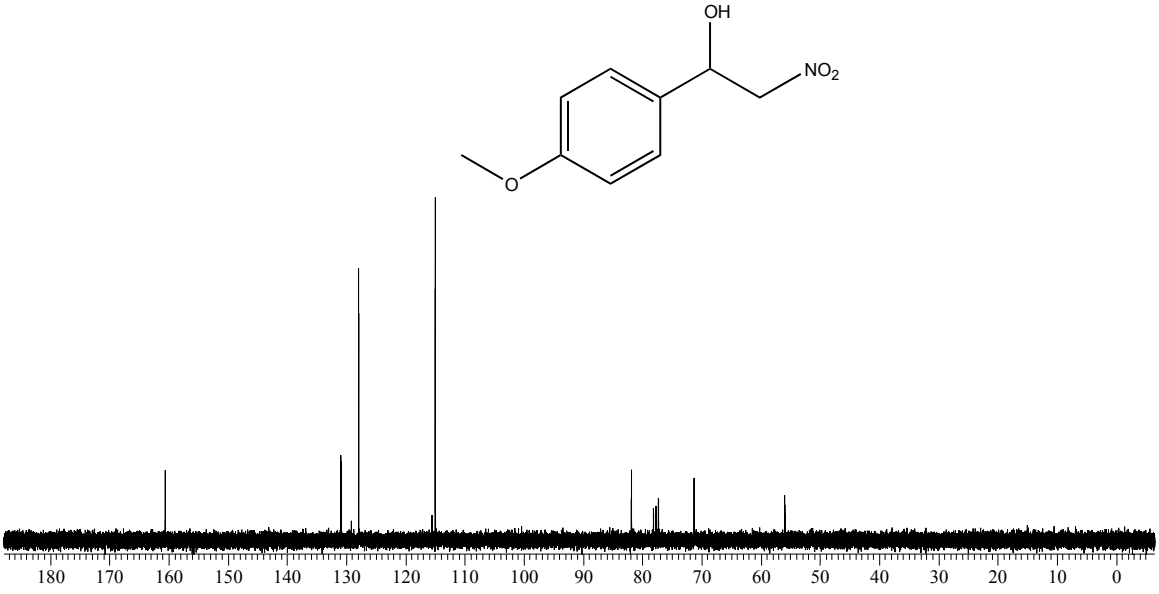


2-Nitro-1-(4-fluorophenyl)ethanol (8)

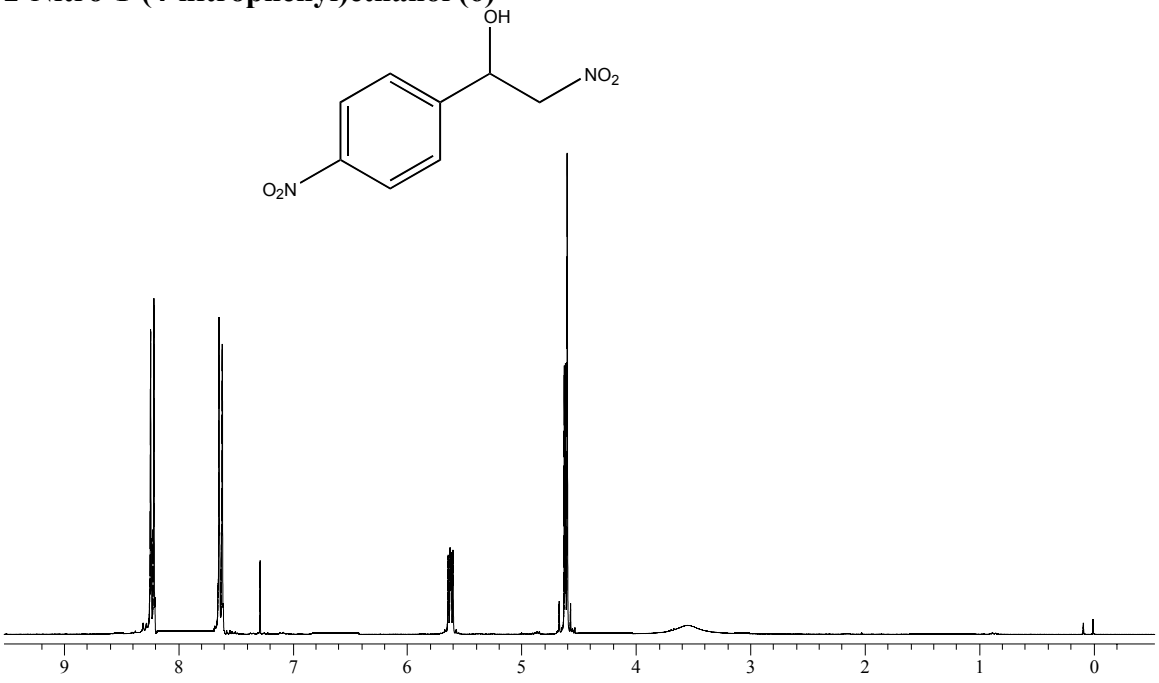


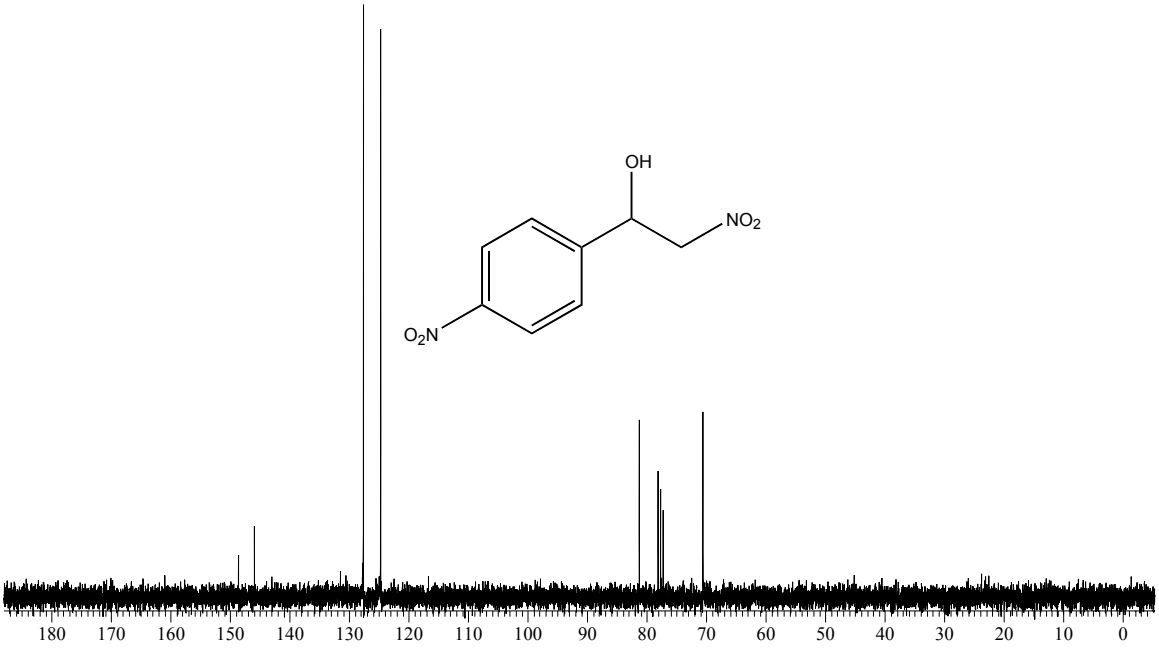
2-Nitro-1-(4-methoxyphenyl)ethanol (9)



