

Supporting Information

**Enantioselective, Organocatalytic Reduction of Ketones using
Bifunctional Thiourea-Amine Catalysts**

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I. General information

All reactions were maintained under an argon atmosphere. Anhydrous solvents were freshly distilled from sodium benzophenone ketyl, except for CH₂Cl₂, which was distilled from CaH₂. Extracts were dried over anhydrous Na₂SO₄ and then filtered prior to removal of all volatiles under reduced pressure. Unless otherwise noted, commercially available materials were used without further purification. Flash chromatography (FC) was performed using silica gel 60 (240–400 mesh). Thin layer chromatography was performed using commercial pre-coated glass plates (silica gel 60 PF254, 0.25 mm).

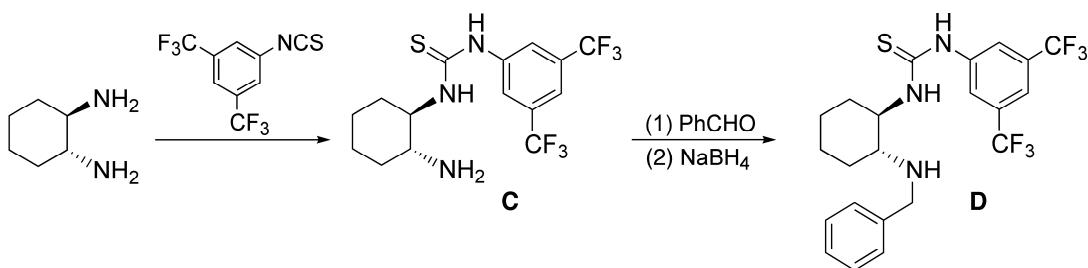
Nuclear magnetic resonance (NMR) spectra were recorded at operating frequencies of 300/400 MHz (¹H) or 75/100 MHz (¹³C). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform $\delta = 7.26$ for ¹H NMR or $\delta = 77.23$ for proton decoupled ¹³C NMR) and coupling constants (J) in Hz. Multiplicities are tabulated as s for singlet, d for doublet, t for triplet, q for quadruplet, and m for multiplet, whereby the prefix app is applied in cases where the true multiplicity is unresolved and br when the signal in question is broadened.

Ketone **29** was prepared by condensing (acetylmethylene)triphenylphosphorane with hydrocinnamaldehyde at 80 °C in toluene. Racemic alcohols were prepared by reduction of corresponding commercial ketones with NaBH₄ or NaBH₄/CeCl₃ (for enones)¹ and purified by flash column chromatography. The isothiocyanates used below were prepared according to Jacobsen.² Absolute configuration of **28**, **32**, **34**, and **36** assigned via comparison with authentic standards; the absolute configuration of **30** was made by analogy with the other examples generated using catalyst **D**.

(1) Luche, J.-P. *J. Am. Chem. Soc.* **1978**, *100*, 2226.

(2) Zuend, S. J.; Jacobsen, E. N. *J. Am. Chem. Soc.* **2007**, *129*, 15872–15883.

II. Preparation of catalysts

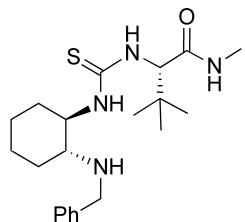


Catalysts C and D. 3,5-Bis(trifluoromethyl)phenyl isothiocyanate (1.3 mL, 7.1 mmol, 1.0 equiv) was added to a rt solution of (*R,R*)-cyclohexanediamine (970 mg, 8.51 mmol, 1.2 equiv) in anhydrous dichloromethane (20 mL). After 10 h, the reaction mixture was loaded onto a silica gel column and chromatographed using EtOAc/MeOH/NH₄OH (200:5:1→100:20:1) to give 1-((1*R,2R*)-2-aminocyclohexyl)-3-(3,5-bis(trifluoromethyl)phenyl)thiourea (catalyst **C**)³ (2.0 g, 73%) as a yellow foam. TLC: R_f ~ 0.31 (EtOAc/MeOH/NH₄OH, 100:5:1); [α]²⁰_D = +76.9 (c 1.7, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 8.01 (s, 2H), 7.55 (s, 1H), 6.62 (br s, 1H), 3.37 (br s, 1H), 2.69-2.65 (m, 1H), 2.04 (br s, 2H, NH₂), 1.98-1.91 (m, 2H), 1.80-1.65 (m, 2H), 1.40-1.20 (m, 4H); ¹³C NMR (CDCl₃, 75 MHz) δ 183.3, 142.0, 131.8 (q, J_{CF} = 33.9 Hz), 128.8, 125.2, 122.9, 121.5, 117.9, 117.7, 63.4, 56.8, 35.1, 32.3, 24.7; HRMS (FAB, NBA) Calcd. for C₁₅H₁₈N₃SF₆ [MH]⁺ *m/z* 386.1125, found 386.1128.

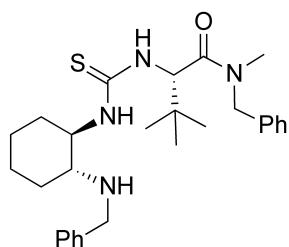
Benzaldehyde (0.578 mL, 5.45 mmol, 1.05 equiv) was added to a rt solution of catalyst **C** (2 g, 5.19 mmol, 1.0 equiv) in anhydrous MeOH (15 mL) under an argon atmosphere. After 4 h, the reaction mixture was cooled to 0 °C and NaBH₄ (211 mg, 5.71 mmol, 1.1 equiv) was added in portions. After another 20 min, saturated aq. NH₄Cl solution (50 mL) was added followed by conc. NH₄OH (2 mL). The resulting mixture was stirred for an additional 20 min, then extracted with dichloromethane (100 mL × 5). After drying over Na₂SO₄, the combined organic extracts were evaporated to dryness. The residue was purified by flash chromatography on silica gel using hexanes/EtOAc/MeOH/NH₄OH (400:100:5:1) to afford 1-((1*R,2R*)-2-(benzylamino)cyclohexyl)-3-(3,5-bis(trifluoromethyl)phenyl)thiourea (catalyst **D**)³ (2.02 g, 82%) as a pale yellow foam. The product was recrystallized from hexanes/CH₂Cl₂ as a white solid (75%), mp 140 °C-141 °C. TLC: R_f ~ 0.48 (CH₂Cl₂/MeOH, 9:1); ¹H NMR (CDCl₃, 300 MHz) δ

(3) Han, B.; Liu, Q.-P.; Li, R.; Tian, X.; Xiong, X.-F.; Deng, J.-G.; Chen, Y.-C. *Chem. Eur. J.* **2008**, *14*, 8094–8097.

7.69 (s, 2H), 7.48 (s, 1H), 7.29-7.20 (m, 5H), 6.59 (br s, 1H), 3.97 (d, $J = 12.3$ Hz, 1H), 3.82 (d, $J = 12.3$ Hz, 1H), 3.44 (br s, 1H), 2.49-2.42 (m, 1H), 2.18-2.00 (m, 2H), 1.90-1.75 (m, 2H), 1.40-1.20 (m, 4H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 183.3, 141.8, 138.0, 131.5 (q, $J_{\text{CF}} \sim 30$ Hz), 129.1, 128.6, 128.3, 128.1, 125.0, 122.4, 121.4, 117.1, 64.4, 62.5, 54.7, 33.2, 32.6, 24.9, 24.6; HRMS (FAB, NBA) Calcd. for $\text{C}_{22}\text{H}_{24}\text{N}_3\text{SF}_6$ $[\text{MH}]^+$ m/z 476.1595, found 476.1598.

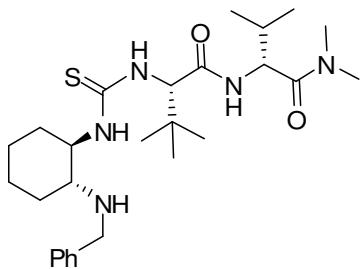


Catalyst G. Following the general procedure used to synthesize catalyst **D**, (*S*)-2-isothiocyanato-*N*,3,3-trimethylbutanamide was used to prepare catalyst **G** (54%). $[\alpha]^{20}_D = +54.9$ (c 1.30, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz) δ 7.99 (br s, 1H), 7.39-7.20 (m, 5H), 5.94 (br s, 2H), 4.77 (d, $J = 7.8$ Hz, 1H), 3.79 (br s, 2H), 3.45 (br s, 1H), 2.78 (d, $J = 4.8$ Hz, 3H), 2.40-2.35 (m, 1H), 2.10-1.95 (m, 1H), 1.95-1.80 (m, 2H), 1.75-1.65 (m, 2H), 1.27-1.12 (m, 4H), 1.00 (s, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 183.4, 171.5, 139.9, 128.7 (2C), 128.4 (2C), 127.3, 67.2, 61.1, 60.2, 51.7, 34.7, 32.8, 32.0, 27.0 (3C), 26.2, 24.8, 24.7; HRMS (ES) Calcd. for $\text{C}_{21}\text{H}_{35}\text{N}_4\text{OS}$ $[\text{MH}]^+$ m/z 391.2532, found 391.2529.



Catalyst H. Following the general procedure used to synthesize catalyst **D**, (*S*)-*N*-benzyl-2-isothiocyanato-*N*,3,3-trimethylbutanamide was used to prepare catalyst **H** (50%). $[\alpha]^{20}_D = +24.5$ (c 0.80, CHCl_3); ^1H NMR (CDCl_3 , 400 MHz) δ 7.92 (br s, 1H), 7.35-7.20 (m, 5H), 6.06 (d, $J = 1.2$ Hz, 1H), 5.59 (d, $J = 9.2$ Hz, 1H), 4.94 (d, $J = 14.4$ Hz, 1H), 4.15 (d, $J = 14.4$ Hz, 1H), 3.80 (d, $J = 13.6$ Hz, 1H), 3.76 (d, $J = 13.6$ Hz, 1H), 3.46 (br s, 2H), 3.15 (s, 3H), 2.34-2.28 (m, 1H), 2.10-1.95 (m, 1H), 1.95-1.80 (m, 2H), 1.70-1.65 (m, 2H), 1.30-1.10 (m, 4H), 1.00 (s, 9H); ^{13}C

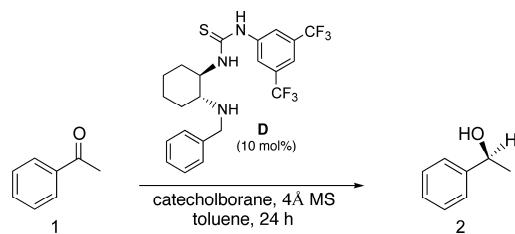
¹NMR (CDCl₃, 75 MHz) δ 183.1, 172.2, 140.1, 137.1, 128.7 (2C), 128.6 (2C), 128.4 (4C), 127.6, 127.2, 61.4, 61.1, 60.2, 51.7, 51.4, 36.3, 36.2, 32.7, 31.9, 26.9 (3C), 24.8, 24.6; HRMS (FAB, NBA) Calcd. for C₂₈H₄₁N₄OS [MH]⁺ *m/z* 481.3001, found 481.2998.



Catalyst I. Following the general procedure used to synthesize catalyst **D**, (*S*)-*N*-((*R*)-1-(dimethylamino)-3-methyl-1-oxobutan-2-yl)-2-isothiocyanato-3,3-dimethylbutanamide was used to prepare catalyst **I** (50%). $[\alpha]^{20}_D = +32.9$ (*c* 2.05, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.99 (br s, 1H), 7.29-7.18 (m, 5H), 6.75 (d, *J* = 7.8 Hz, 1H), 6.20 (d, *J* = 5.4 Hz, 1H), 4.78 (d, *J* = 5.1 Hz, 1H), 4.76 (d, *J* = 5.4 Hz, 1H), 3.77 (br s, 2H), 3.48 (br s, 1H), 3.03 (s, 3H), 2.91 (s, 3H), 2.40-2.35 (m, 1H), 2.10-1.80 (m, 3H), 1.75-1.60 (m, 2H), 1.27-1.08 (m, 4H), 1.01 (s, 9H), 0.94 (d, *J* = 7.2 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 183.4, 171.1, 170.5, 140.1, 128.5 (2C), 128.3 (2C), 127.1, 67.2, 61.1, 60.2, 53.8, 51.7, 37.4, 35.7, 34.8, 32.5, 31.9, 31.7, 27.2 (3C), 24.7, 24.6, 19.9, 17.7; HRMS (ES) Calcd. for C₂₇H₄₆N₅O₂S [MH]⁺ *m/z* 504.3372, found 504.3374.

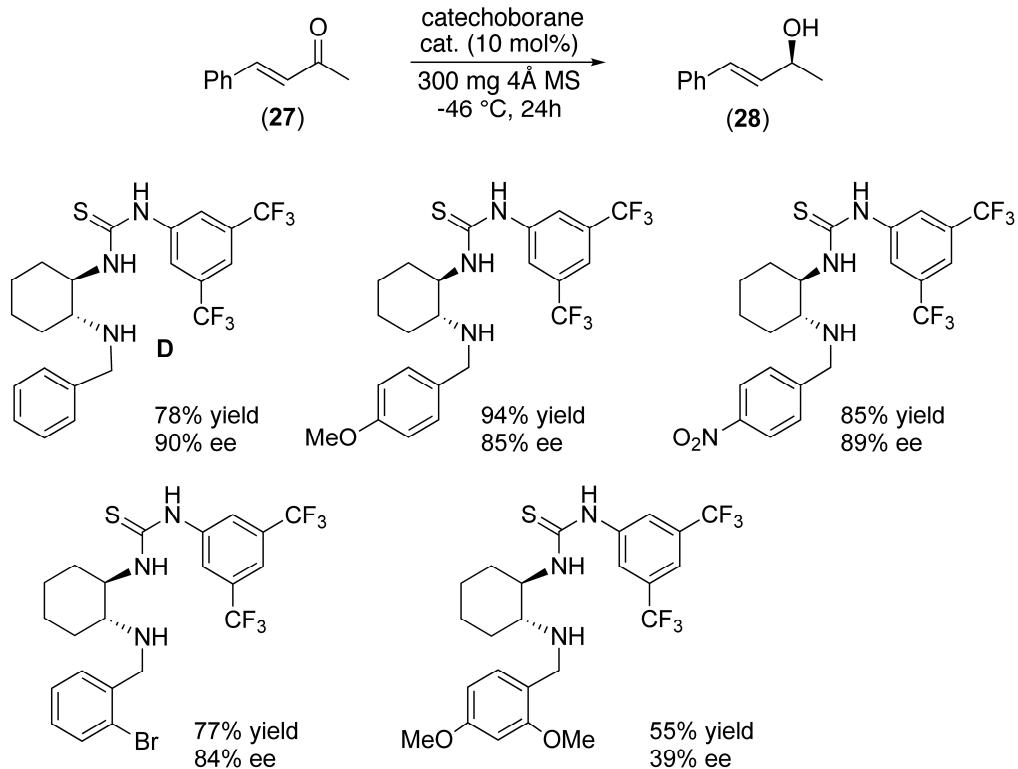
III. Reaction parameter optimization

a. Temperature dependence

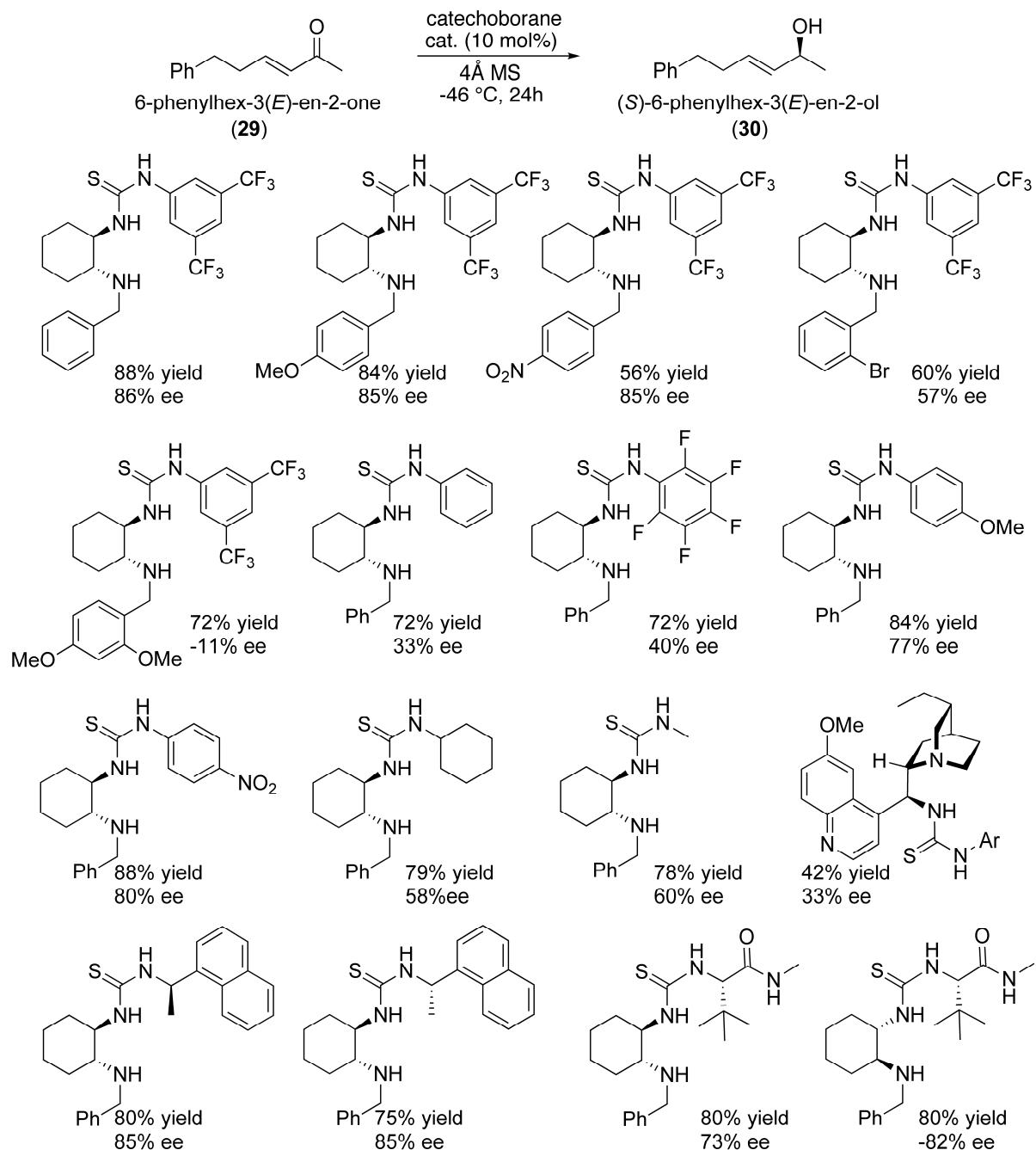


entry	temperature (°C)	yield (%)	ee (%) ^a
1	-78	24	73
2	-65	64	96
3	-59 ~ -62	80	97.7
4	-55	81	97.9
5	-46	88	98
6	-46 (in THF)	39	22
7	-46 (in CH ₂ Cl ₂)	49	49
8	-41 ~ -42	76	97.3
9	-30	88	85
10	-20	60	85