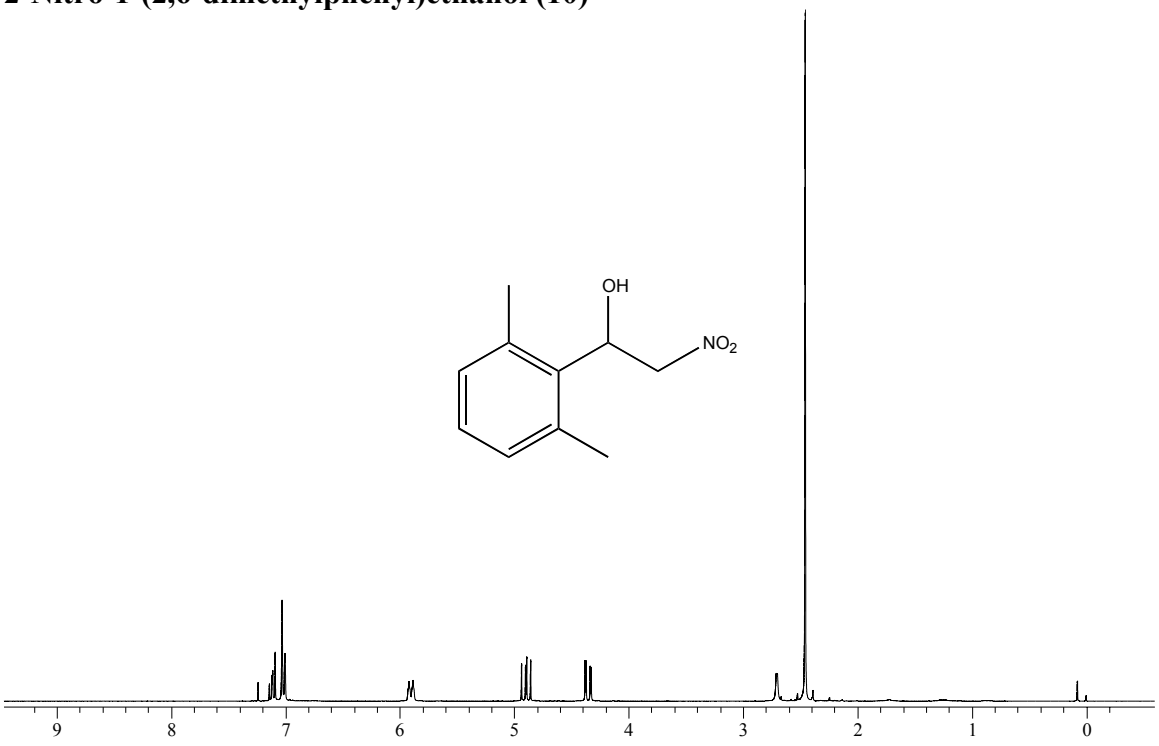


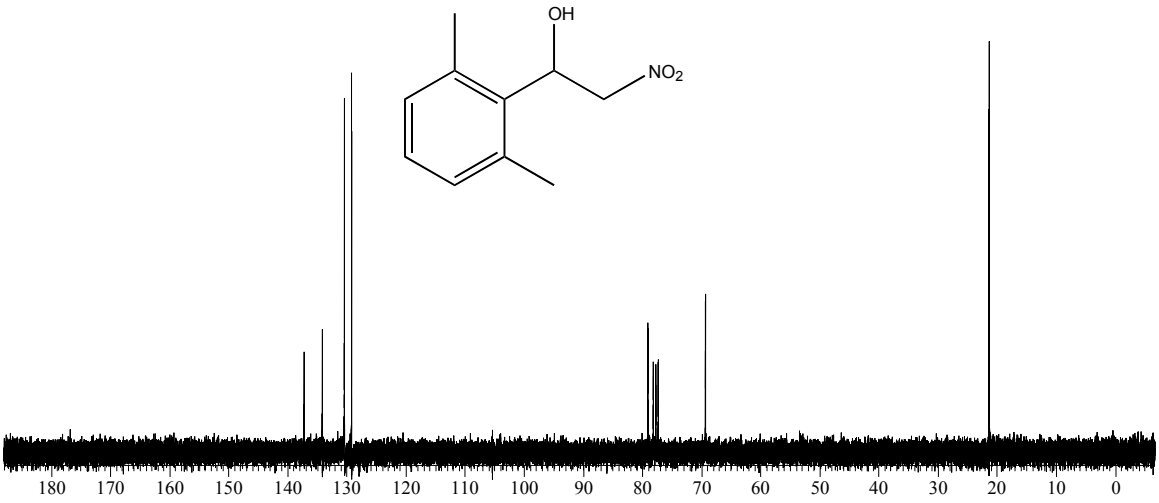
2-Nitro-1-(4-nitrophenyl)ethanol (6)