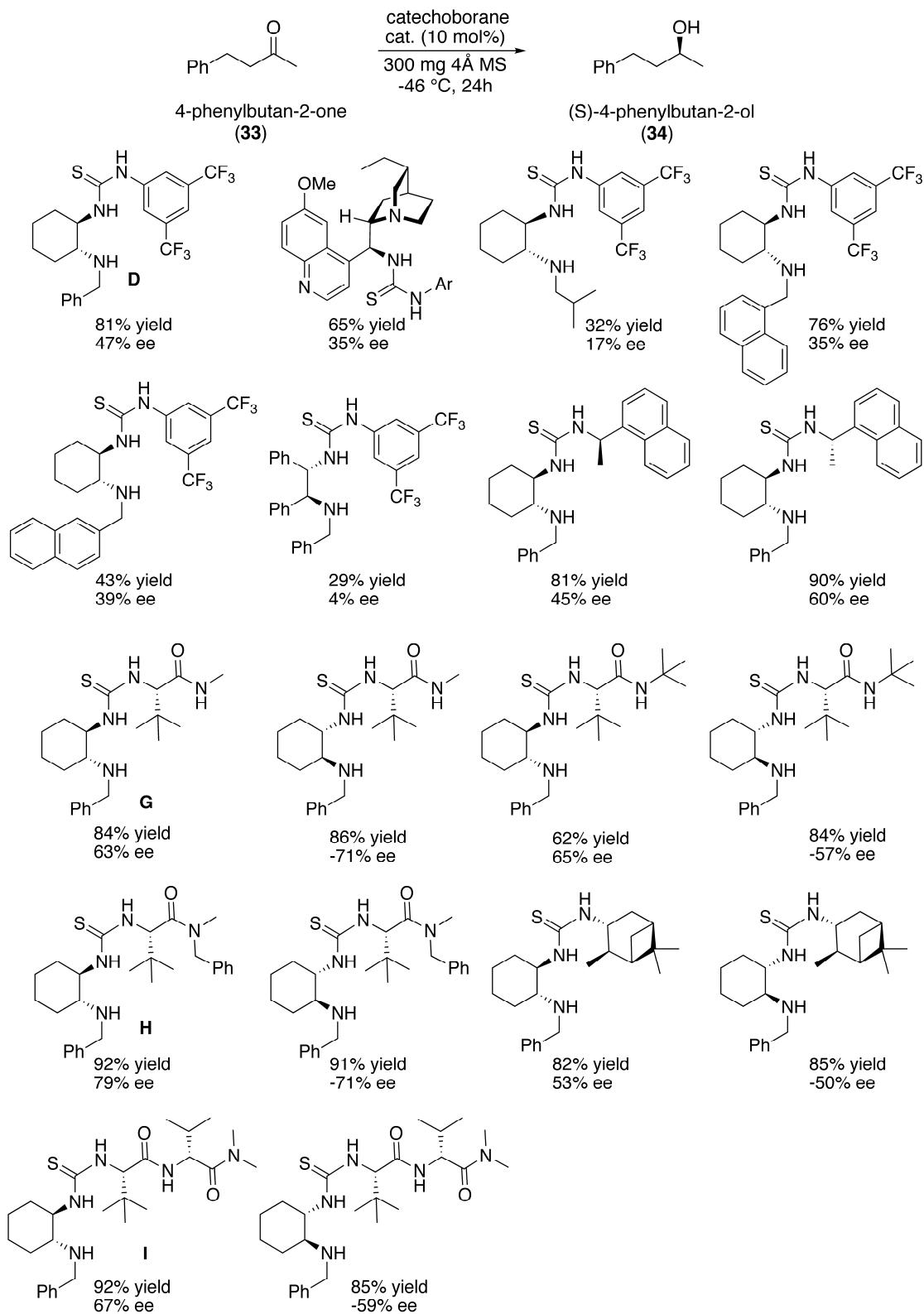
b. Catalyst survey of 4-phenylbut-3(*E*)-en-2-one

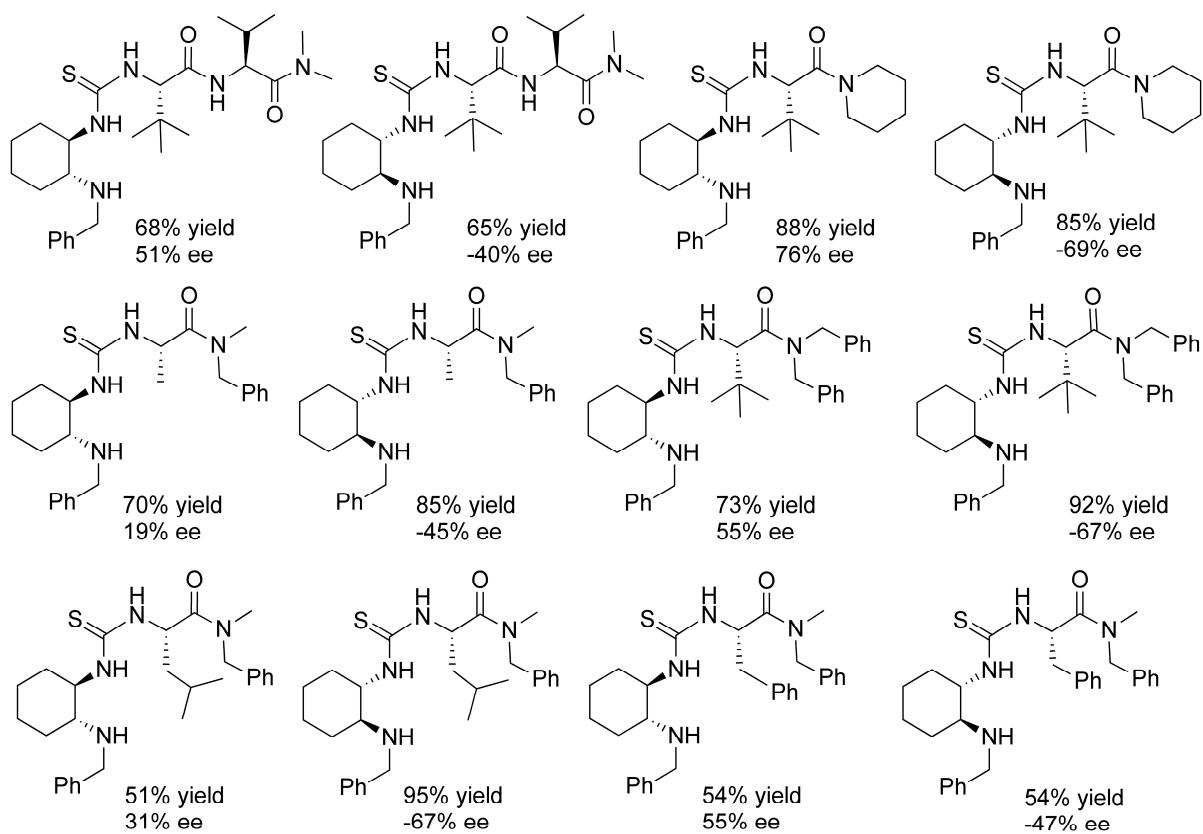


c. Catalyst survey for 6-phenylhex-3(*E*)-en-2-one (**29**)



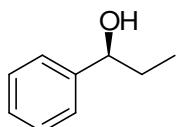
d. Catalyst survey for 4-phenylbutan-2-one (**33**)





IV. General reduction procedure and examples

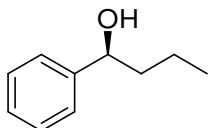
A mixture of catalyst **D** (12 mg, 0.025 mmol), 4Å molecular sieves (250 mg, freshly activated), and acetophenone (30 mg, 0.25 mmol) in toluene (0.7 mL) under an argon atmosphere was cooled to -78 °C. A solution of catecholborane (0.4 mL, 1.0 M in toluene, 0.4 mmol) was added slowly and the reaction mixture was placed in a -46 °C bath. After stirring for 24 h at -46 °C, MeOH (1 mL) followed by 3 N NaOH solution (1 mL) were added. The mixture was gradually warmed to room temperature and stirred for another 1 h and then extracted with Et₂O (20 mL × 3), dried over Na₂SO₄ and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel using hexanes/EtOAc (6:1) as eluent to give (*S*)-1-phenylethanol (**2**) as a colorless oil (27 mg, 88%). HPLC analysis: 98% ee using Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t(major) = 19.2 min, R_t(minor) = 15.0 min; major isomer $[\alpha]^{20}_D = -51.7$ (*c* 1.09, CHCl₃), lit.⁴ (*R*)-1-phenylethanol, 96% ee, $[\alpha]_D = +42.92$ (*c* 1.04, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.38-7.23 (m, 5H), 4.87 (q, *J* = 6.3 Hz, 1H), 2.03 (br s, 1H), 1.90 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 145.9, 128.6 (2C), 127.6, 125.5 (2C), 70.5, 25.3.



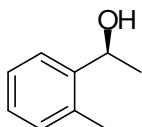
(S)-1-Phenylpropan-1-ol (4). 86% yield, 99% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t(major) = 17.3 min, R_t(minor) = 14.4 min; major isomer $[\alpha]^{20}_D = -47.4$ (*c* 1.48, CHCl₃), lit.⁵ (*S*)-1-phenylpropan-1-ol, 98% ee, $[\alpha]^{25}_D = -48.4$ (*c* 2.31, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.38-7.26 (m, 5H), 4.57 (t, *J* = 6.3 Hz, 1 H), 2.25 (br s, 1 H), 1.87-1.69 (m, 2H), 0.91 (t, *J* = 7.8 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 145.0, 128.5 (2C), 127.6, 126.1 (2C), 74.5, 41.3, 19.1, 14.1.

(4) Sokeirik, Y. S.; Mori, H.; Omote, M.; Sato, K.; Tarui, A.; Kumadaki, I.; Ando, A. *Org. Lett.* **2007**, 9, 1927–1929.

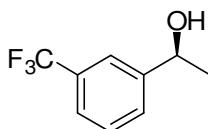
(5) Lutz, C.; Knochel, P. *J. Org. Chem.* **1997**, 62, 7895–7898.



(S)-1-Phenylbutan-1-ol (6). 81% yield, 99% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 23.8 min, R_t (minor) = 21.8 min; major isomer $[\alpha]^{20}_D = -47.6$ (*c* 0.50, CHCl₃), lit.⁶ (*R*)-1-phenylbutan-1-ol, 93% ee, $[\alpha]^{24}_D = +42$ (*c* 0.28, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.38-7.26 (m, 5H), 4.64 (t, *J* = 6.3 Hz, 1 H), 2.21 (br s, 1 H), 1.83-1.63 (m, 2H), 1.47-1.26 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 144.7, 128.5 (2C), 127.6, 126.1 (2C), 76.1, 31.9, 10.2.



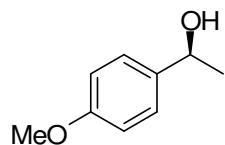
(S)-1-o-Tolylethanol (8). 71% yield, 95% ee. HPLC analysis: Chiraldak AD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 24.4 min, R_t (minor) = 21.8 min; major isomer $[\alpha]^{20}_D = -70.0$ (*c* 1.0, CHCl₃), lit.⁷ (*S*)-1-o-tolylethanol, 98% ee, $[\alpha]^{25}_D = -39.7$ (*c* 0.56, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.52-7.49 (m, 1H), 7.27-7.12 (m, 3 H), 5.10 (q, *J* = 6.3 Hz, 1H), 2.34 (s, 3H), 2.16 (br s, 1 H), 1.45 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 143.9, 134.2, 130.4, 127.2, 126.4, 124.5, 66.8, 24.0, 19.0.



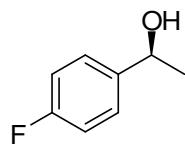
(S)-1-(3-(Trifluoromethyl)phenyl)ethanol (10). 92% yield, 96% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 14.0 min, R_t (minor) = 17.6 min; major isomer $[\alpha]^{20}_D = -31.0$ (*c* 1.95, CHCl₃), lit.⁸ (*S*)-1-(3-(trifluoromethyl)phenyl)ethanol, >99% ee, $[\alpha]^{20}_D = -27.9$ (*c* 1.64 in CH₃OH); ¹H NMR (CDCl₃,

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- (6) Node, M.; Nishide, K.; Shigeta, Y.; Shiraki, H.; Obata, K. *J. Am. Chem. Soc.* **2000**, *122*, 1927–1936.
 - (7) Evans, D. A.; Michael, F. E.; Tedrow, J. S.; Campos, K. R. *J. Am. Chem. Soc.* **2003**, *125*, 3534–3543.
 - (8) Tanaka, K.; Katsurada, M.; Ohno, F.; Shiga, Y.; Oda, M. *J. Org. Chem.* **2000**, *65*, 432.

300 MHz) δ 7.63 (d, J = 0.6 Hz, 1H), 7.54-7.42 (m, 3H), 4.93 (q, J = 6.3 Hz, 1H), 2.33 (br s, 1H), 1.48 (d, J = 6.3 Hz, 3H); ^{13}C NMR (CDCl₃, 75 MHz) δ 146.8, 131.0 (q, J_{CF} = 32.1 Hz), 129.1, 128.9, 124.4, 124.3, 122.4, 69.9, 25.4.

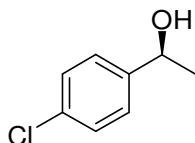


(S)-1-(4-Methoxyphenyl)ethanol (12). 80% yield, 97% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), 10% iPrOH/hexane, 0.5 mL/min, 254 nm, R_t(major) = 21.2 min, R_t(minor) = 18.6 min; major isomer $[\alpha]^{20}_{\text{D}} = -52.3$ (*c* 1.55, CHCl₃), lit.⁴ (*R*)-1-(4-methoxyphenyl)ethanol, 92% ee, $[\alpha]_{\text{D}} = +40.64$ (*c* 1.53, CHCl₃); ^1H NMR (CDCl₃, 300 MHz) δ 7.30-7.25 (m, 2H), 6.89-6.84 (m, 2H), 4.82 (q, J = 6.6 Hz, 1H), 3.79 (s, 3H), 2.22 (br s, 1H), 1.46 (d, J = 6.6 Hz, 3H); ^{13}C NMR (CDCl₃, 75 MHz) δ 159.0, 138.1, 126.8 (2C), 113.9 (2C), 70.0, 55.4, 25.1.

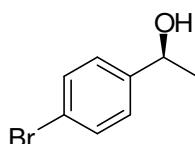


(S)-1-(4-Fluorophenyl)ethanol (14). 84% yield, 99% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), 1% iPrOH/hexane, 0.6 mL/min, 254 nm, R_t(major) = 36.4 min, R_t(minor) = 43.0 min; major isomer $[\alpha]^{20}_{\text{D}} = -44.8$ (*c* 1.40, CHCl₃), lit.⁹ (*S*)-1-(4-fluorophenyl)ethanol, 97% ee, $[\alpha]_{\text{D}} = -47.4$ (*c* 0.0576, CHCl₃); ^1H NMR (CDCl₃, 300 MHz) δ 7.34-7.26 (m, 2H), 7.05-7.698 (m, 2H), 4.84 (q, J = 6.6 Hz, 1H), 2.27 (br s, 1H), 1.45 (d, J = 6.3 Hz, 3H); ^{13}C NMR (CDCl₃, 75 MHz) δ 162.2 (d, J_{CF} = 243.8 Hz), 141.7 (d, J = 3.1 Hz), 127.2 (d, J = 7.9 Hz, 2C), 115.4 (d, J = 21.2 Hz, 2C), 69.8, 25.4.

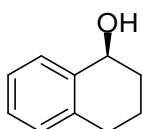
(9) Carter, M. B.; Schiøtt, B.; Gutiérrez, A.; Buchwald, S. L. *J. Am. Chem. Soc.* **1994**, *116*, 11667.



(S)-1-(4-Chlorophenyl)ethanol (16). 94% yield, 99% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm, R_t (major) = 22.1 min, R_t (minor) = 25.8 min; major isomer $[\alpha]^{20}_D = -44.2$ (*c* 1.80, CHCl₃), lit.¹⁰ (*S*)-1-(4-chlorophenyl)ethanol, 96% ee, $[\alpha]^{27}_D = -45.0$ (*c* 0.90, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.33-7.25 (m, 4H), 4.86 (q, *J* = 6.0 Hz, 1H), 2.00 (br s, 1H), 1.43 (d, *J* = 6.0 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 144.4, 133.2, 128.7 (2C), 126.9 (2C), 69.9, 25.4.



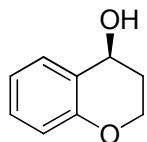
(S)-1-(4-Bromophenyl)ethanol (18). 95% yield, 99% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm; R_t (major) = 24.5 min, R_t (minor) = 28.9 min; major isomer $[\alpha]^{20}_D = -36.9$ (*c* 2.40, CHCl₃), lit.¹⁰ (*S*)-1-(4-bromophenyl)ethanol, 98% ee, $[\alpha]^{27}_D = -37.3$ (*c* 1.1, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.48-7.44 (m, 2H), 7.26-7.21 (m, 2H), 4.84 (q, *J* = 6.3 Hz, 1H), 2.02 (br s, 1H), 1.46 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 144.9, 131.7 (2C), 127.3 (2C), 121.3, 69.9, 25.4.



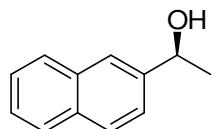
(S)-1,2,3,4-Tetrahydronaphthalen-1-ol (20). 86% yield, 99% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 16.2 min, R_t (minor) = 18.6 min; major isomer $[\alpha]^{20}_D = +38.9$ (*c* 1.45, CHCl₃), lit.⁷ (*S*)-1,2,3,4-tetrahydronaphthalen-1-ol, 91% ee, $[\alpha]^{25}_D = +31.2$ (*c* 0.54, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.44-7.41 (m, 1H), 7.23-7.19 (m, 2H), 7.12-7.10 (m, 1H), 4.77 (t, *J* = 4.5 Hz, 1H), 2.88-2.68

(10) Utsukihara, T.; Misumi, O.; Kato, N.; Kuroiwa, T.; Horiuchi C. A. *Tetrahedron: Asymmetry* **2006**, 17, 1179–1185.

(m, 2H), 2.05 (br s, 1H), 2.02-1.75 (m, 4H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 138.9, 137.2, 129.1, 128.8, 127.6, 126.3, 68.2, 32.3, 29.3, 18.9.

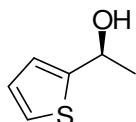


(S)-Chroman-4-ol (22). 95% yield, 98% ee. HPLC analysis: Chiralcel OJ-H column (250 mm \times 4.6 mm), 5% *iPrOH/hexane*, 1.0 mL/min, 254 nm, R_t (major) = 16.1 min, R_t (minor) = 21.2 min; major isomer $[\alpha]^{20}_{\text{D}} = -62.0$ (*c* 1.8, CHCl_3), lit.¹¹ (*R*)-chroman-4-ol, >99% ee, $[\alpha]^{20}_{\text{D}} = +65$ (*c* 1.0, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz) δ 7.27 (dd, $J = 7.5, 1.5$ Hz, 1H), 7.20 (dt, $J = 9.0, 1.5$ Hz, 1H), 6.90 (dt, $J = 6.6, 0.9$ Hz, 1H), 6.82 (d, $J = 8.1$ Hz, 1H), 4.70 (q, $J = 5.1$ Hz, 1H), 4.23 (dd, $J = 3.0, 1.2$ Hz, 1H), 4.21 (d, $J = 3.9$ Hz, 1H), 2.51 (d, $J = 4.8$ Hz, 1H), 2.12-1.91 (m, 2H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 154.6, 129.9, 129.8, 124.4, 120.6, 117.1, 63.2, 62.0, 30.9.

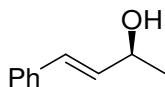


(S)-1-(Naphthalen-2-yl)ethanol (24). 93% yield, 98% ee. HPLC analysis: Chiralcel OJ-H column (250 mm \times 4.6 mm), 5% *iPrOH/hexane*, 1.0 mL/min, 254 nm, R_t (major) = 25.7 min, R_t (minor) = 33.7 min; major isomer $[\alpha]^{20}_{\text{D}} = -50.0$ (*c* 2.0, CHCl_3), lit.⁷ (*S*)-1-(naphthalen-2-yl)ethanol, 94% ee, $[\alpha]^{25}_{\text{D}} = -40.6$ (*c* 0.8, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz) δ 7.85-7.79 (m, 2H), 7.52-7.45 (m, 2H), 5.03 (q, $J = 6.6$ Hz, 1H), 2.33 (br s, 1H), 1.57 (d, $J = 6.3$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 143.3, 133.4, 133.0, 128.4, 128.0, 127.8, 126.2, 125.9, 124.0, 123.9, 70.6, 25.2.

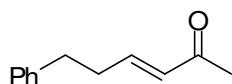
(11) Wettergren, J.; Boeveig, A.; Portier, M.; Adolfsson, H. *Adv. Synth. Catal.* **2006**, 348, 1277-1282.



(S)-1-(Thiophen-2-yl)ethanol (26). 66% yield, 97% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 30.0 min, R_t (minor) = 38.3 min; major isomer $[\alpha]^{20}_D = -24.6$ (*c* 0.90, CHCl₃), lit.¹² (*S*)-1-(thiophen-2-yl)ethanol, 99% ee, $[\alpha]^{24}_D = -26.0$ (*c* 1.02, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.26-7.21 (m, 1H), 6.98-6.94 (m, 2H), 5.09 (q, *J* = 6.6 Hz, 1H), 2.61 (br s, 1H), 1.57 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 150.0, 126.7, 124.5, 123.3, 66.2, 25.3.

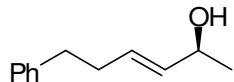


(S)-4-Phenylbut-3(*E*)-en-2-ol (28). 78% yield, 90% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 22.8 min, R_t (minor) = 16.0 min; major isomer $[\alpha]^{20}_D = -28.6$ (*c* 1.4, CHCl₃), lit.⁴ (*S*)-4-phenylbut-3(*E*)-en-2-ol, 92% ee, $[\alpha]^{25}_D = +16.4$ (*c* 0.9, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.40-7.22 (m, 5H), 6.57 (d, *J* = 15.9 Hz, 1H), 6.27 (ddd, *J* = 15.9, 6.3, 0.9 Hz, 1H), 4.87 (p, *J* = 6.3 Hz, 1H), 2.12 (br s, 1H), 1.37 (d, *J* = 6.3 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 136.8, 133.7, 129.5, 128.7 (2C), 127.7, 126.5 (2C), 69.0, 23.5.

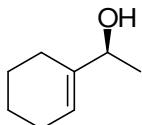


6-Phenylhex-3(*E*)-en-2-one (29). ¹H NMR (CDCl₃, 300 MHz) δ 7.32-7.17 (m, 5H), 6.82 (dt, *J* = 16.2, 6.6 Hz, 1H), 6.09 (d, *J* = 16.2 Hz, 1H), 2.79 (t, *J* = 8.4 Hz, 2H), 2.59-2.51 (m, 2H), 2.22 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 198.6, 147.1, 140.7, 131.7, 128.6 (2C), 128.4 (2C), 126.3, 34.4, 34.1, 26.9; ES-MS [MH]⁺ *m/z* 175.1.

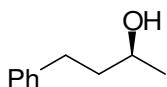
(12) Ohkuma, T.; Koizumi, M.; Yoshida, M.; Noyori, R. *Org. Lett.* **2000**, 2, 1749–1751.



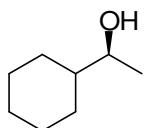
(S)-6-Phenylhex-3(E)-en-2-ol (30). 88% yield, 86% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 20.5 min, R_t (minor) = 13.0 min; major isomer $[\alpha]^{20}_D = -8.8$ (*c* 1.95, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.32-7.19 (m, 5H), 5.68 (dt, *J* = 15.6, 6.5 Hz, 1H), 5.53 (dd, *J* = 15.6, 6.3 Hz, 1H), 4.26 (p, *J* = 6.3 Hz, 1H), 2.71 (t, *J* = 7.5 Hz, 2H), 2.35 (q, *J* = 7.5 Hz, 2H), 1.68 (br s, 1H), 1.25 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 141.9, 135.0, 130.2, 128.6 (2C), 128.5 (2C), 126.0, 69.1, 35.8, 34.1, 23.5; ES-MS [MH]⁺ *m/z* 177.1.



(S)-1-Cyclohexenylethanol (32). 82% yield, 97% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), 0.5% *i*PrOH/hexane, 0.5 mL/min, 202 nm, R_t (major) = 12.7 min, R_t (minor) = 16.0 min; major isomer $[\alpha]^{20}_D = -9.5$ (*c* 1.2, CHCl₃), lit.⁷ (*S*)-1-cyclohexenylethanol, 91% ee, $[\alpha]^{25}_D = -9.4$ (*c* 1.5, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 5.67 (br s, 1H), 4.22-4.10 (m, 1H), 2.02-1.99 (m, 4H), 1.67-1.53 (m, 4H), 1.38 (d, *J* = 3.6 Hz, 1H), 1.25 (d, *J* = 6.6 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 141.4, 121.7, 72.4, 25.0, 23.8, 22.8, 22.7, 21.7.



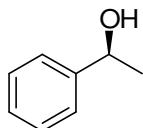
(S)-4-Phenylbutan-2-ol (34). 92% yield, 79% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 15.5 min, R_t (minor) = 11.1 min; major isomer $[\alpha]^{20}_D = +13.8$ (*c* 1.70, CHCl₃), lit.⁴ (*R*)-4-phenylbutan-2-ol, 88% ee, $[\alpha]^{22}_D = -17.32$ (*c* 1.6, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 7.34-7.18 (m, 5H), 3.89-3.80 (m, 1H), 2.83-2.64 (m, 2H), 1.89 (br s, 1H), 1.86-1.75 (m, 2H), 1.25 (d, *J* = 6.0 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 142.2, 128.5 (4C), 125.9, 67.5, 40.9, 32.2, 23.6.



(S)-1-Cyclohexylethanol (36). 68% yield, 91% ee. HPLC analysis of 4-nitrobenzoate: Chiralcel OJ-H column (250 mm × 4.6 mm), 0.1% *i*PrOH/hexane, 0.4 mL/min, 254 nm, R_t (major) = 31.9 min, R_t (minor) = 35.0 min; major isomer $[\alpha]^{20}_D$ = +3.71 (*c* 0.70, CHCl₃), lit. ¹³ (*R*)-1-cyclohexylethanol, 67% ee, $[\alpha]_D$ = -1.90 (*c* 0.75, CHCl₃); ¹H NMR (CDCl₃, 300 MHz) δ 3.52 (p, *J* = 6.3 Hz, 1H), 1.85-1.63 (m, 5H), 1.52 (br s, 1H), 1.35-0.85 (m, 6H), 1.13 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 72.3, 45.2, 28.8, 28.5, 26.6, 26.3, 26.2, 20.5.

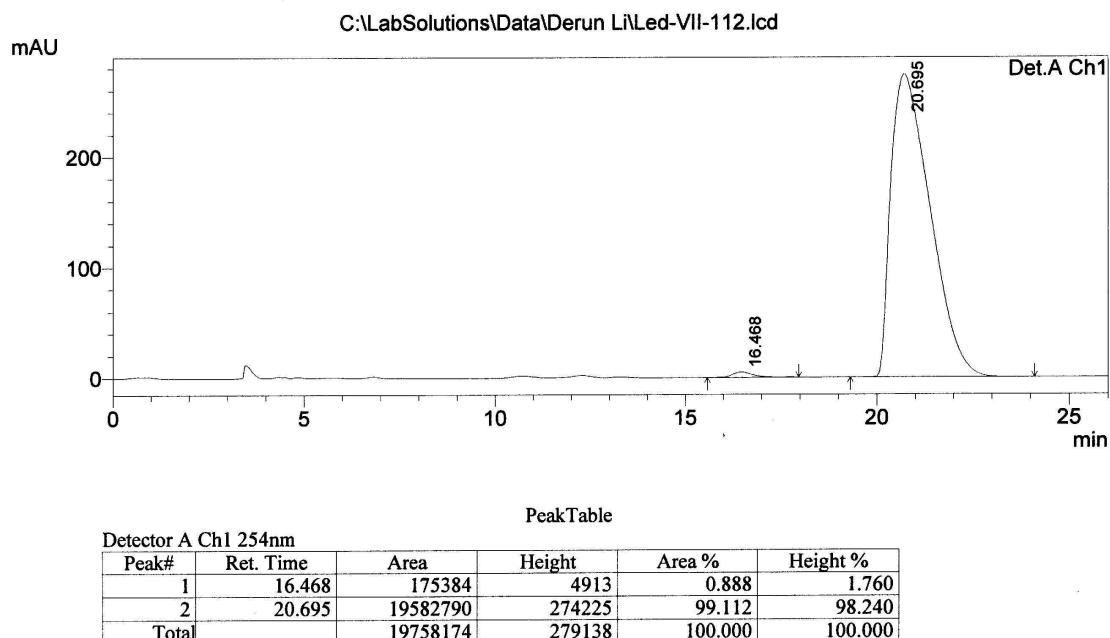
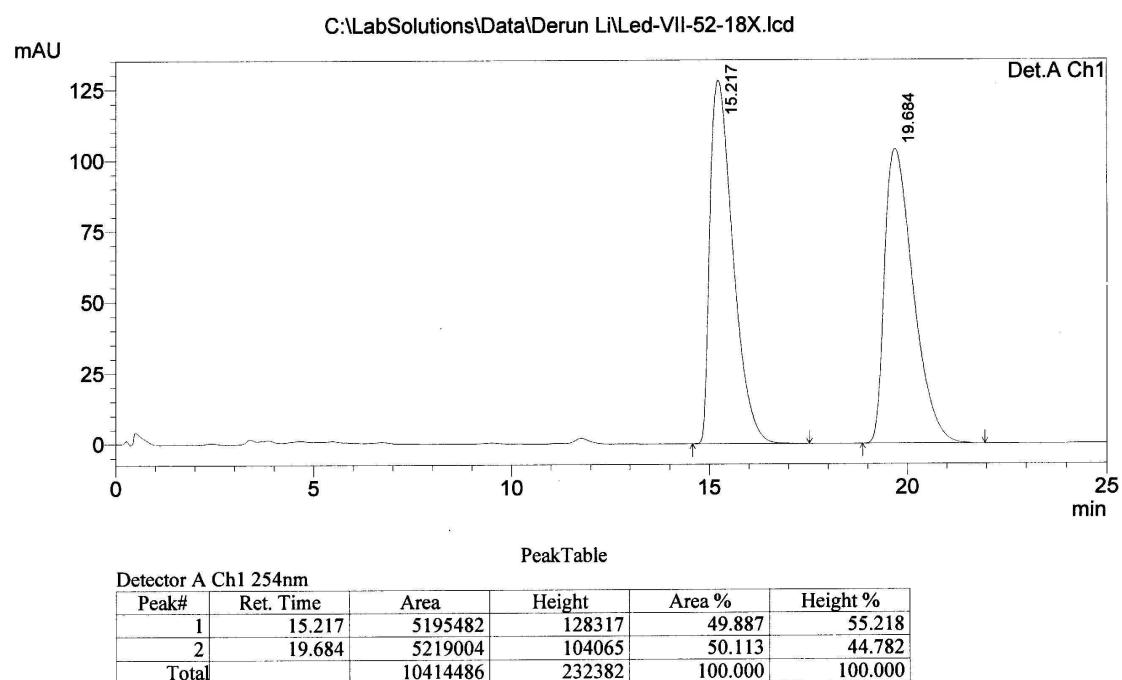
(13) Gamble, M. P.; Smith, A. R. C.; Wills, M. *J. Org. Chem.* **1998**, *63*, 6068–6071.

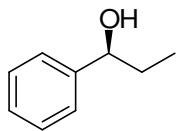
V. Chiral HPLC analyses



(S)-1-Phenylethanol (2)

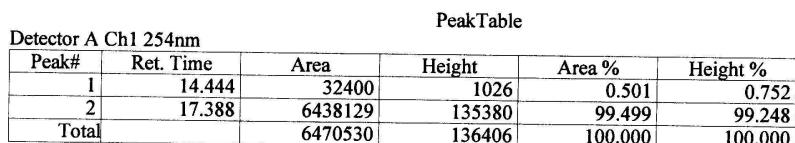
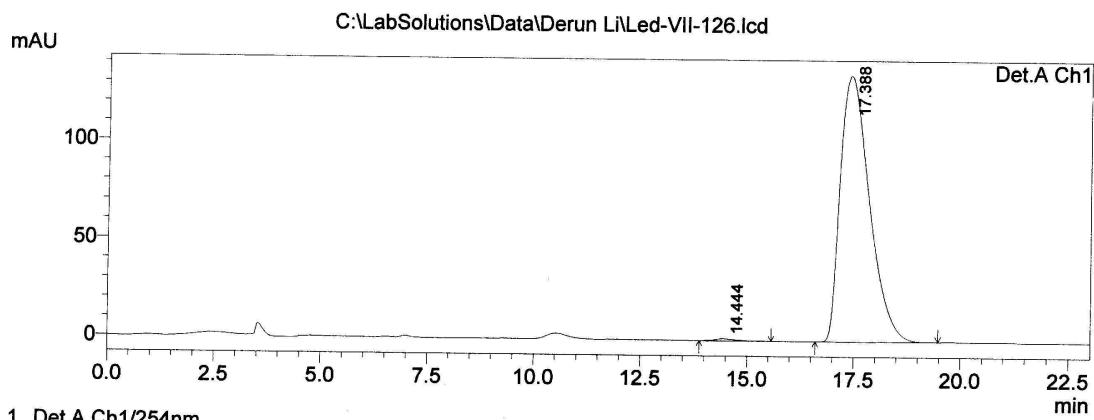
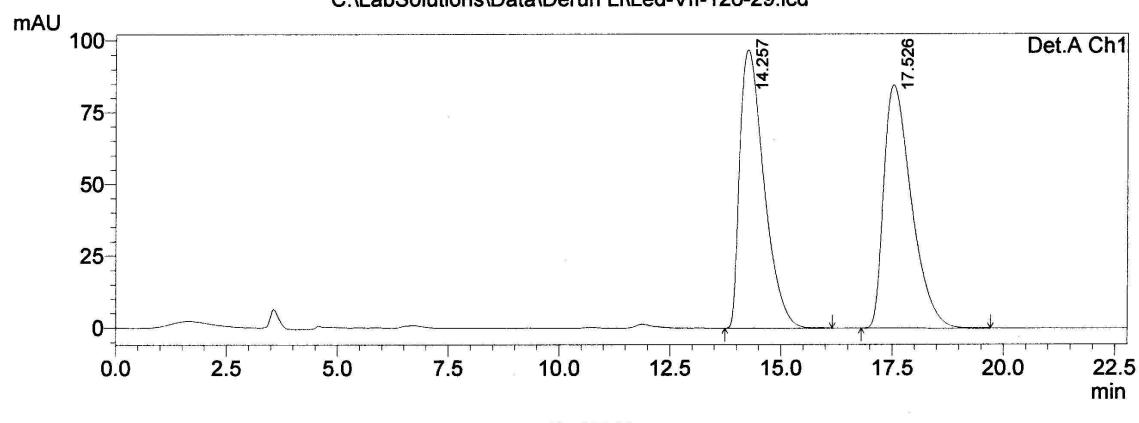
[Chiralcel OD column (250 mm × 4.6 mm), 2% iPrOH/hexane, 1.0 mL/min, 254 nm]

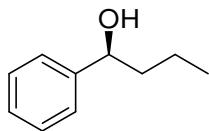




[Chiralcel OD column (250 mm × 4.6 mm), 2% iPrOH/hexane, 1.0 mL/min, 254 nm]

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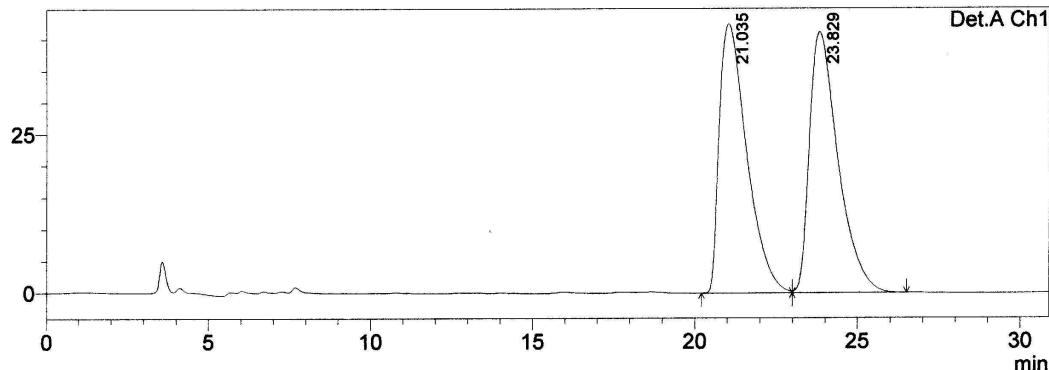


(S)-1-Phenylbutan-1-ol (6)

[Chiralcel OD column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 1.0 mL/min, 254 nm]

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mAU



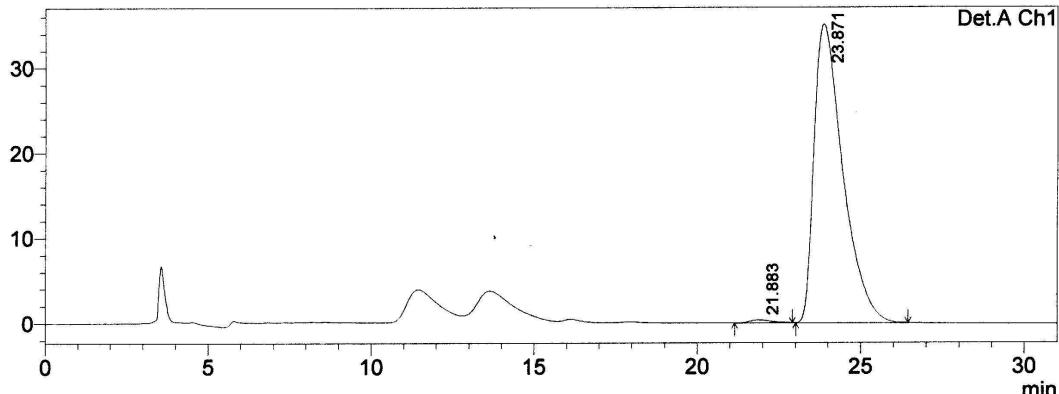
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.035	2502206	42447	49.845	50.752
2	23.829	2517729	41190	50.155	49.248
Total		5019935	83637	100.000	100.000

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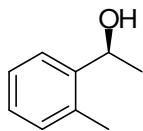
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PeakTable

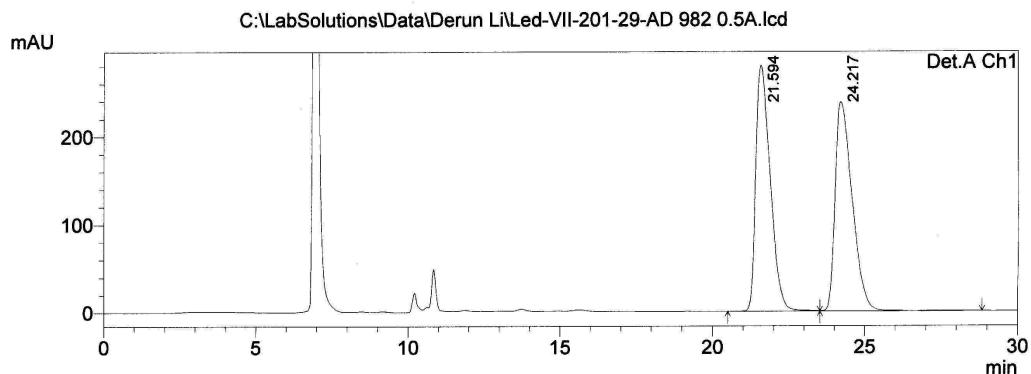
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.883	15556	358	0.719	1.011
2	23.871	2147085	35083	99.281	98.989
Total		2162641	35442	100.000	100.000