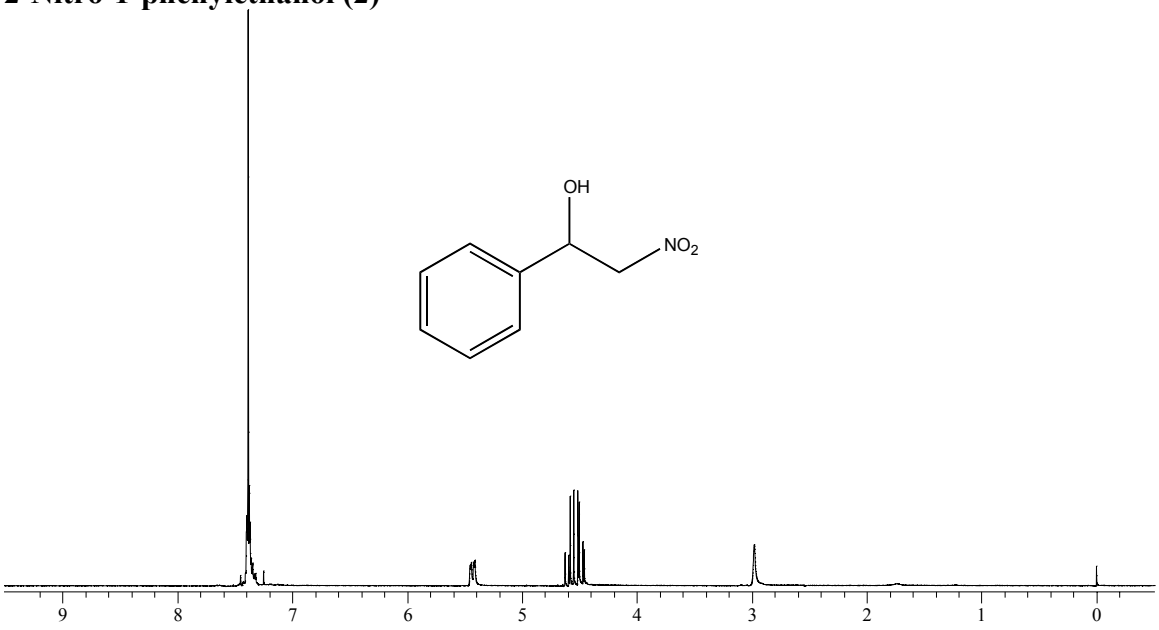


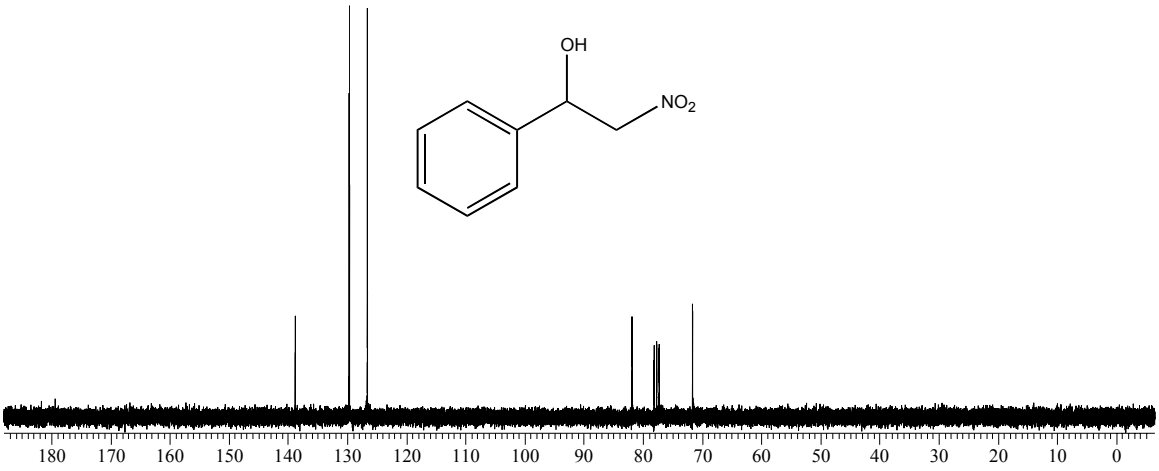
2-Nitro-1-(2,6-dimethylphenyl)ethanol (10)



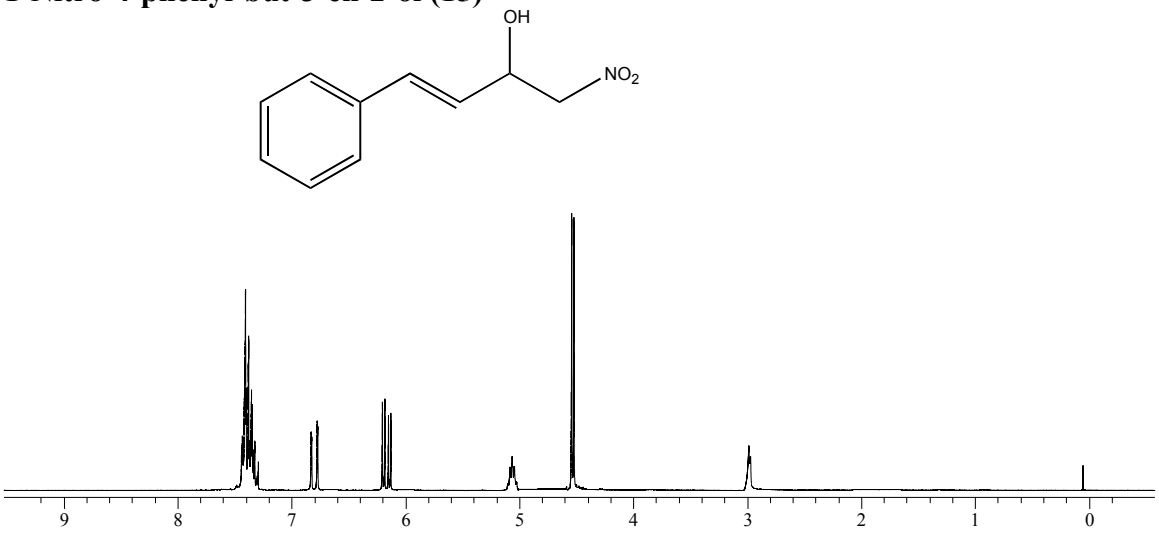


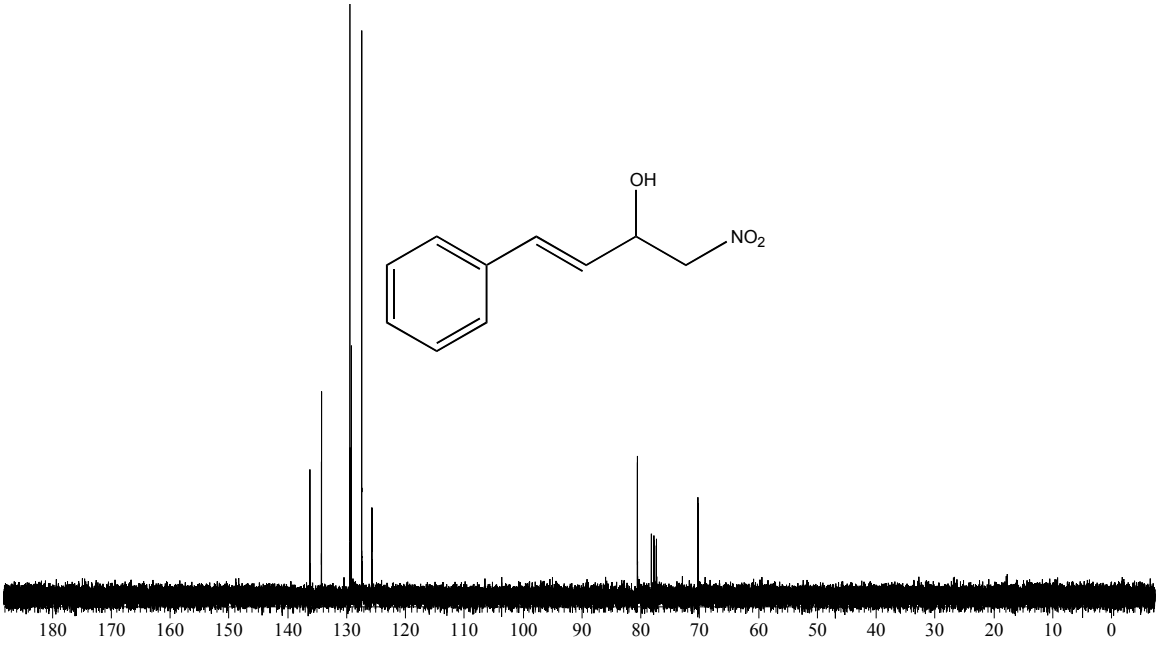
2-Nitro-1-phenylethanol (2)



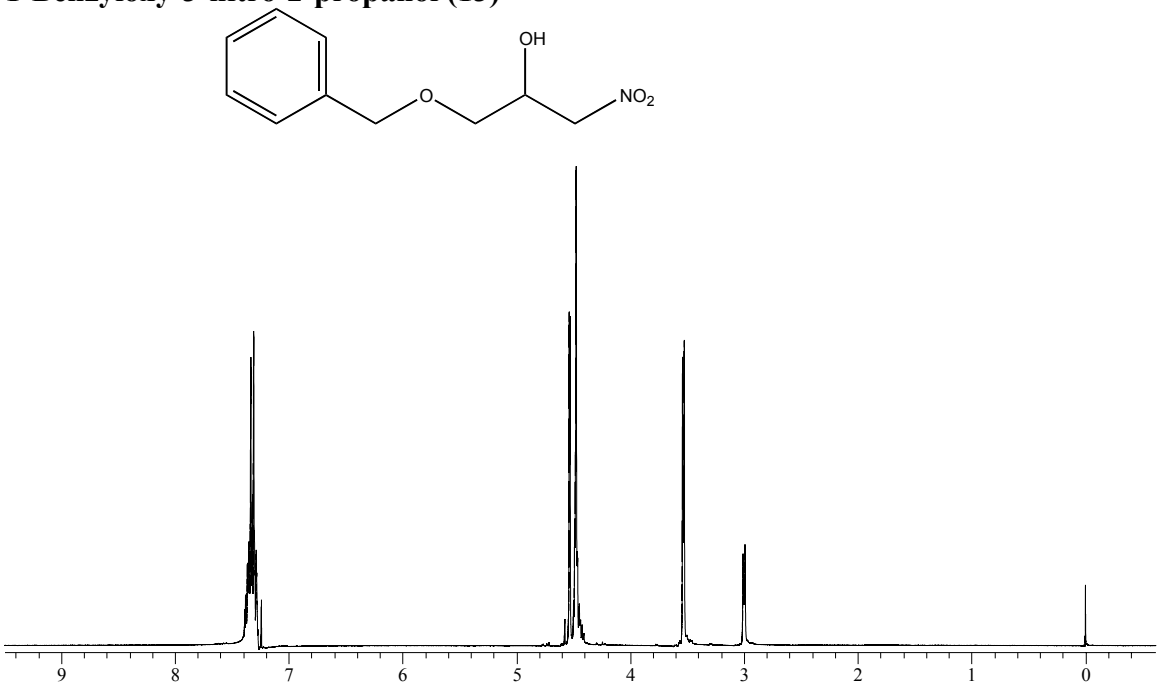


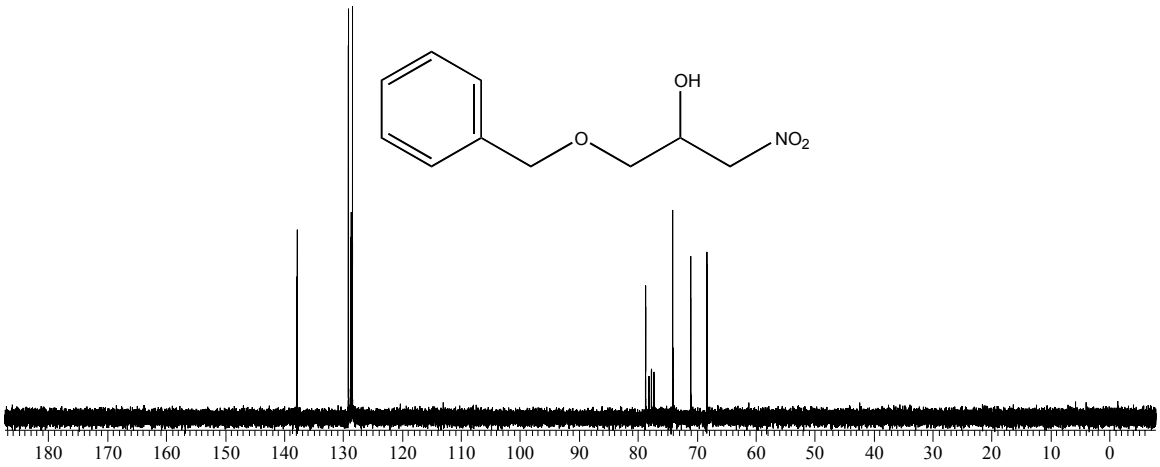
1-Nitro-4-phenyl-but-3-en-2-ol (15)