(S)-1-*o*-Tolylethanol (8)

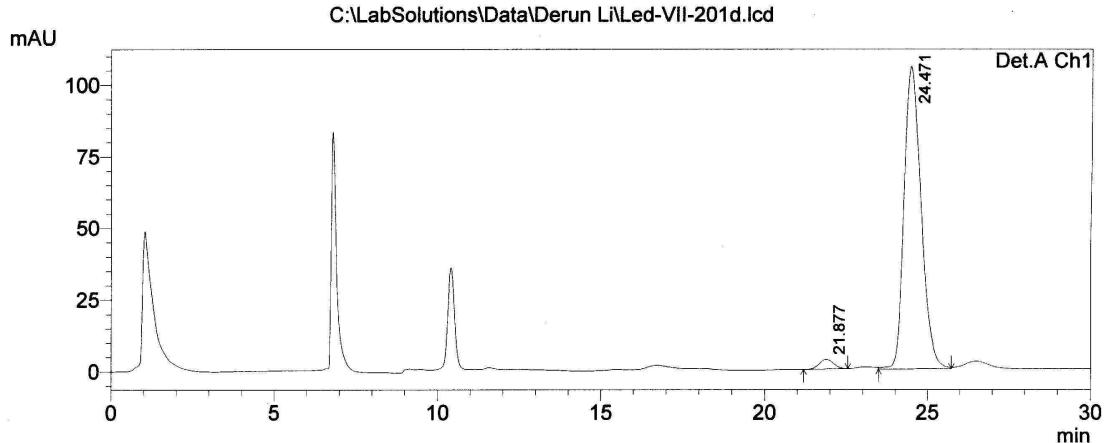
[Chiralpak AD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 245nm

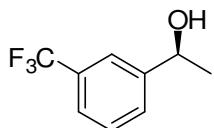
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.594	9263298	279274	49.986	54.041
2	24.217	9268335	237510	50.014	45.959
Total		18531633	516784	100.000	100.000



PeakTable

Detector A Ch1 245nm

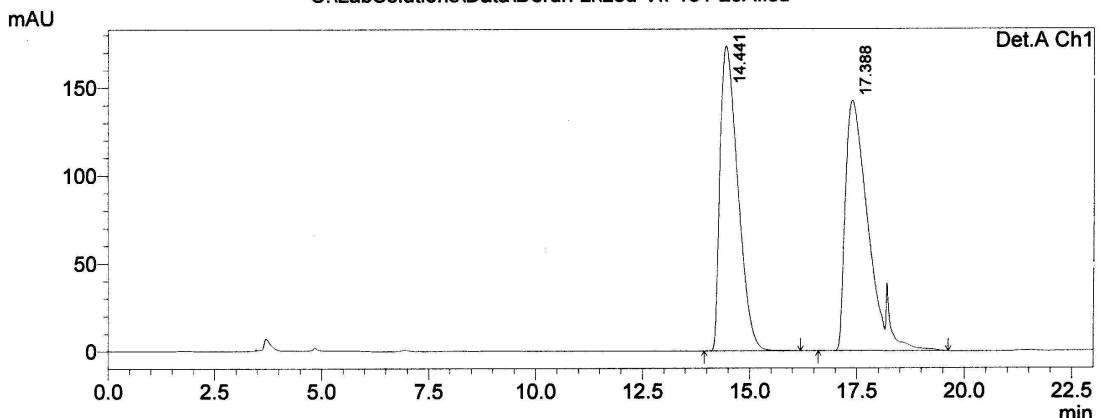
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.877	103001	3384	2.597	3.105
2	24.471	3863280	105604	97.403	96.895
Total		3966282	108988	100.000	100.000



(S)-1-(3-(Trifluoromethyl)phenyl)ethanol (10)

[Chiralcel OD column (250 mm × 4.6 mm), 2% iPrOH/hexane, 1.0 mL/min, 254 nm]

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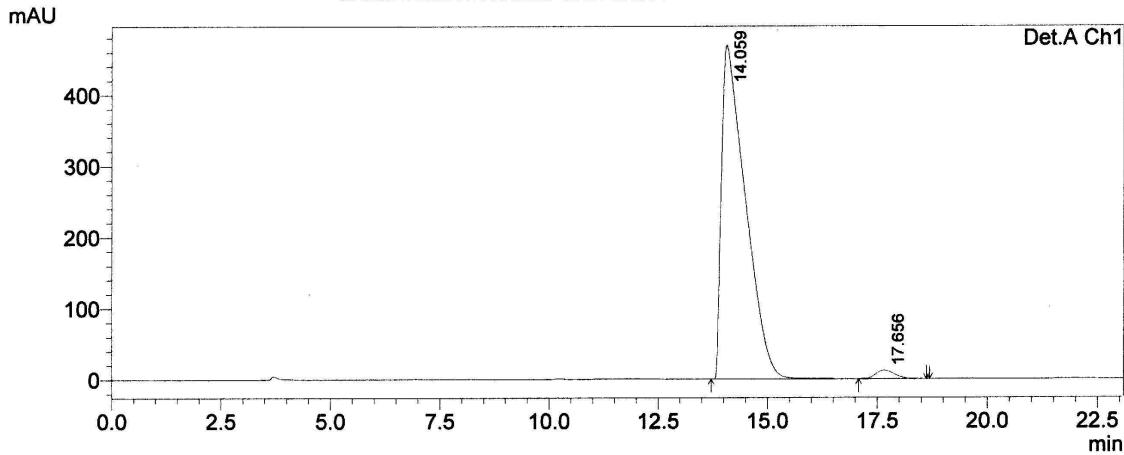
1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.441	5024407	173407	48.499	54.921
2	17.388	5335511	142332	51.501	45.079
Total		10359918	315739	100.000	100.000

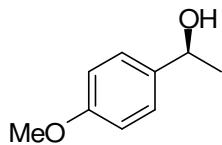
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PeakTable

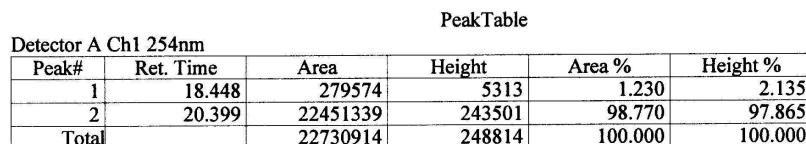
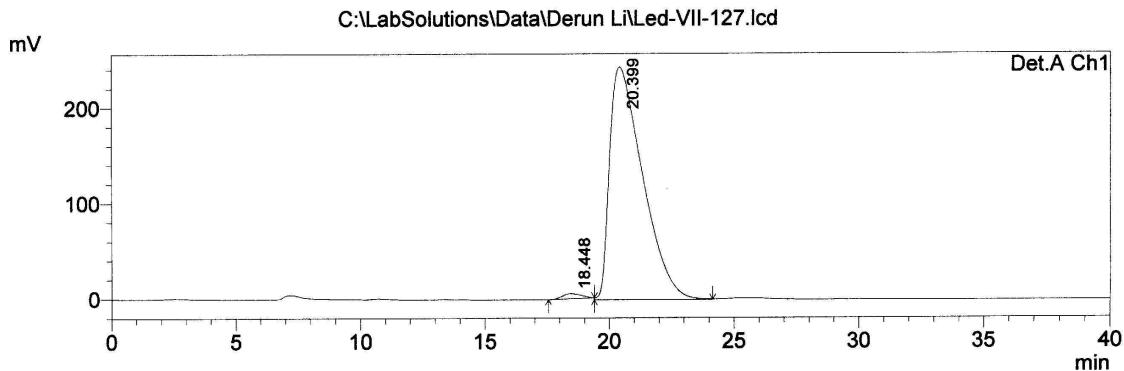
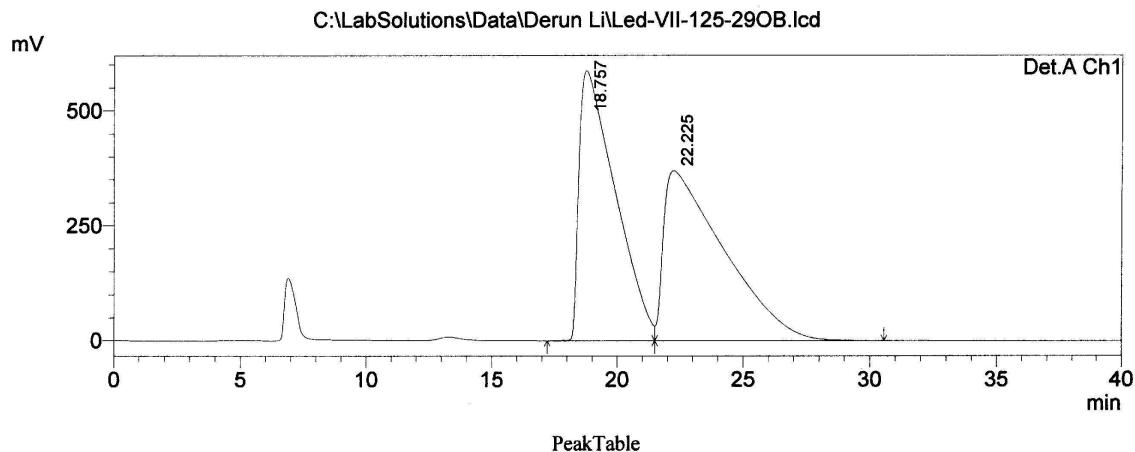
Detector A Ch1 254nm

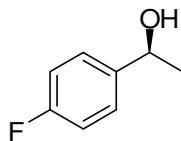
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.059	17841692	469701	98.027	97.510
2	17.656	359153	11992	1.973	2.490
Total		18200845	481694	100.000	100.000



(S)-1-(4-Methoxyphenyl)ethanol (12)

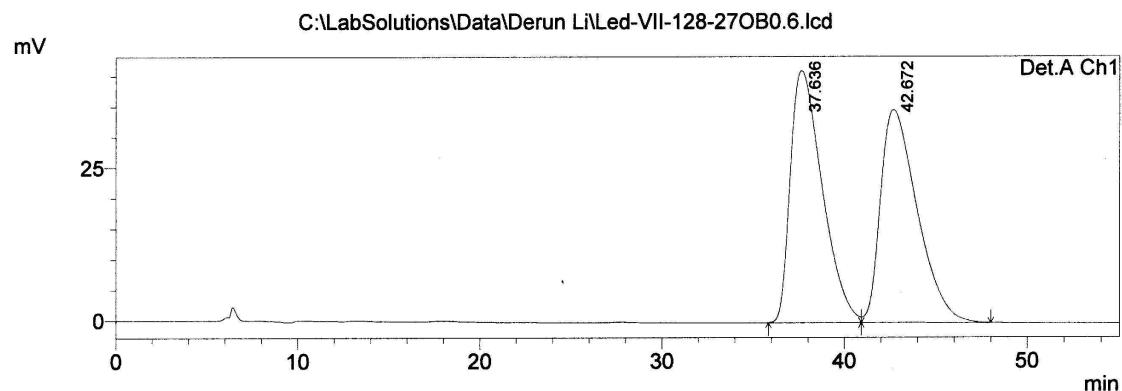
[Chiralcel OB column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm]





(S)-1-(4-Fluorophenyl)ethanol (14)

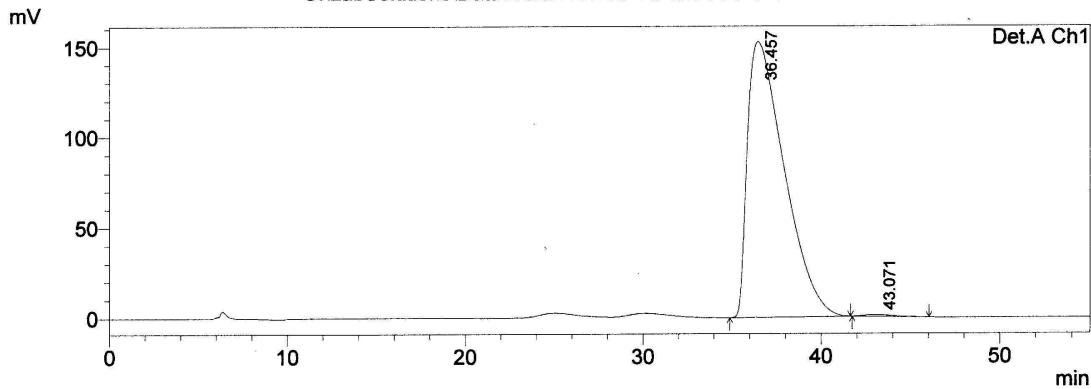
[Chiralcel OB column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 0.6 mL/min, 254 nm]



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.636	5012167	41185	50.687	54.167
2	42.672	4876271	34848	49.313	45.833
Total		9888439	76033	100.000	100.000

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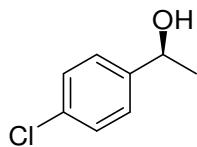


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

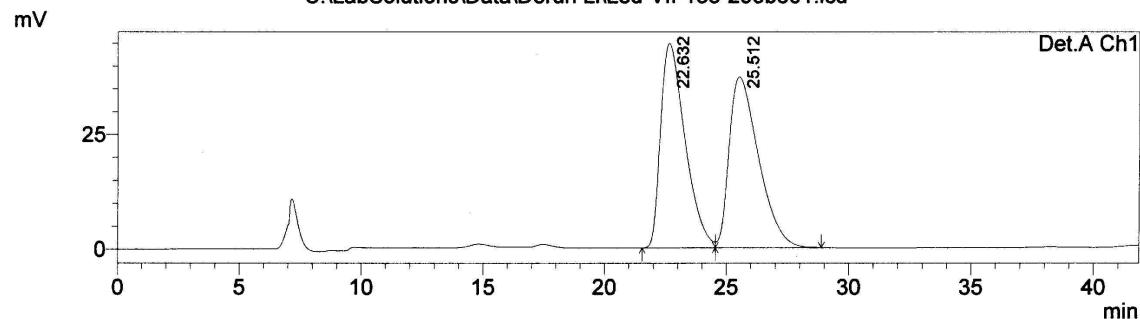
Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.457	21935304	152759	99.530	99.336
2	43.071	103686	1021	0.470	0.664
Total		22038989	153781	100.000	100.000



(S)-1-(4-Chlorophenyl)ethanol (16)

[Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-158-29ob601.lcd

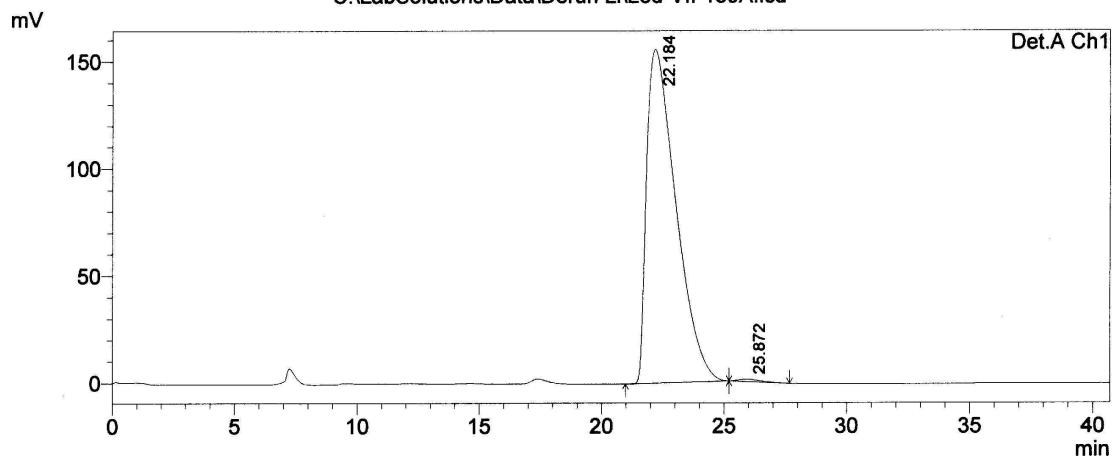


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.632	3112787	44596	49.903	54.482
2	25.512	3124831	37258	50.097	45.518
Total		6237618	81854	100.000	100.000

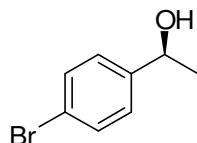
C:\LabSolutions\Data\Derun Li\Led-VII-159A.lcd



PeakTable

Detector A Ch1 254nm

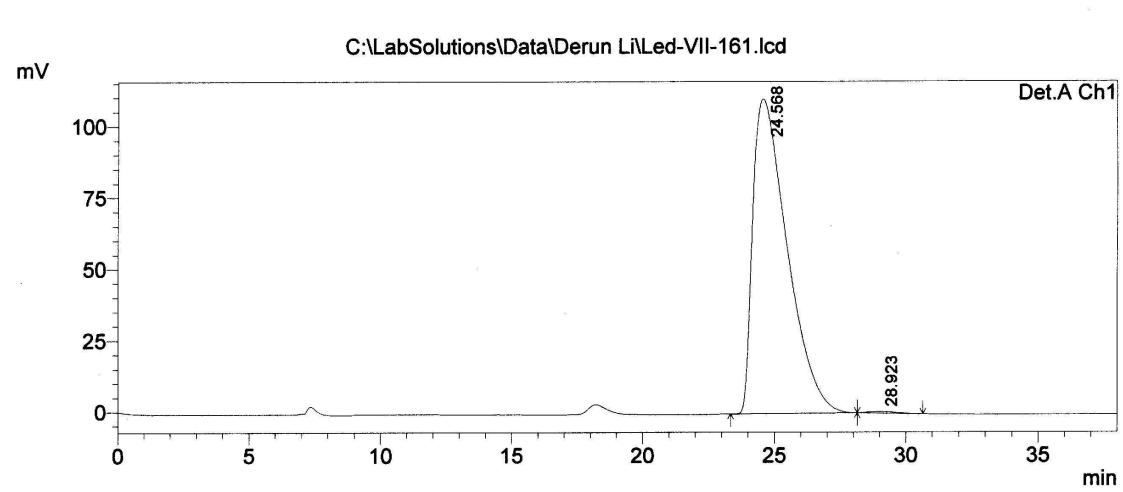
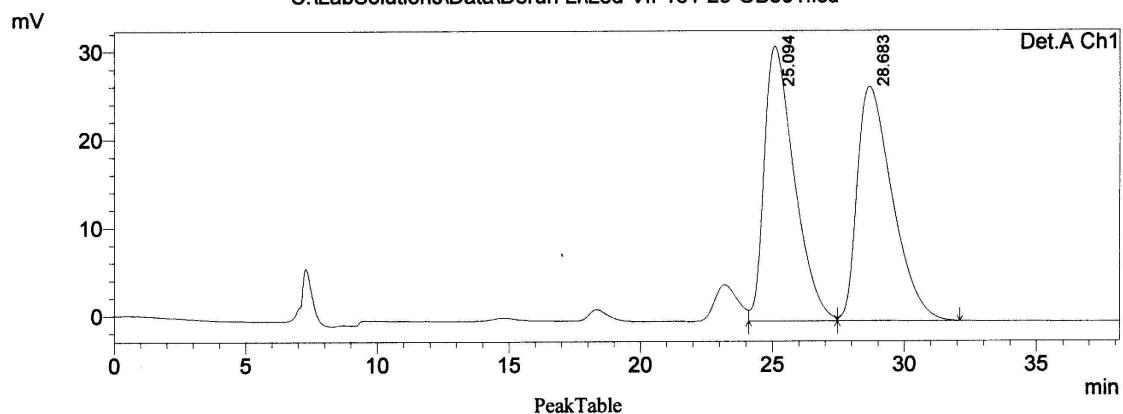
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.184	12950920	155583	99.494	99.328
2	25.872	65834	1052	0.506	0.672
Total		13016754	156636	100.000	100.000