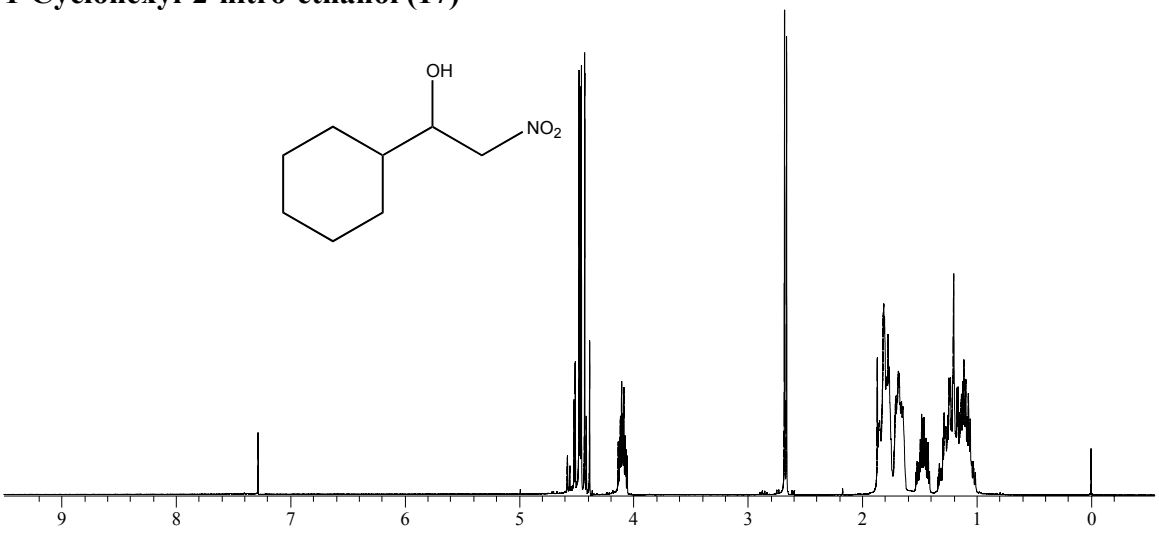


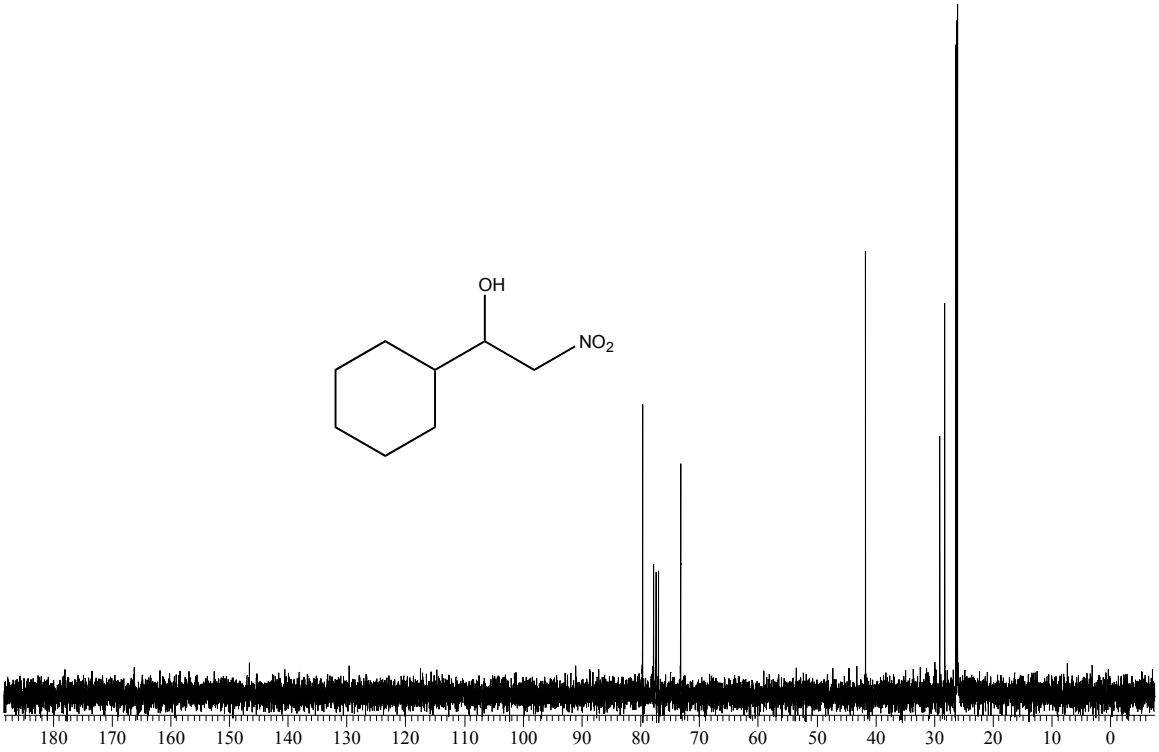
1-Benzyloxy-3-nitro-2-propanol (13)



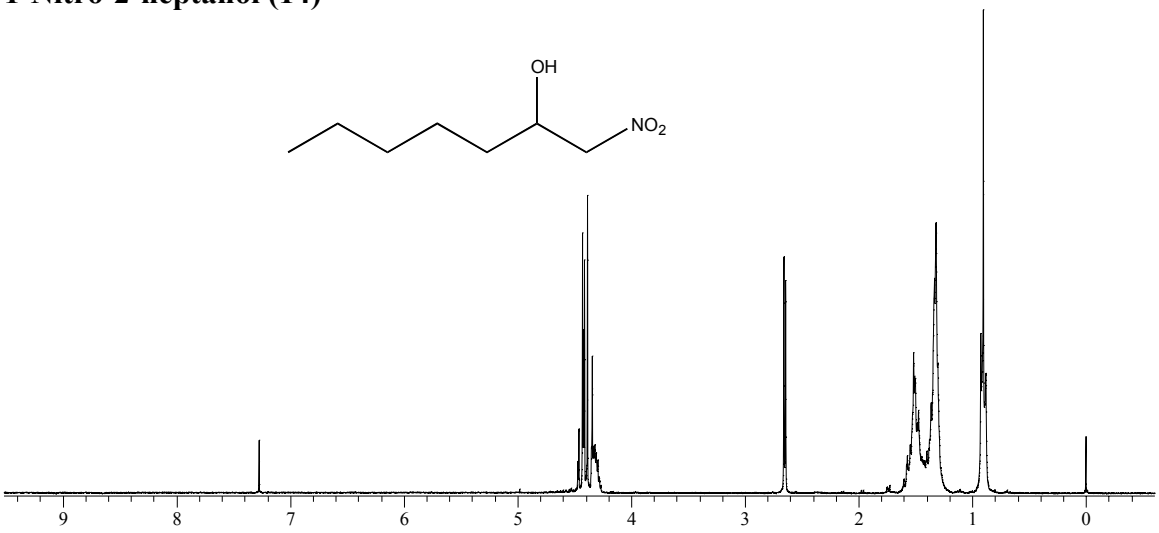


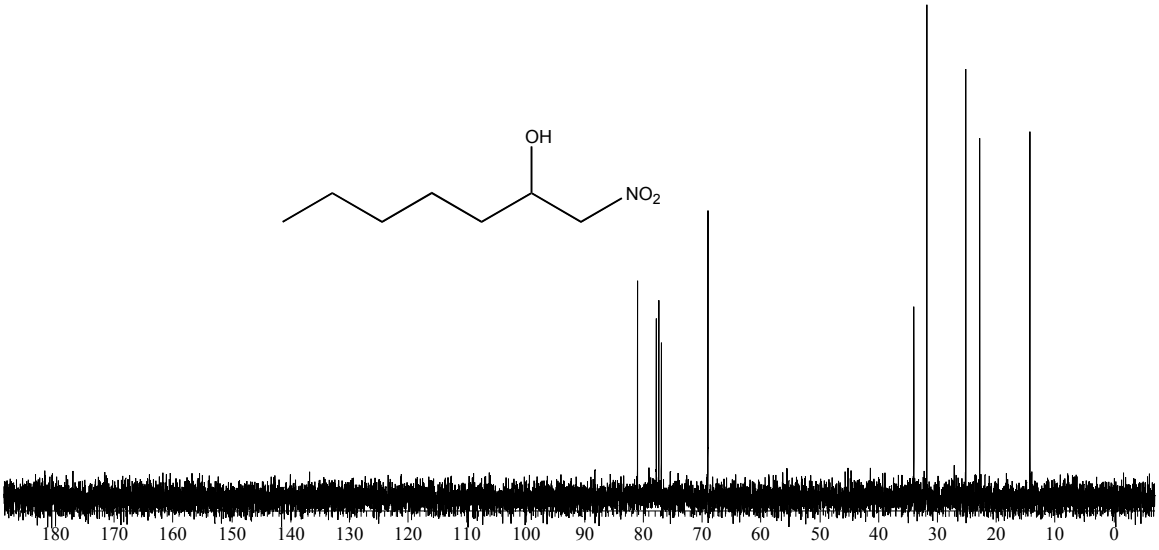
1-Cyclohexyl-2-nitro-ethanol (17)



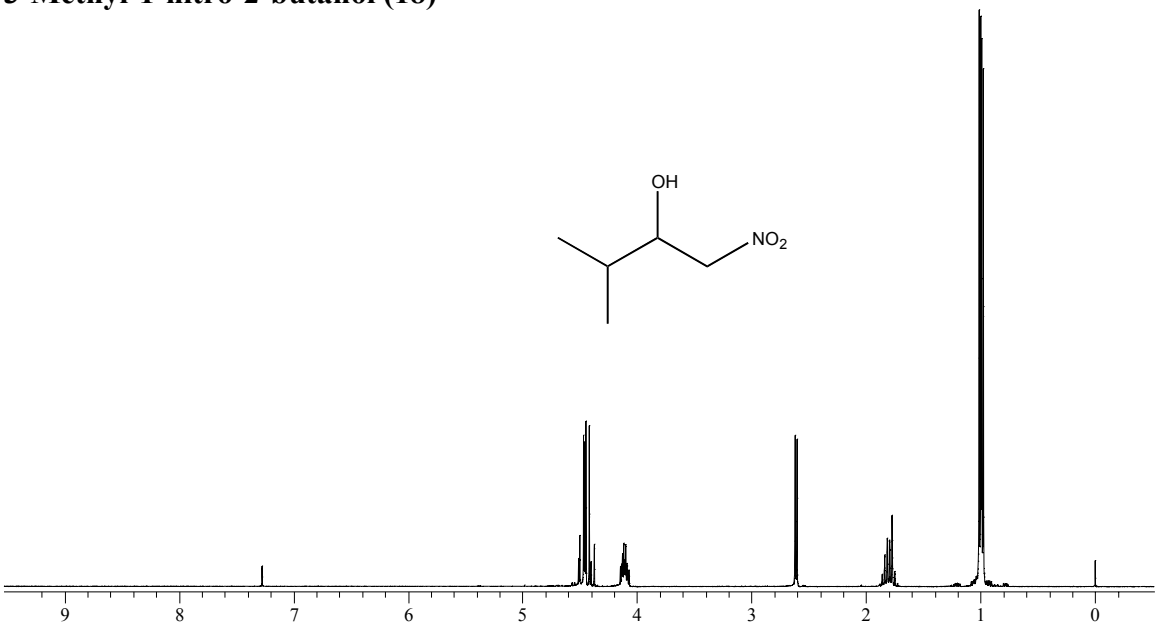


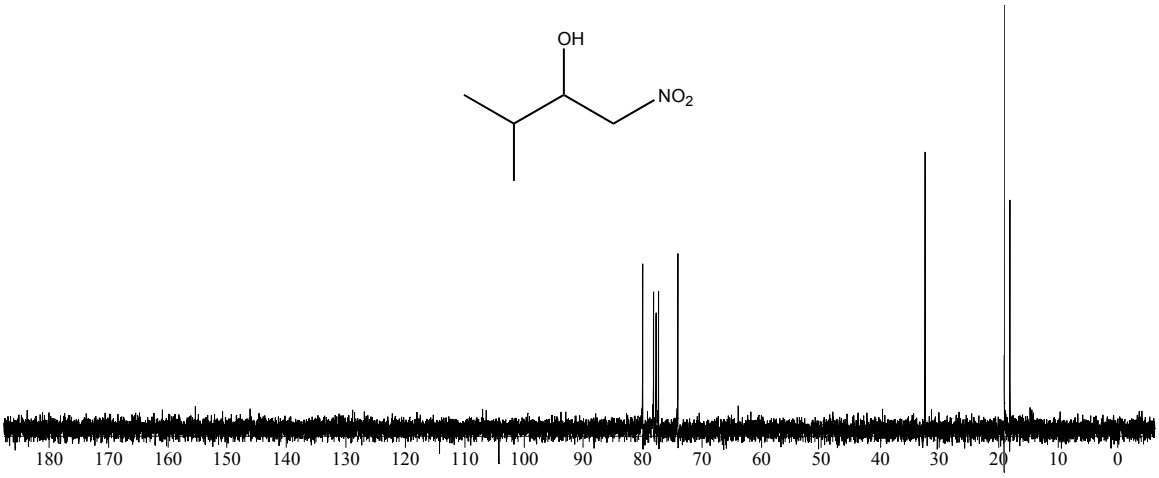
1-Nitro-2-heptanol (14)