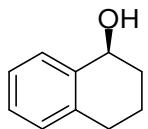
(S)-1-(4-Bromophenyl)ethanol (18)

[Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-161-29-OB601.lcd

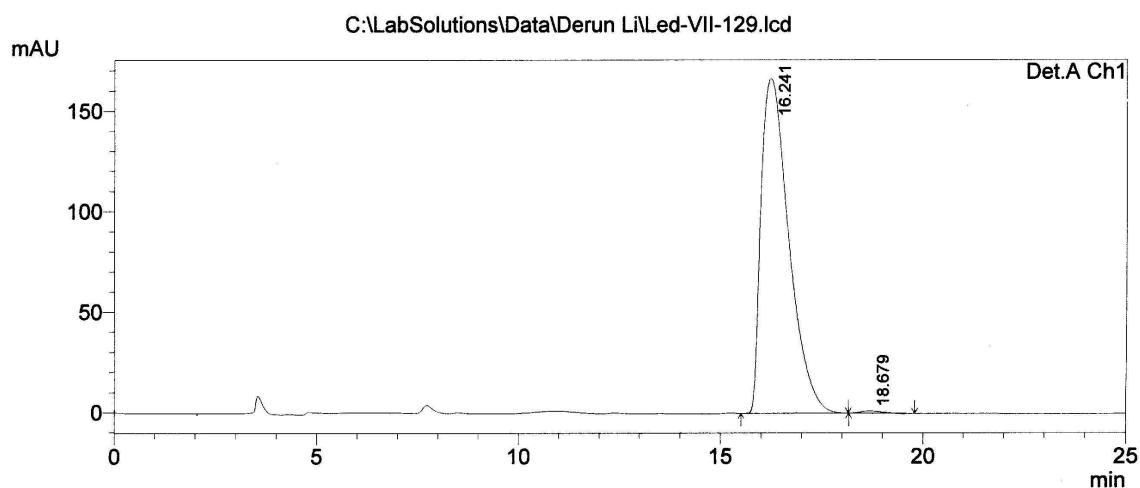
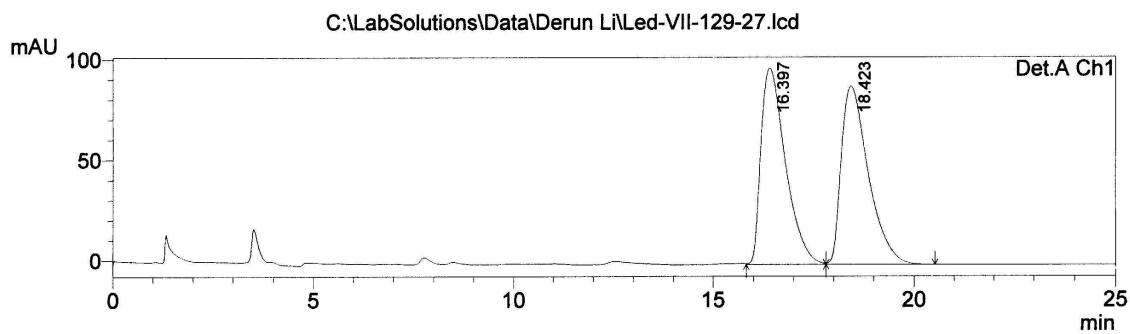


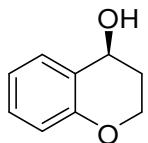
1 Det.A Ch1/254nm



(S)-1,2,3,4-Tetrahydronaphthalen-1-ol (20)

[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]

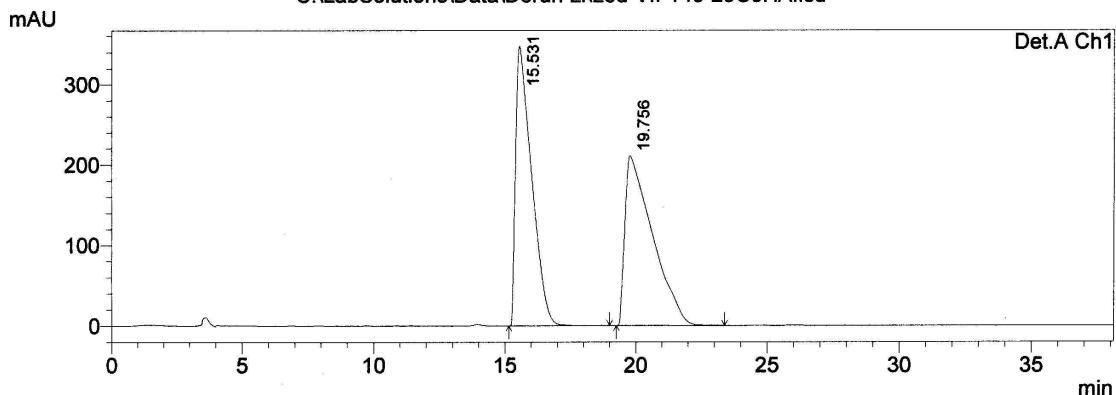




(S)-Chroman-4-ol (22)

[Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-140-29OJHA.lcd

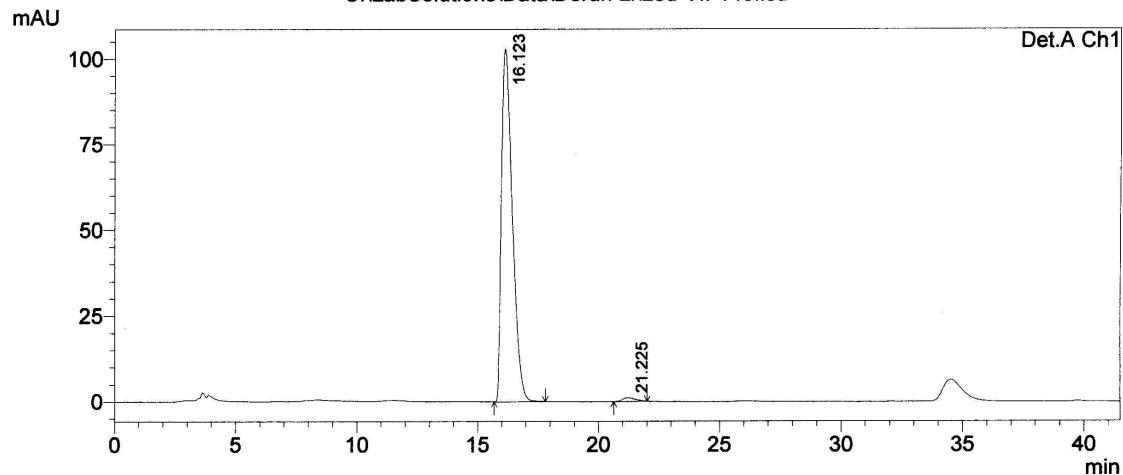


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.531	14775439	347688	48.862	62.245
2	19.756	15463959	210895	51.138	37.755
Total		30239399	558583	100.000	100.000

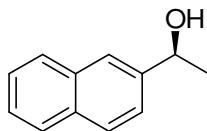
C:\LabSolutions\Data\Derun Li\Led-VII-140.lcd



PeakTable

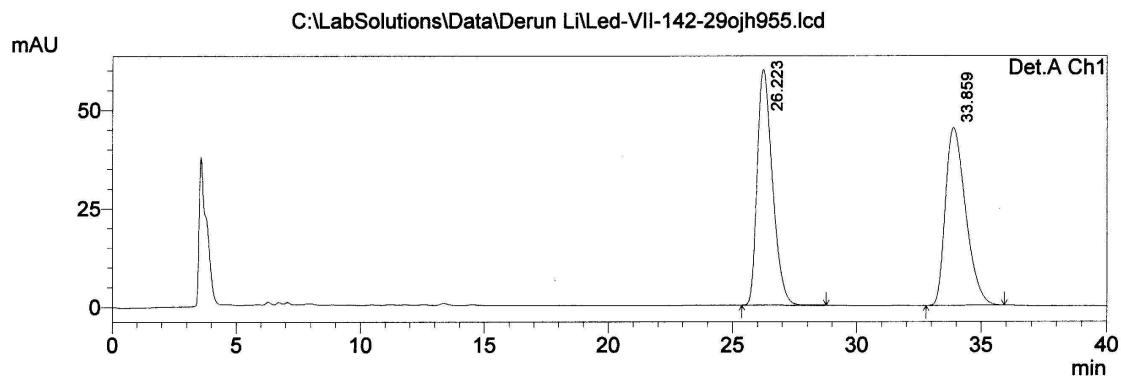
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.123	3230933	102963	98.773	98.918
2	21.225	40140	1126	1.227	1.082
Total		3271073	104090	100.000	100.000



(S)-1-(Naphthalen-2-yl)ethanol (24)

[Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]

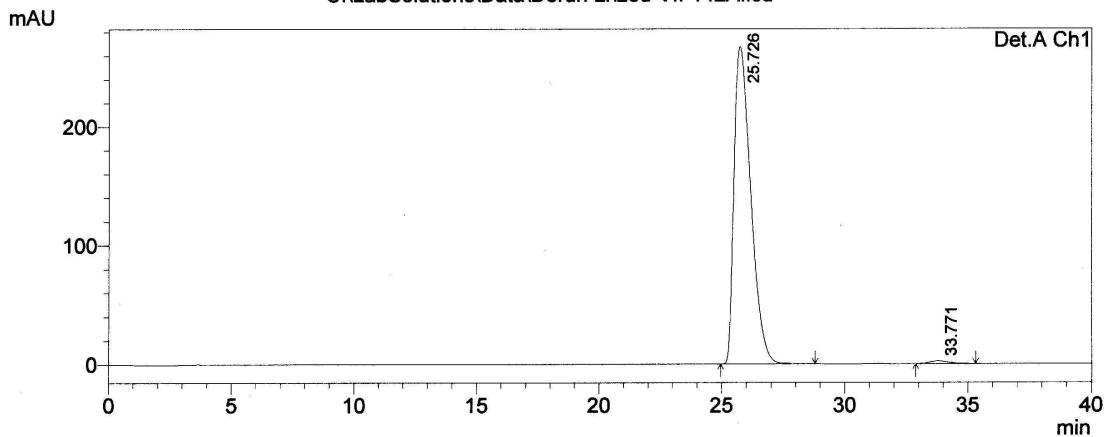


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.223	2552584	59862	49.960	57.015
2	33.859	2556668	45131	50.040	42.985
Total		5109252	104993	100.000	100.000

C:\LabSolutions\Data\Derun Li\Led-VII-142A.lcd

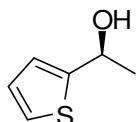


1 Det.A Ch1/254nm

PeakTable

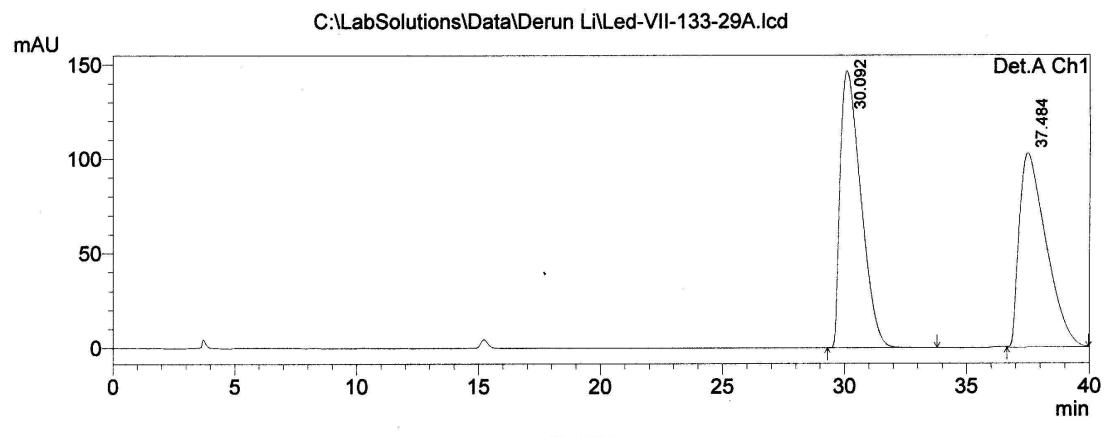
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.726	12702118	267391	99.085	99.166
2	33.771	117315	2250	0.915	0.834
Total		12819433	269641	100.000	100.000



(S)-1-(Thiophen-2-yl)ethanol (26)

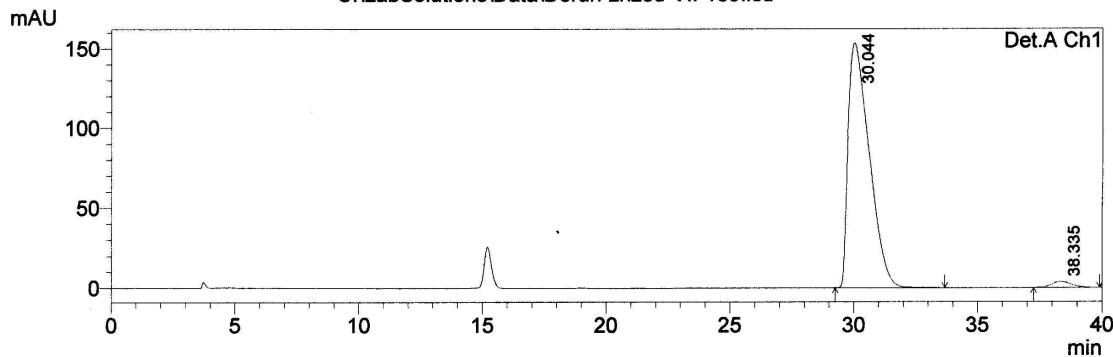
[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



Detector A Ch1 254nm

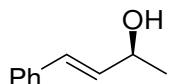
Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.092	8637146	146707	52.589	58.809
2	37.484	7786764	102755	47.411	41.191
Total		16423910	249462	100.000	100.000

C:\LabSolutions\Data\Derun Li\Led-VII-139.lcd



Detector A Ch1 254nm

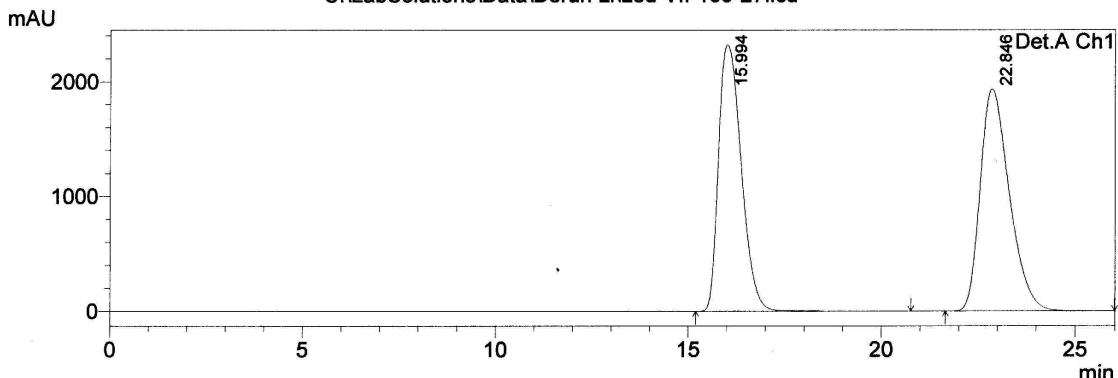
Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.044	9106451	153540	97.761	97.574
2	38.335	208539	3818	2.239	2.426
Total		9314989	157357	100.000	100.000



(S)-4-Phenylbut-3(E)-en-2-ol (28)

[Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-109-27.lcd

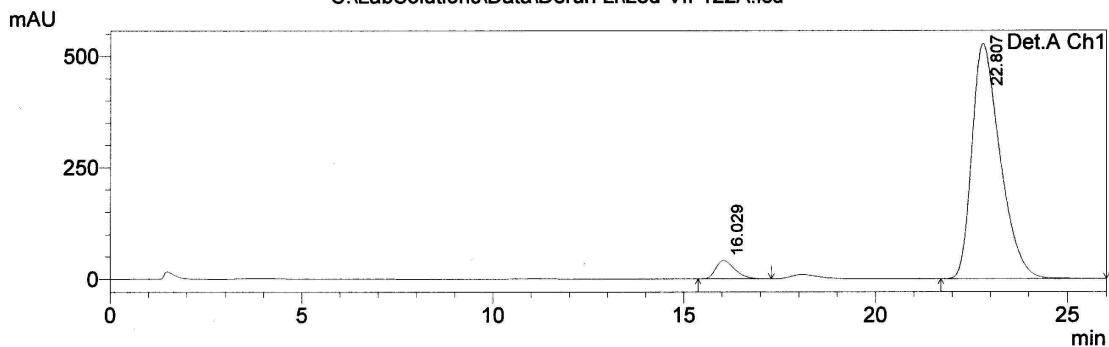


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.994	95021379	2317777	48.402	54.523
2	22.846	101294977	1933241	51.598	45.477
Total		196316355	4251018	100.000	100.000

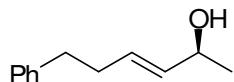
C:\LabSolutions\Data\Derun Li\Led-VII-122A.lcd



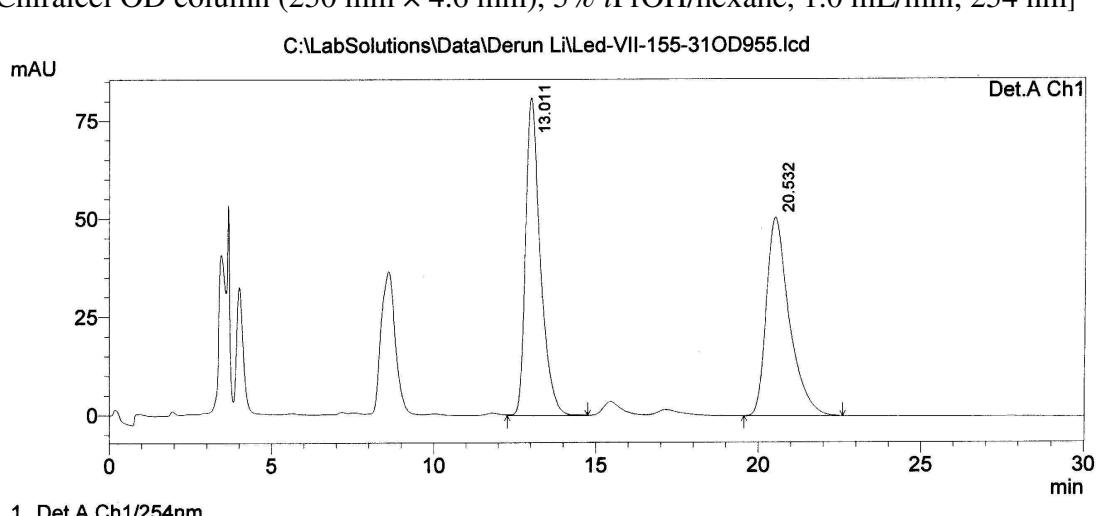
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.029	1430847	41278	5.054	7.250
2	22.807	26881031	528072	94.946	92.750
Total		28311878	569350	100.000	100.000