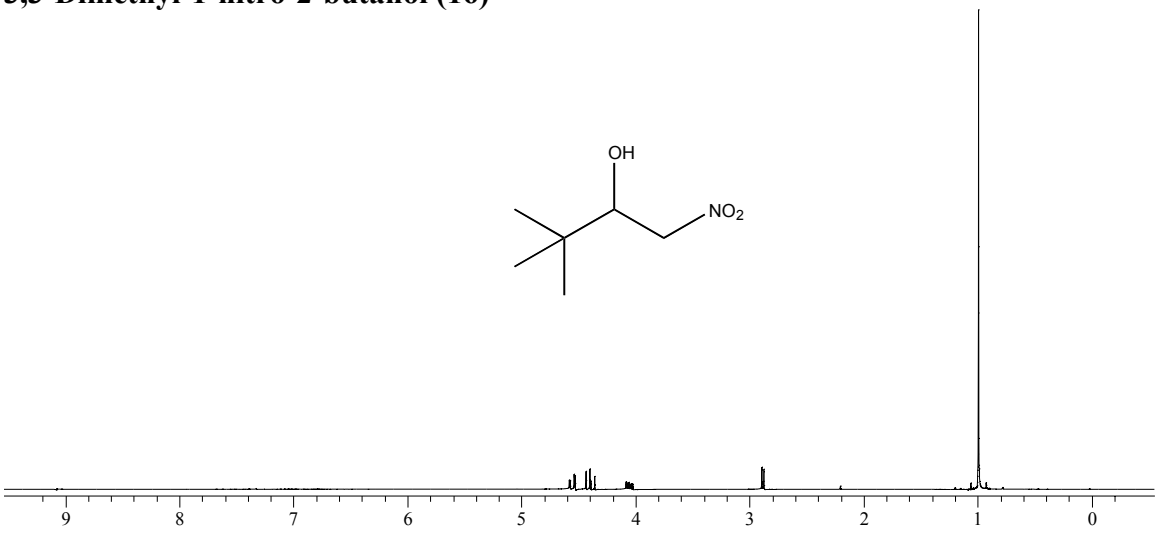


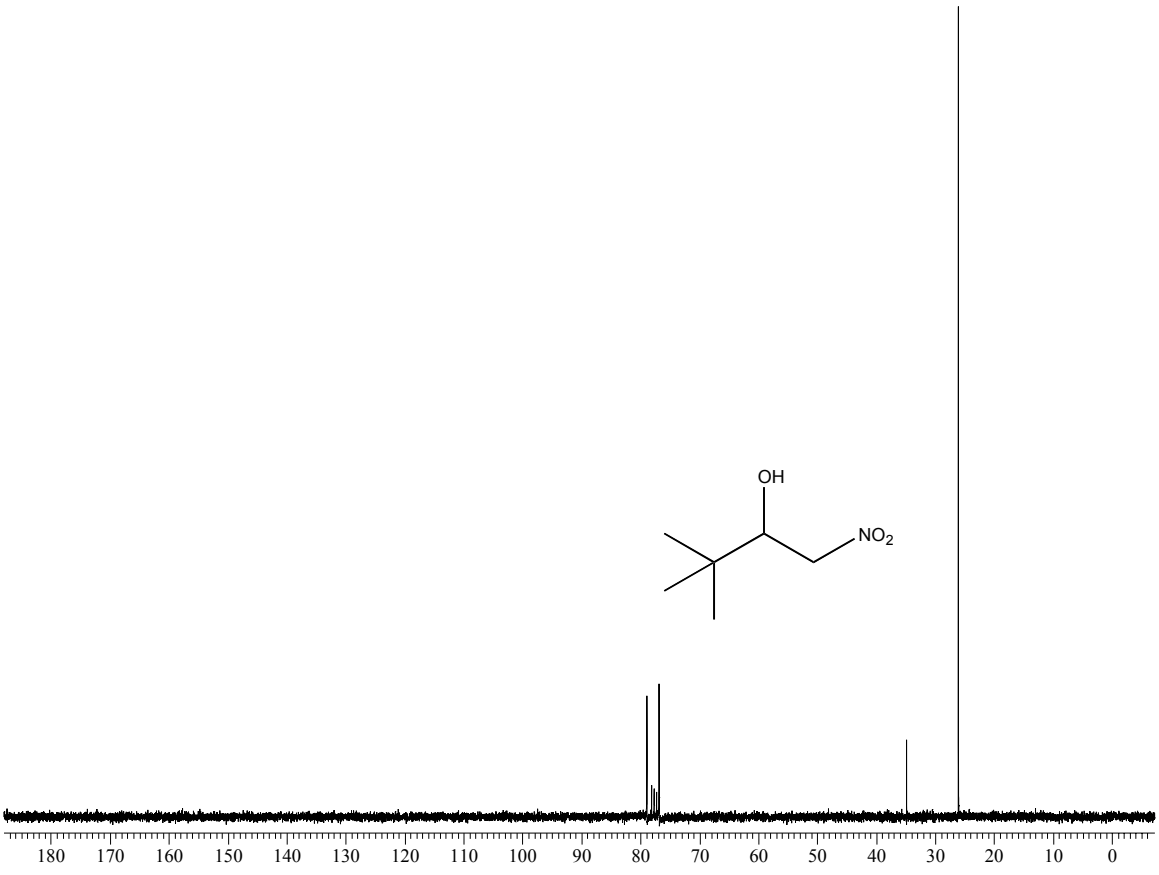
3-Methyl-1-nitro-2-butanol (18)



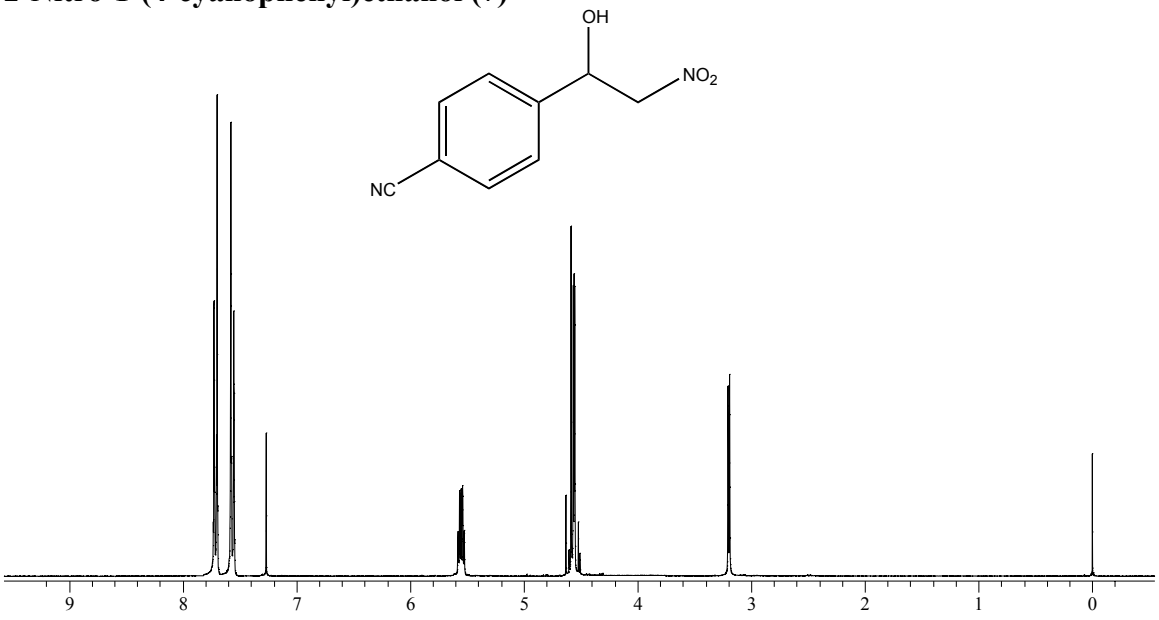


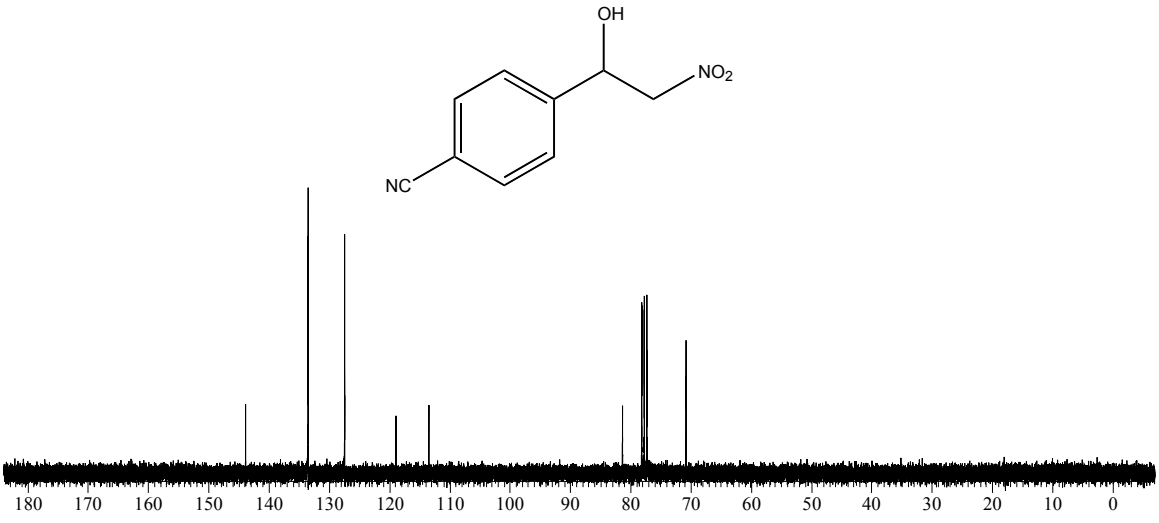
3,3-Dimethyl-1-nitro-2-butanol (16)