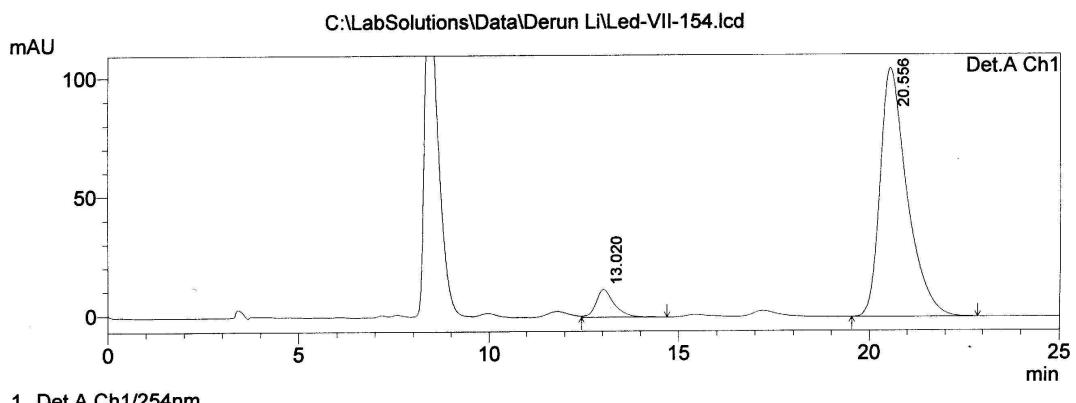


[Chiralcel OD column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



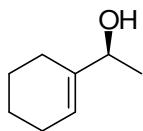
PeakTable

Detector A Ch1 254nm					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.011	2559620	80983	50.768	61.555
2	20.532	2482162	50580	49.232	38.445
Total		5041781	131563	100.000	100.000



PeakTable

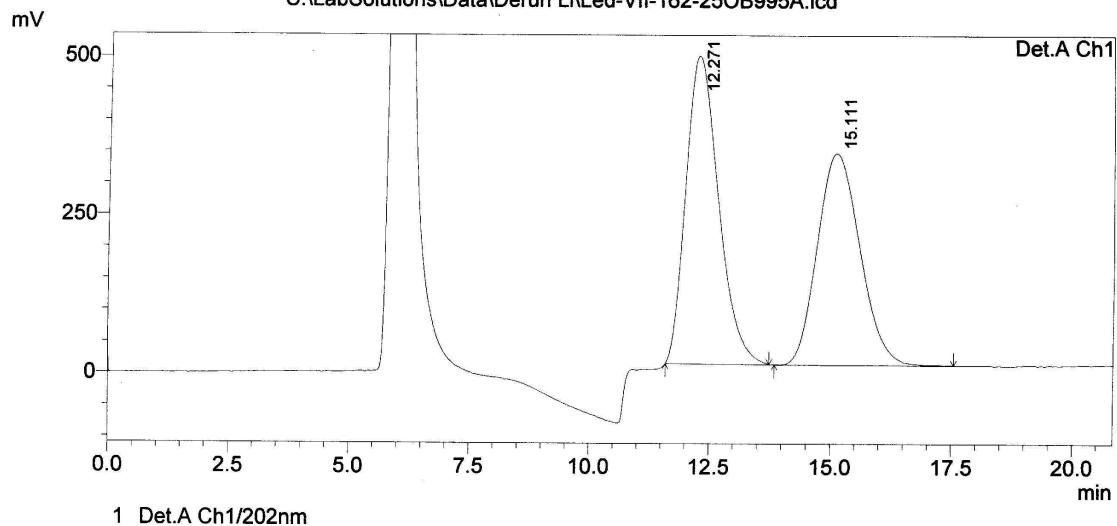
Detector A Ch1 254nm					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.020	382399	11539	6.915	9.937
2	20.556	5147705	104585	93.085	90.063
Total		5530104	116125	100.000	100.000



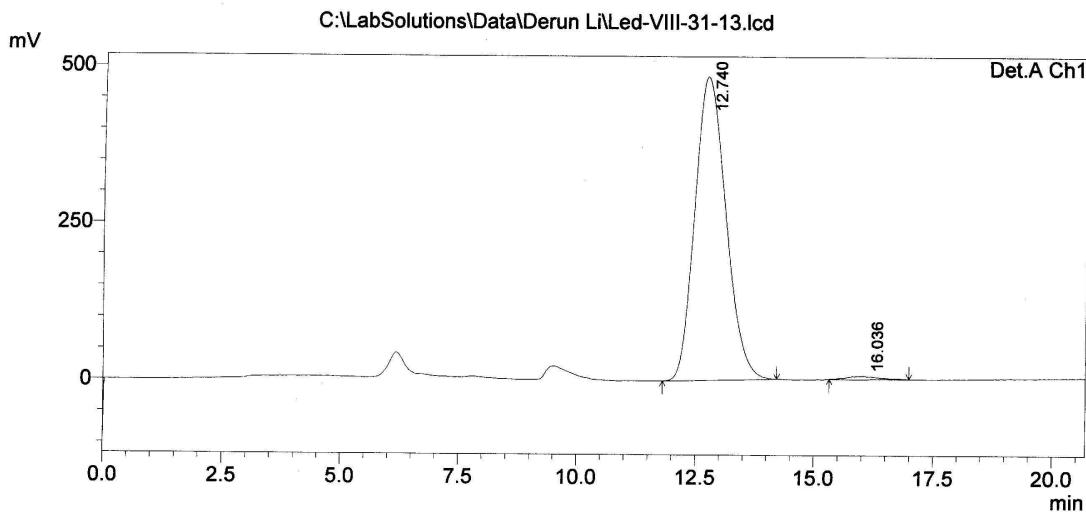
(S)-1-Cyclohexenylethanol (32)

[Chiralcel OB column (250 mm × 4.6 mm), 0.5% *i*PrOH/hexane, 0.5 mL/min, 202 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-162-25OB995A.lcd

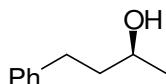


Peak#	Ret. Time	Area	Height	Area %
1	12.271	23025141	486438	52.962
2	15.111	20449762	334456	47.038
Total		43474904	820894	100.000



PeakTable

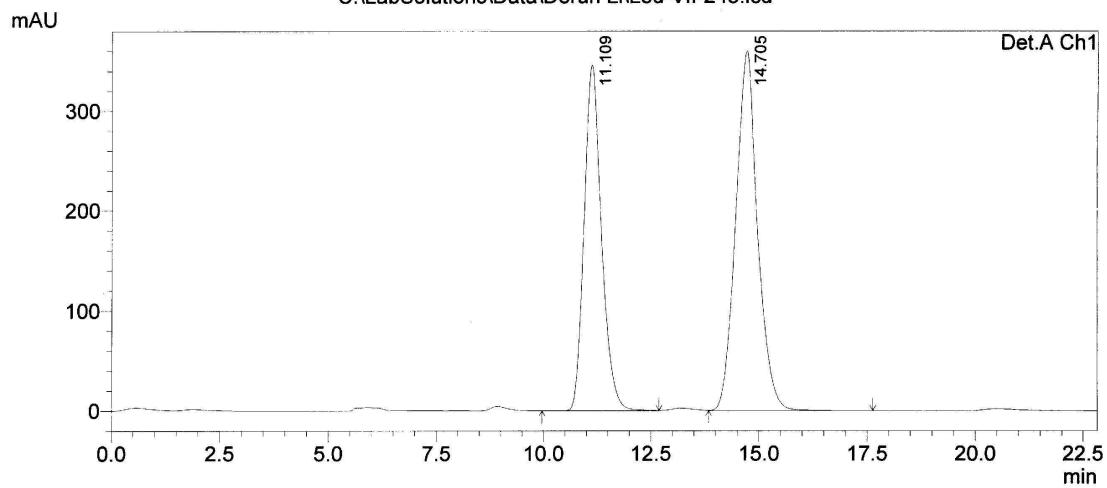
Peak#	Ret. Time	Area	Height	Area %
1	12.740	22007040	483626	98.860
2	16.036	253845	5611	1.140
Total		22260885	489237	100.000



(S)-4-Phenylbutan-2-ol (34)

[Chiralcel OD column (250 mm × 4.6 mm), 10% iPrOH/hexane, 0.5 mL/min, 254 nm]

C:\LabSolutions\Data\Derun Li\Led-VII-245.lcd

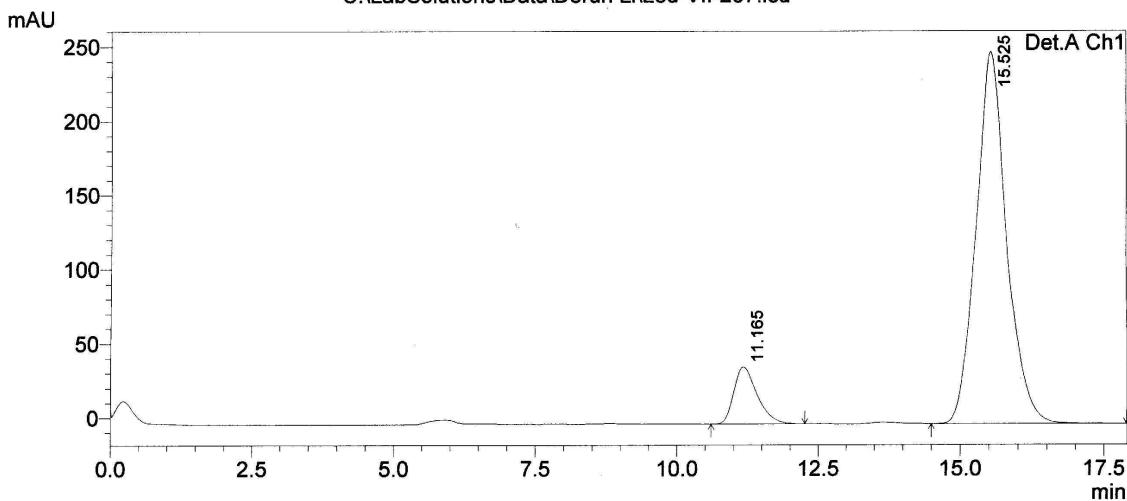


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.109	9759084	346012	43.554	49.022
2	14.705	12647542	359811	56.446	50.978
Total		22406626	705822	100.000	100.000

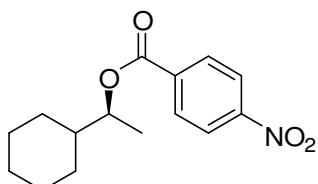
C:\LabSolutions\Data\Derun Li\Led-VII-267.lcd



PeakTable

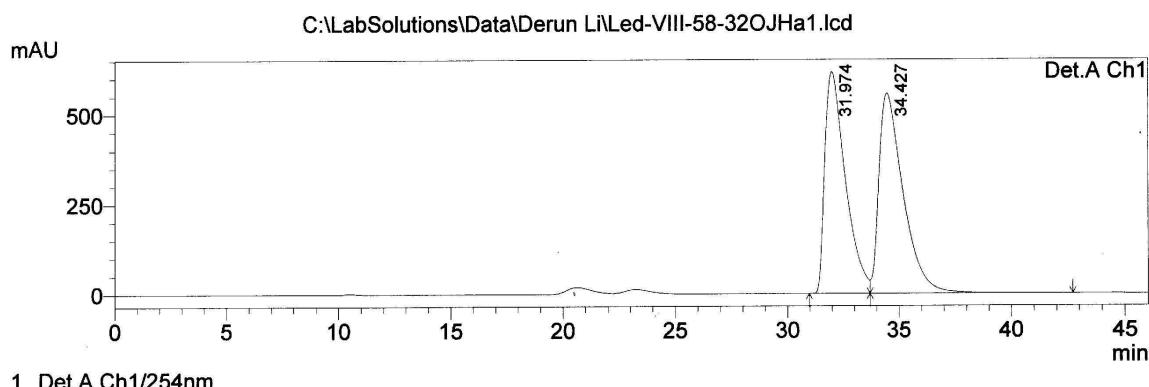
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	11.165	1103157	38484	10.580
2	15.525	9323368	250740	89.420
Total		10426526	289224	100.000



(S)-1-Cyclohexylethyl 4-nitrobenzoate (4-nitrobenzoate of 36)

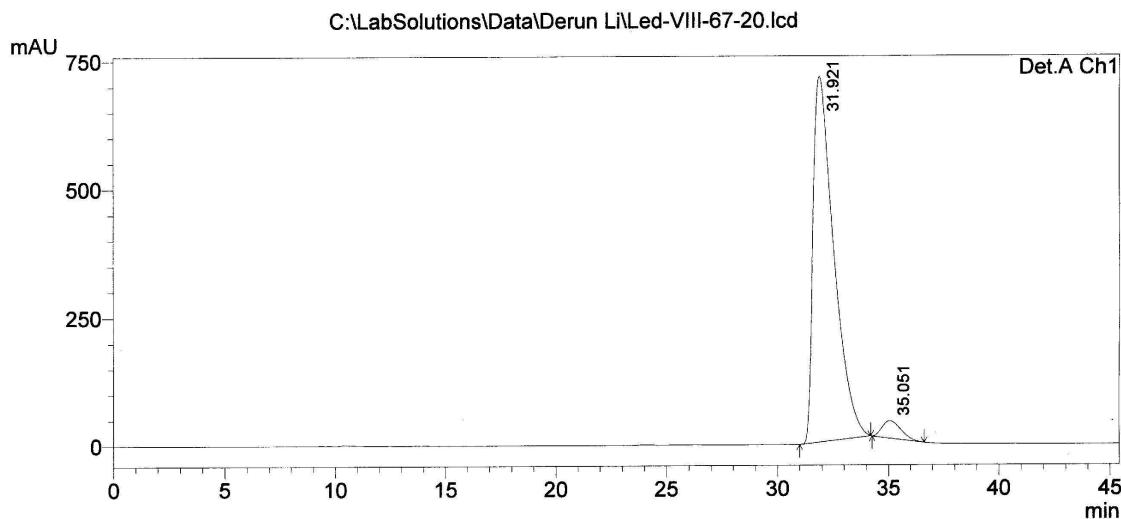
[Chiralcel OJ-H column (250 mm × 4.6 mm), 0.1% *i*PrOH/hexane, 0.4 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	31.974	39677846	617205	48.571
2	34.427	42011827	557503	51.429
Total		81689673	1174708	100.000

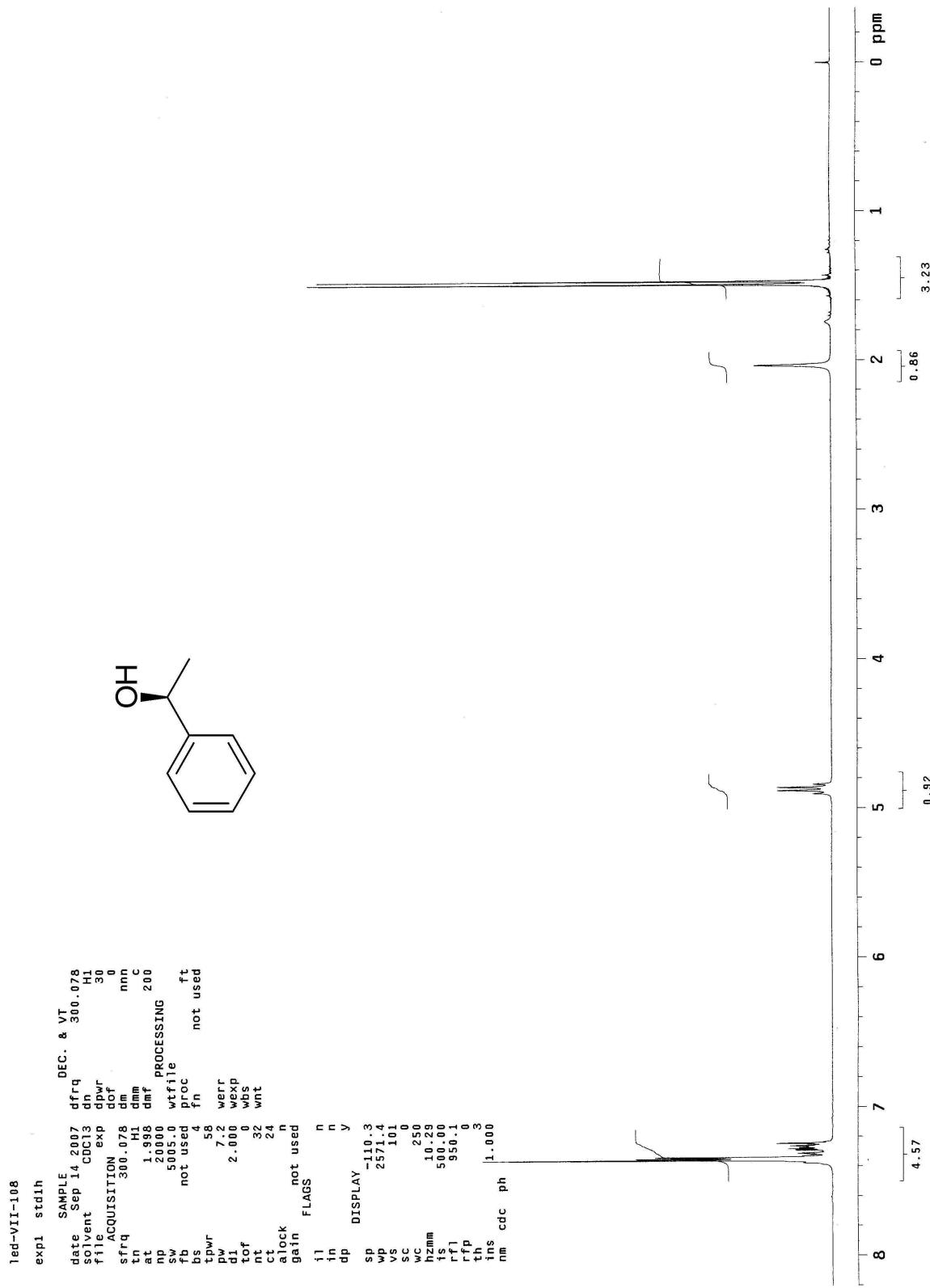


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %
1	31.921	46434821	713230	95.803
2	35.051	2034201	34307	4.197
Total		48469022	747537	100.000

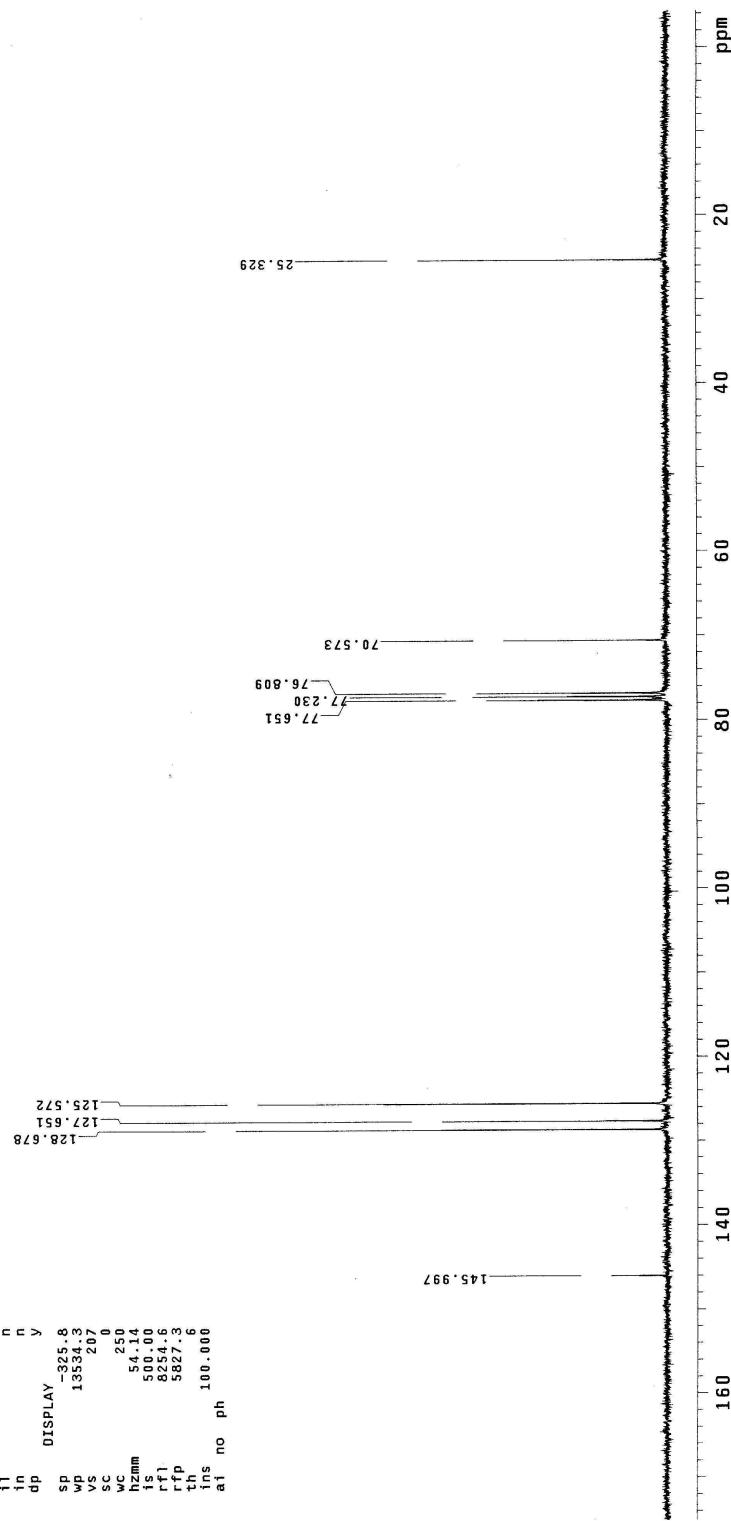
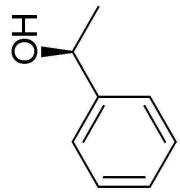
VI. NMR spectra



1ed-VII-108-C13

exp1 std13c

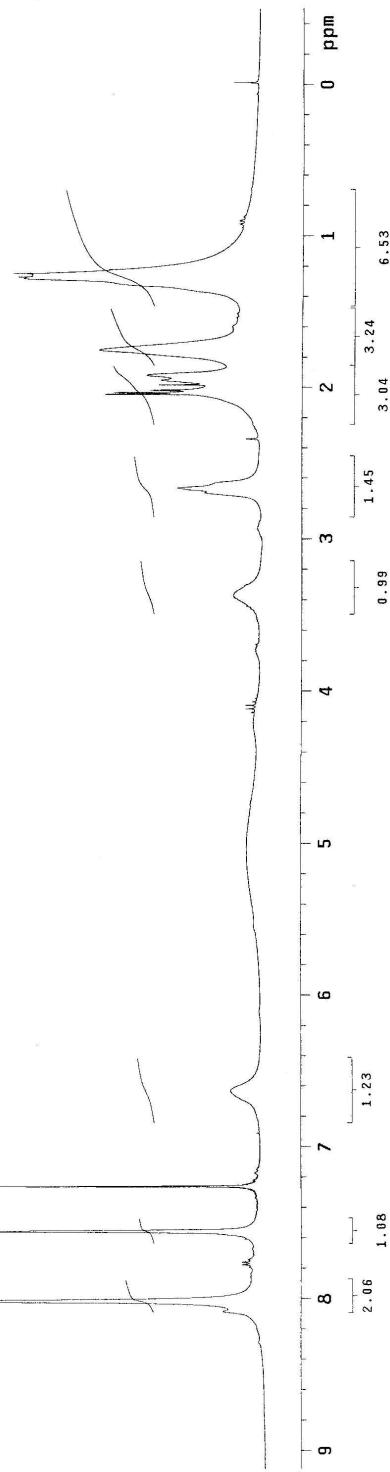
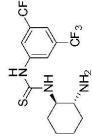
SAMPLE	DEC.	&	VT
date Sep 14 2007	dfrq	300.078	H1
solvent CDCl ₃	dn		
file	dfr	37	
ACQUISITION	dfr	0	
sfrq	dfr	0	
tn	dfr	ny	
C13	dfr		
at	dfr		
NP	dfr		
SW	dfr		
fb	dfr		
bs	dfr		
tpwr	4.4		
fw	1.000	werr	
d1	1.000	werr	
d2	1.000	wexp	
tof	1.000	wbs	
rt	1.000	wmt	
ct	192		
elock	n		
gain	n	not used	
FLAGS	n		
11	n		
1n	n		
dp	y		
DISPLAY	y		
sp	-325.8		
vp	1353.3		
vs	207		
sc	0		
vc	250		
hzmm	54.14		
ts	500.00		
rf1	8254.6		
rfp	5827.3		
th	6		
tms	100.000		
ai	no ph		

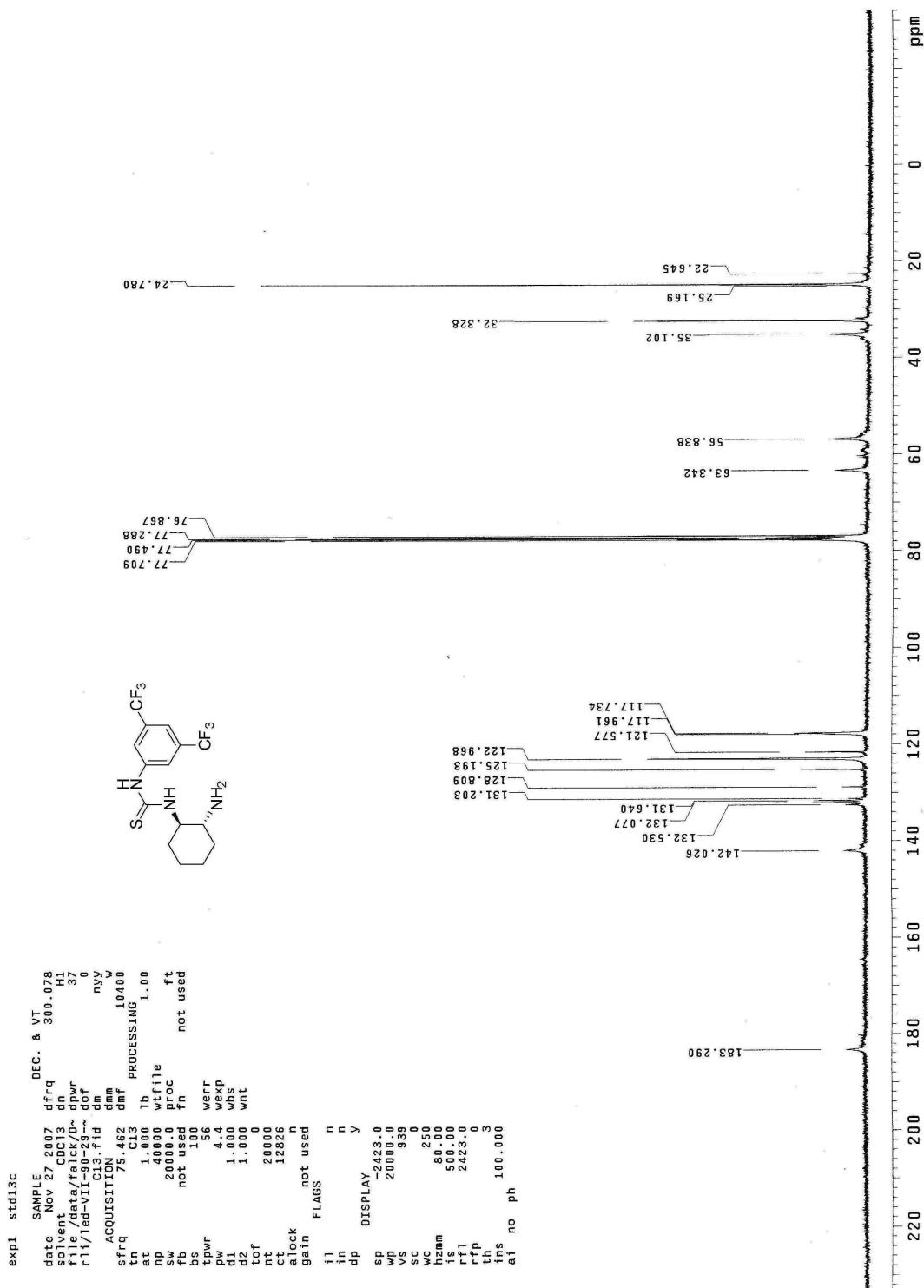


led-VII-90-29

expt st1h

SAMPLE	df,q	DEC. & VT
date Nov 27 2007	CDC13	300.078
solvent d _n	d _{pur}	H1
file exp	d _{pur}	30
ACQUISITION dof	d _{dm}	0
sfrq 300.078	d _{dm}	nnn
tn H1	c	c
at 1.998	dmf	200
np 2000.0	PROCESSING	
sw 5005.0	wtrle	
not used proc	ft	
fb 4	fn	not used
bs	58	
tpwr pw	7.2	werr
di	2.000	wbp
tof	0	wbs
nt	32	wnt
ct	24	
alock	n	
gain	not used	
FLAGS	n	
i1	n	
in	n	
dp	y	
DISPLAY		
sp -150.3		
wp 2837.0		
vs 9.1		
sc 0		
wc 250		
hzmn 11.55		
is 500.00		
rfl 945.9		
rfp 0		
th 6		
ins 2.000		
nm cdc ph		

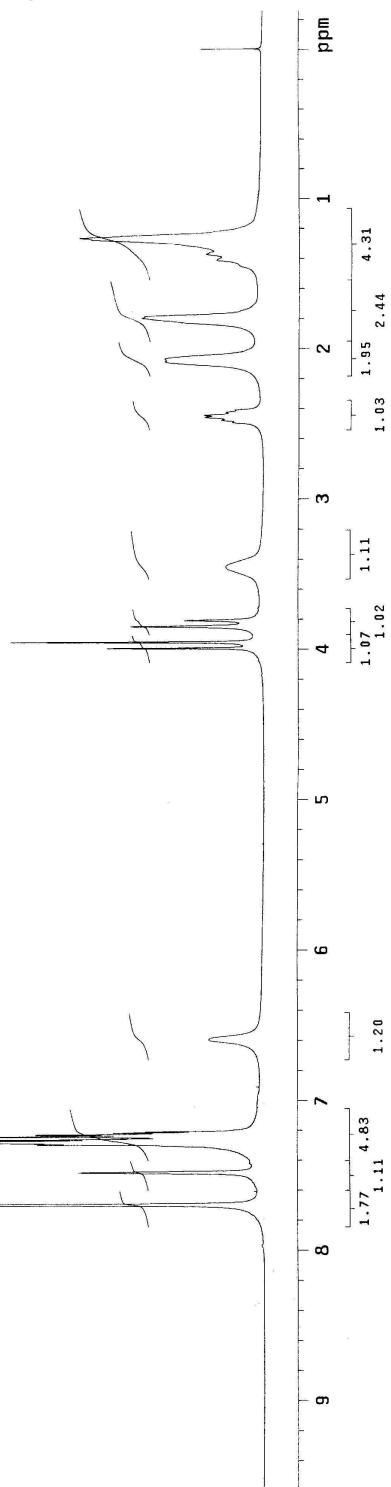
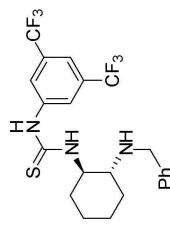




led-VII-9a-21

expt stdih

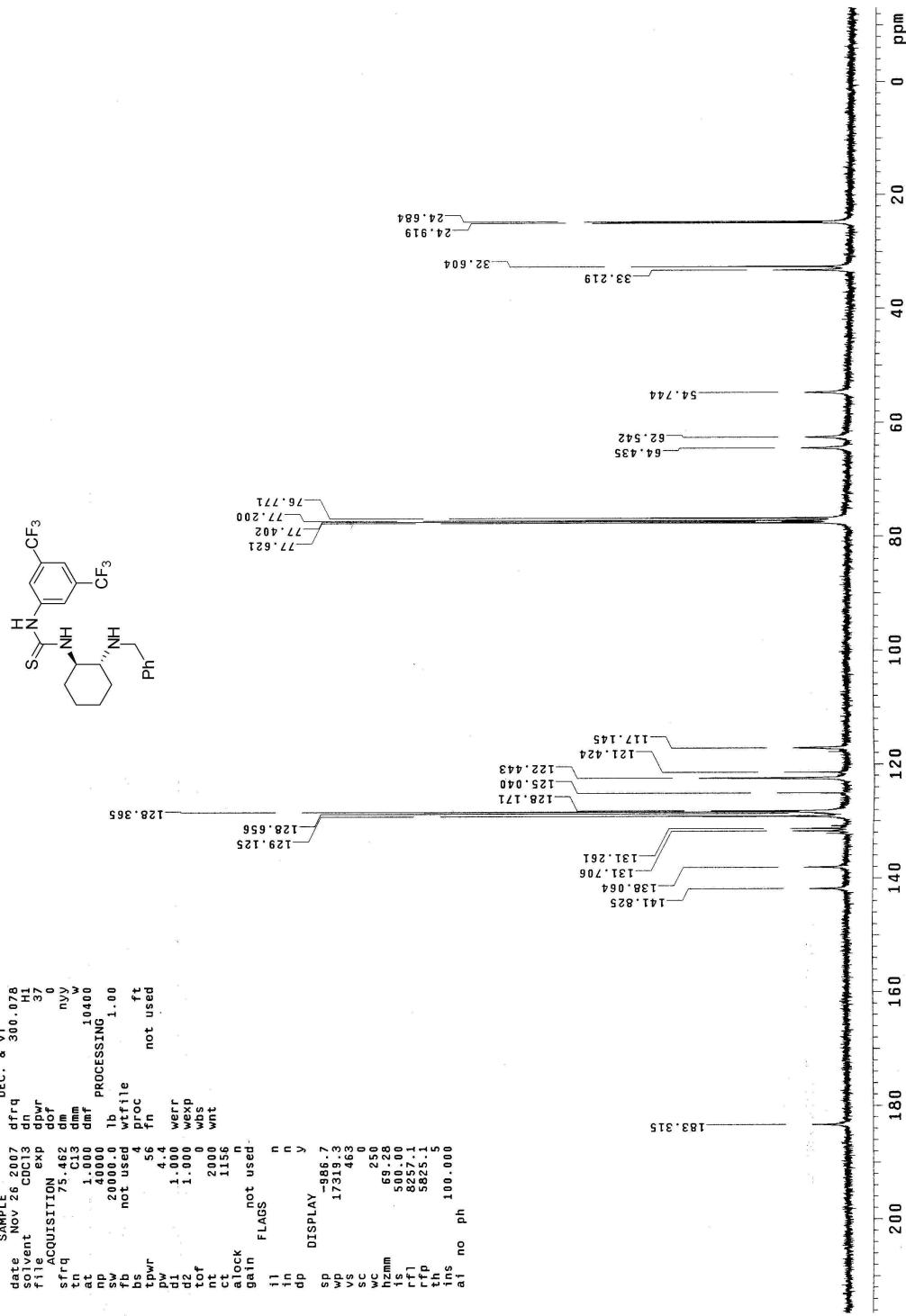
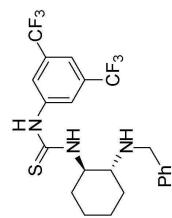
SAMPLE
date Nov 24 2007 dfrq 300.078
solvent CDCl₃ H1
file exp 30 dppr 0
ACQUISITION exp dof 0
sfrq 300.078 dim 0
tn 1.998 dmm c
at 200.00 dmf 200
sw 5005.0 wtfle
fb not used proc ft
bs 4 fn not used
tpwr 58 werr
pw 7.2 wexp
d1 2.00 wbs
t0f 0 wnt
ct 32
a0ck 28
g0in n
FLAGS
i1 n
in n
dp y
DISPLAY
sp -77.9
wp 295.0
vs 119
sc 0
wc 250
hzmm 11.81
ls 500.00
rf1 945.9
rrp 0
th 1.00
ins 1.000
nm cdc ph



led-VII-38-21-C13

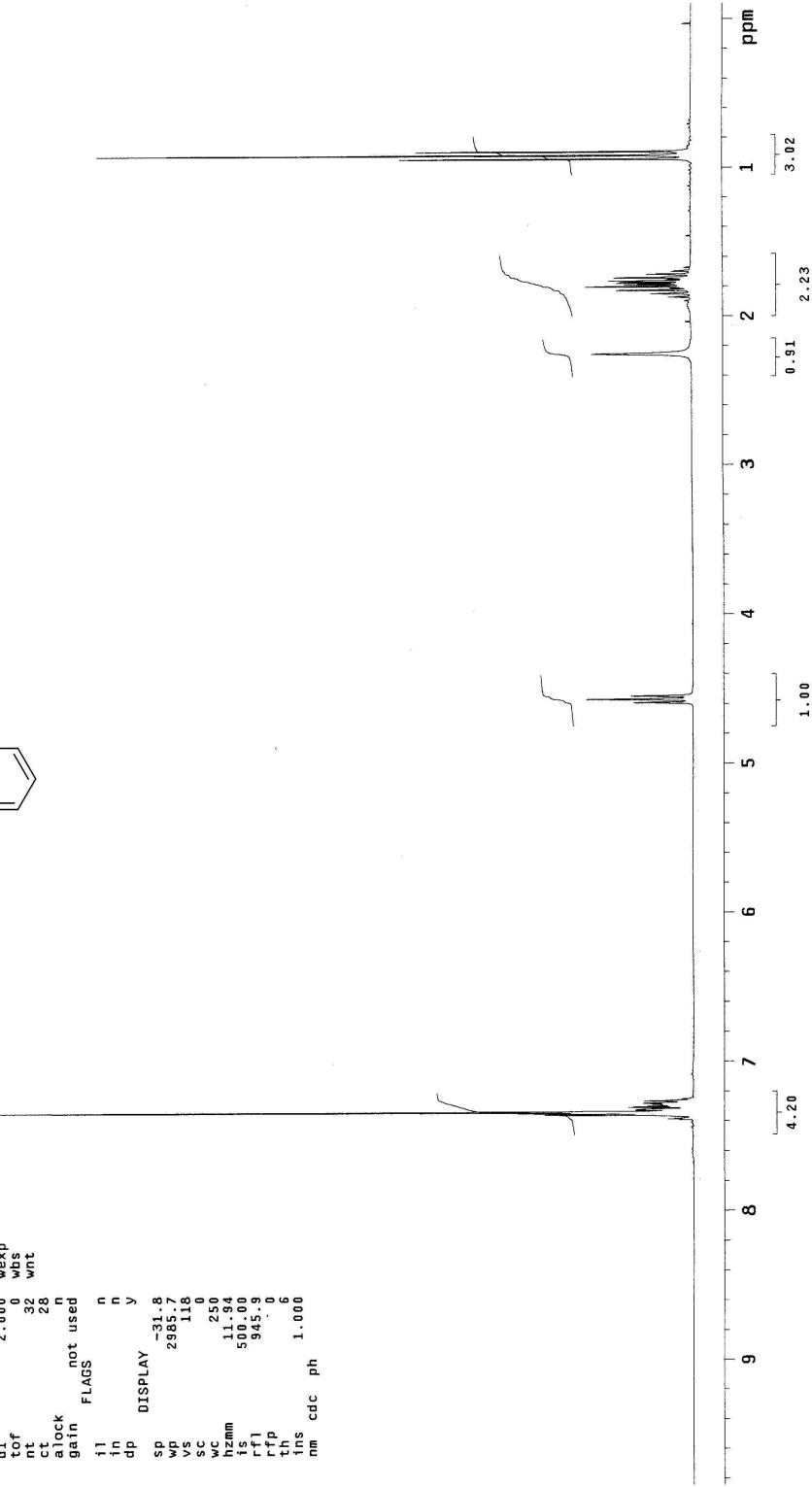
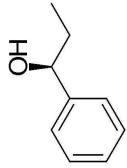
exp1 std13c