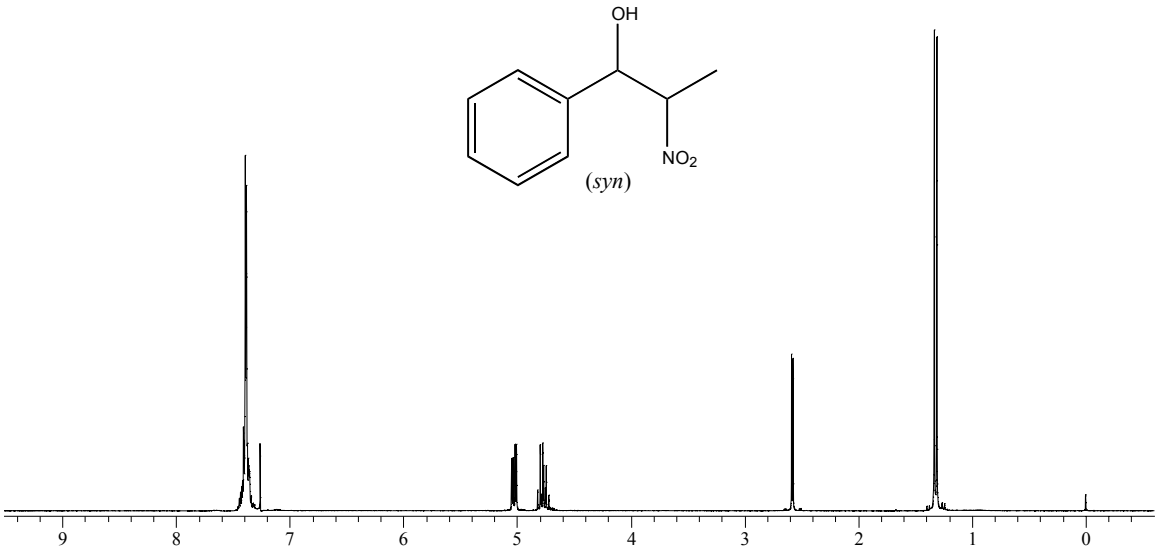


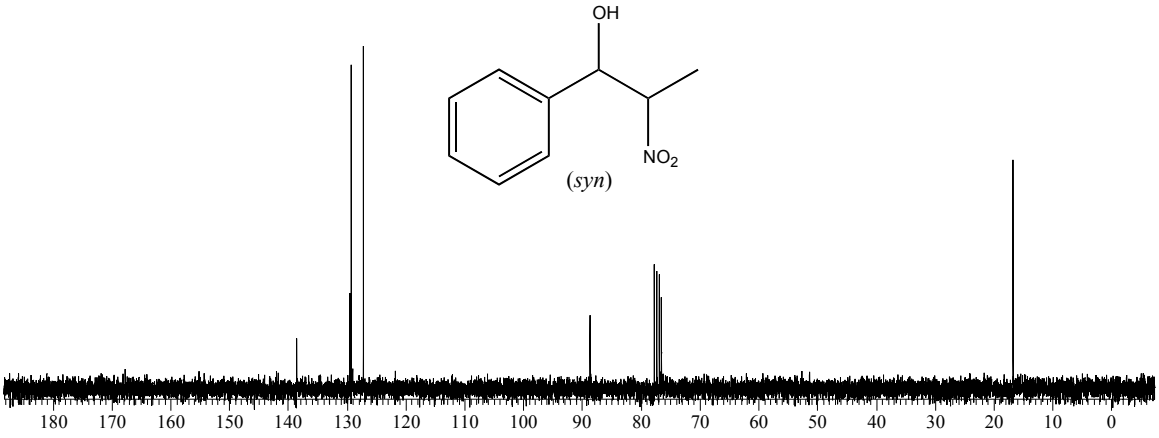
2-Nitro-1-(4-cyanophenyl)ethanol (7)



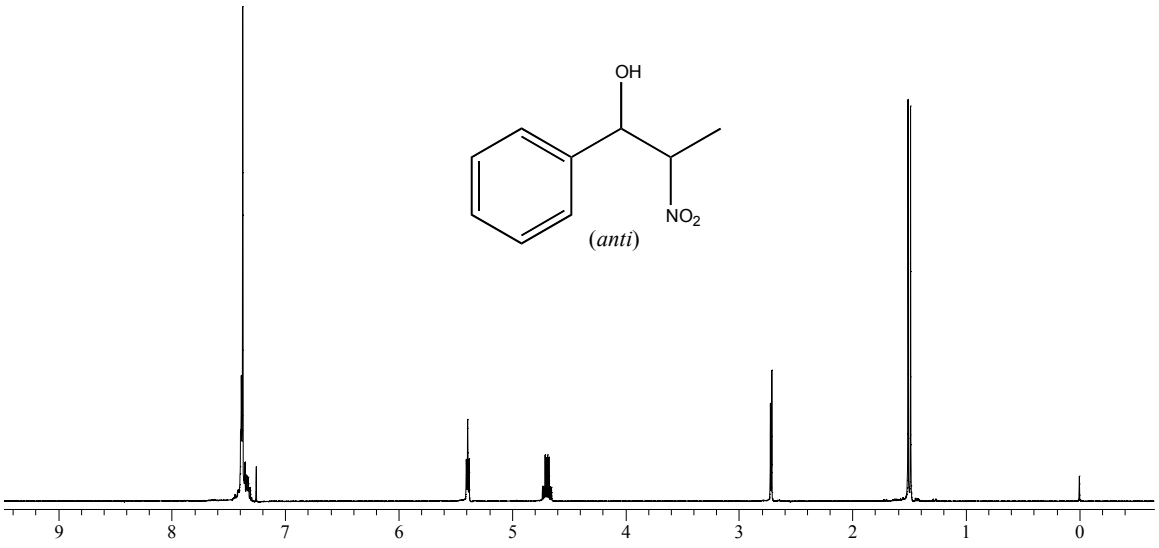


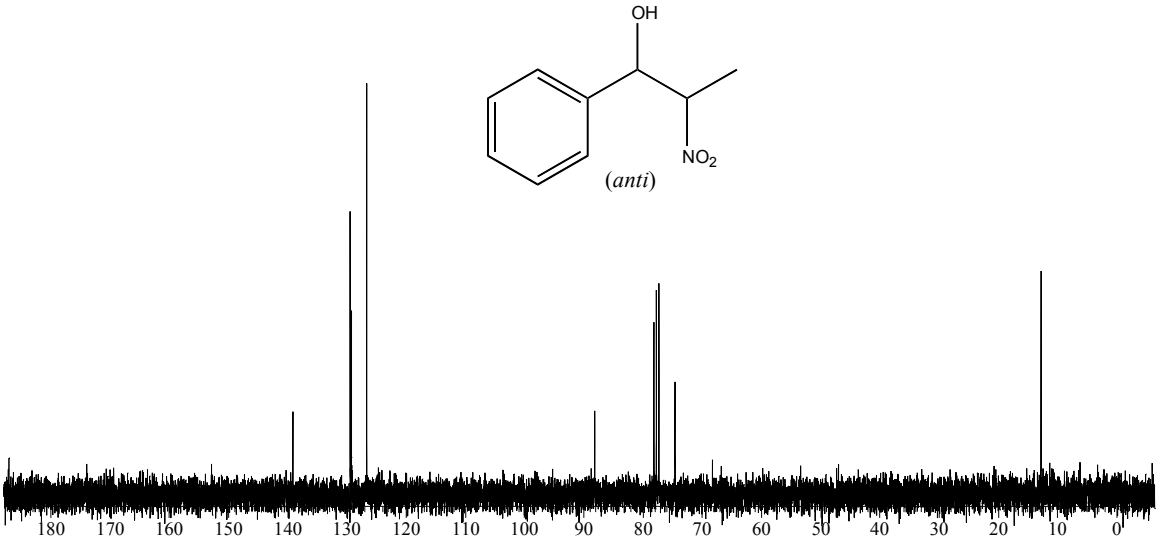
***Syn* 2-nitro-1-phenyl-1-propanol (*syn*-21)**





***Anti* 2-nitro-1-phenyl-1-propanol (*anti*-21)**





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