SAMPLE date Nov 26 2007 DEC. & VT 300.078
solvent CDCl₃ dfrq 300.078
file exp dn H1
ACQUISITION sfrq dpr 37
sfrq 75.462 dpr 0
tn C13 nvy
at 1.000 dm 0.000
np 40000 dmf 10.000
sw 2000.0 lbf 1.000
fb not used prc ft
bs 4 wfile not used
tpwr 56 fn not used
pw 4.4 werr
di 1.000 wexp
d2 1.000 wbs
tof 0 wnt
nt 2000 wnt
ct 1156
alock n
gain not used
11 FLAGS n
in n
dp DISPLAY y
sp -986.7
wp 17319.3
vs 463
sc 0
wc 250
hzmm 69.28
is 500.00
rf 827.1
rrp 585.1
th 100.005
ai no ph



led-VII-126-29

```
expt stdh
SAMPLE Sep 15 2007 dfrq 300.078
date      2007 dfrq 300.078
solvent   CDCl3 dn H1
file      exp dpwr 30
          dof nnn 0
ACQUISITION 300.078 dm
          300.078 dm
          1.998 dmm c
          1.998 dmf 200
          20000 PROCESSING
          5005.0 wfile
          not used proc ft
          4 fn not used
bs      tPWR 58
tPWR 7.2 werr
pw      2.000 wexp
dd1     0 wbs
tOf     0 wnt
nt      32
ct      28
alock   not used
gain    not used
11      n
in      n
dp      DISPLAY -31.8
sp      2955.7
wp      118
vs      0
sc      250
wc      11.94
hzmin  500.00
rs      945.9
rf1     0
rfp     6
th      1.000
ins    1.000
mm    cdc ph
```

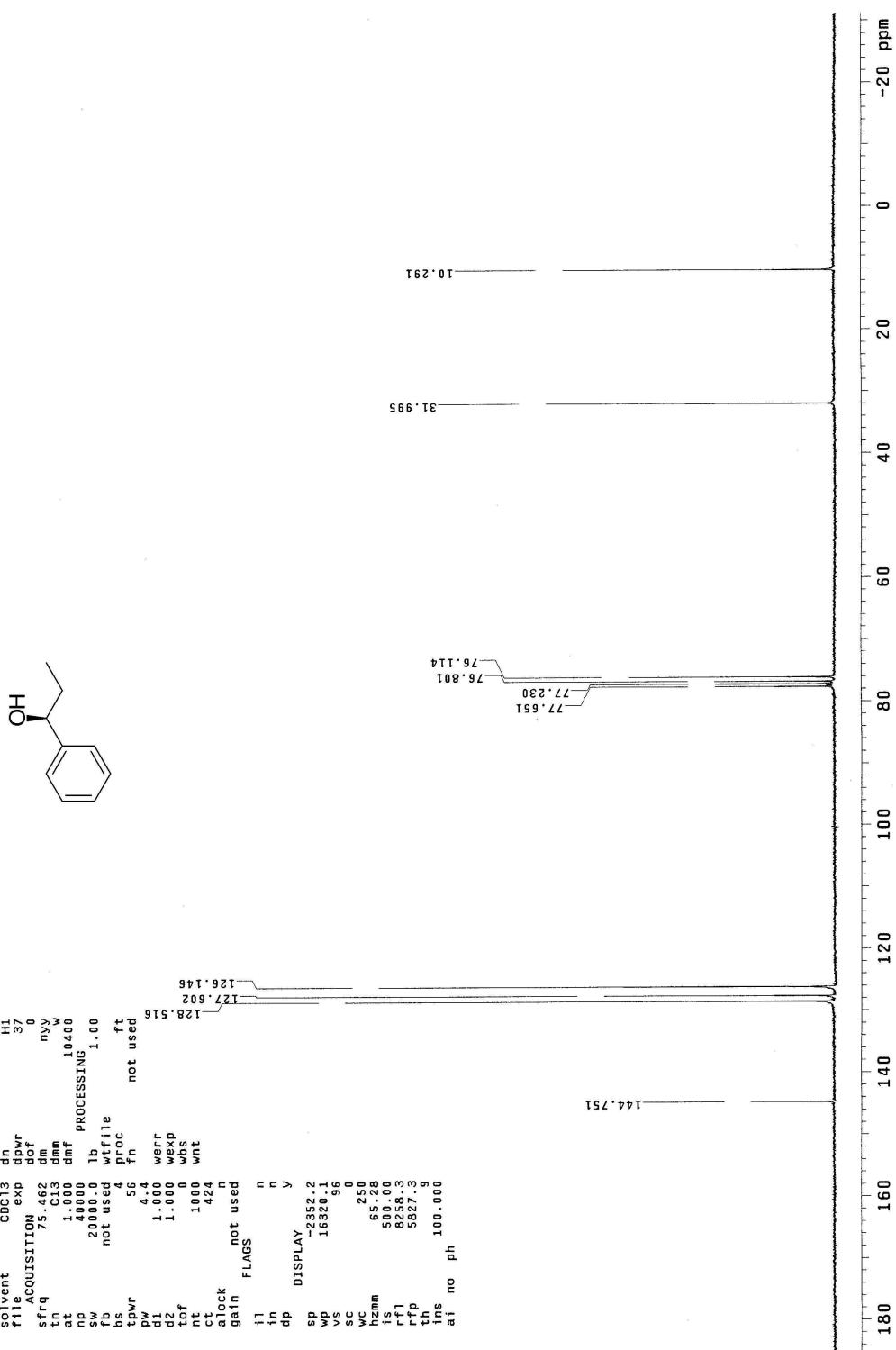
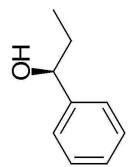


13C OBSERVE

```

expt std13c
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solvent      CDCl3
file          exp 37
ACQUISITION dprw 0
dfrq          exp
tn            71.462 dm ny
C13           0.000 dm w
at            1.000 dmf 10.00
np            40000 PROCESSING 1.00
sw            20000.0 lb 1.00
fb            not used wfile
bs            not used ft
tpwr         4 proc
pw            4.4 fn not used
d1            1.000 werr
d2            1.000 wexp
t0f           0 wbs
nt            1000 wnt
ct            424
alock         n
gain          n
FLAGS         n
i1            n
in            n
dp            DISPLAY y
sp            -2352.2
wp            16320.1
vs            96
sc            0
wc            250
hzmm         65.28
is            500.00
rf1           8258.3
rf2           5327.3
th            9
ins           100.000
ai            no ph

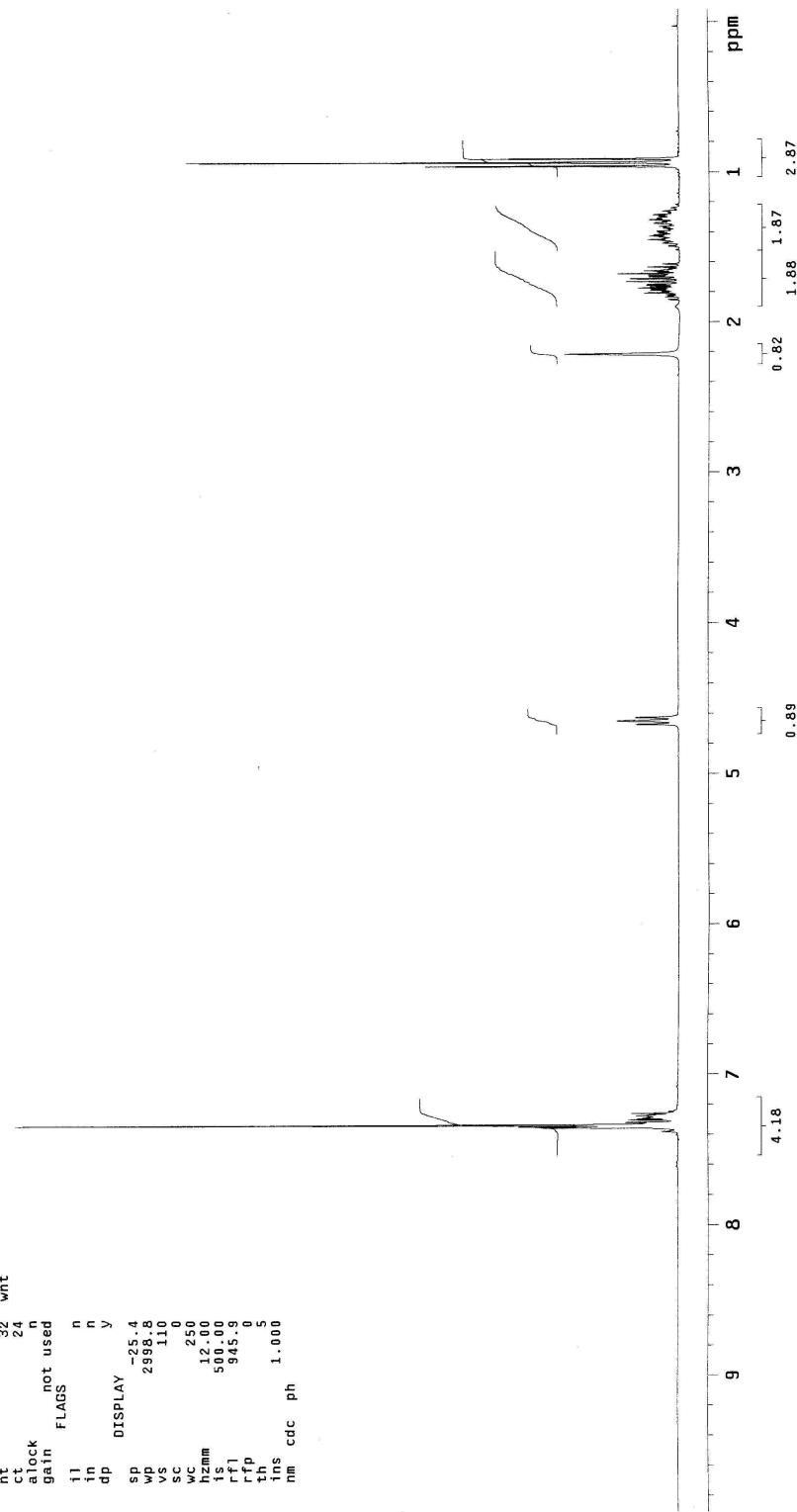
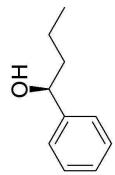
```



led-VII-141-29

exptl stth

SAMPLE	Oct 1 2007	dfrq	DEC. & VT
solvent	CDCl ₃	H1	300.078
file	exp	dpwr	30
ACQUISITION	dof	nnn	0
sfrq	300.078	dim	
tn	1.998	dmf	c
at	1.991	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	24		
clock			
gain	n		
in	FLAGS	not used	
in	n		
dp	DISPLAY	y	
sp	-25.4		
wp	298.8		
ys	1.0		
sc	0		
wc	250		
hzmm	1.00		
is	50.00		
rfl	945.9		
rfd	0		
th	5		
nm	1.000		
ins	cdc	ph	

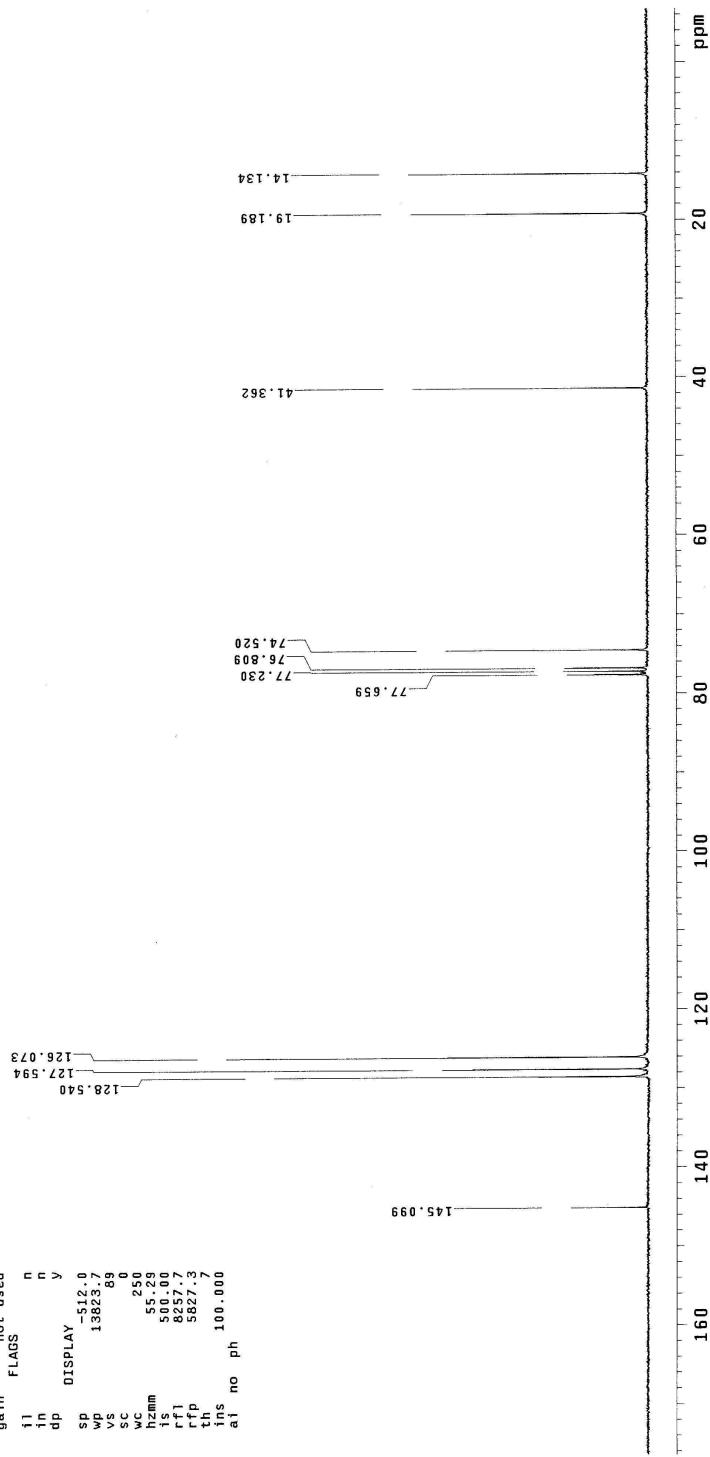
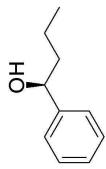


13C OBSERVE

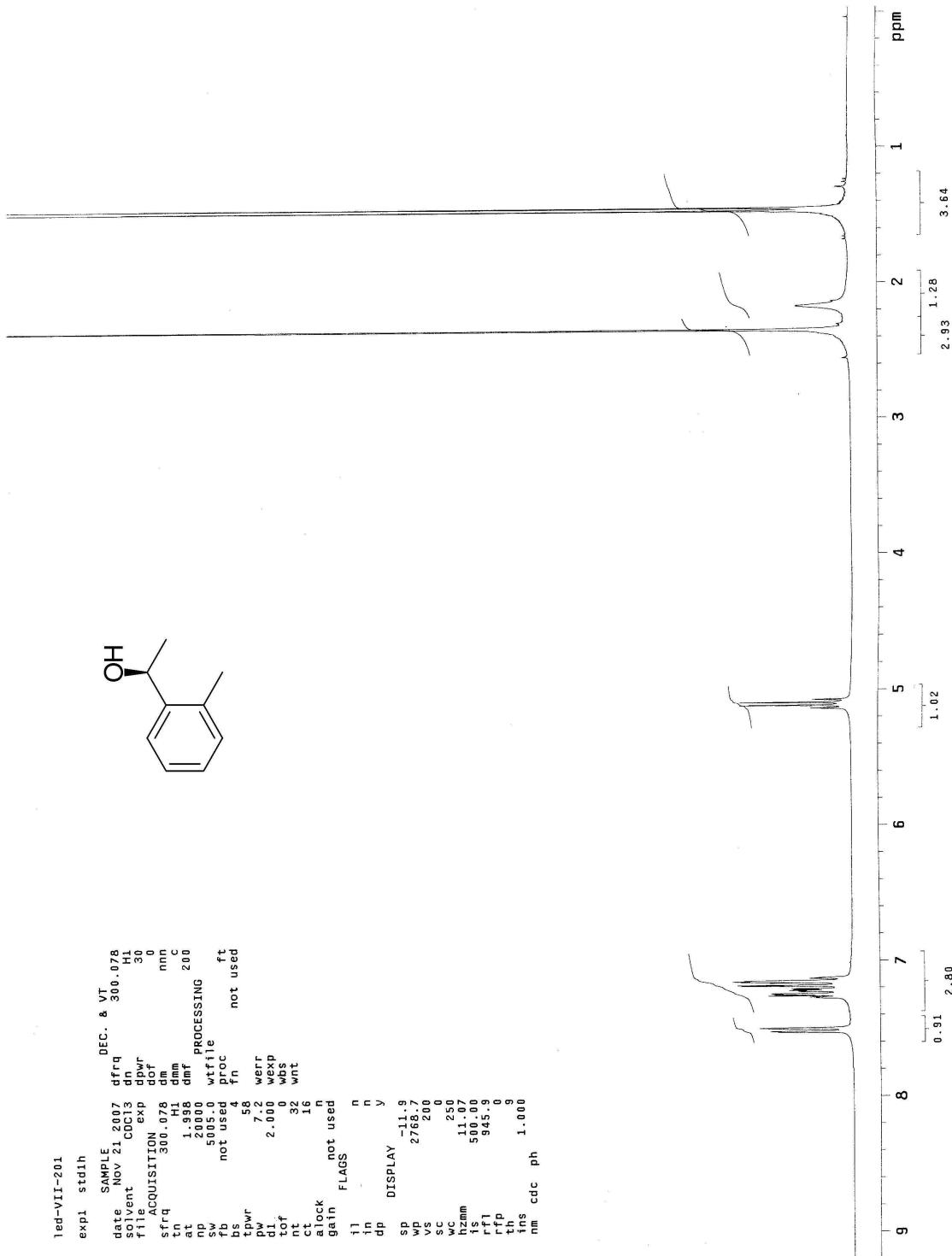
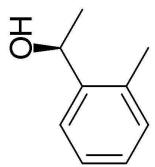
```

expl std13c
SAMPLE          DEC. & VT
date Oct 1 2007 dfrq 300.078
solvent   CDCl3      dn H1
file      exp        dPwr 37
ACQUISITION    d0f      0
sfrq      75.462     dM nyv
tn        C13       dMM w
at        1.000      dMf PROCESSING 10400
np        40000.0    lb
sw        20000.0    not used wfile 1.00
fb        4          proc ft
bs        56         fn not used
tpwr      4.4
pw        1.000      werr
d1        1.000      wexp
d2        1.000      wos
t0f       1000       wnt
nt        1000
ct        232
alock      n
gain      not used n
in        n
in        n
dp        DISPLAY y
sp        -512.0
wp        13623.7
vs        89
sc        0
wc        250
h2mm     55.29
rf1      500.60
rf2      825.77
rf3      5227.3
th        ins 100.000
ai        no ph

```



led-VII-201		DEC. & VT	
expl	std1h	SAMPLE	300.078
		date Nov 21 2007	dfrq
		solvent C013	dn
		sf ACQUISITION	dpur
		sfreq 300.078	df
		tn	dmf
		np	dmf
		sw	processing
		sb	200.0
		bs	500.0
		pwr	wtfile
		pw	not used
		di	proc
		of	ft
		ci	ft
		alock	not used
		gain	n
		FLAGS	n
		ii	n
		in	n
		dp	y
		DISPLAY	-11.9
		wp	2768.7
		vs	200
		sc	0
		wo	250
		hm	11.17
		is	500.00
		r1	945.9
		rfp	9
		nm	1.000
		cdc	ph

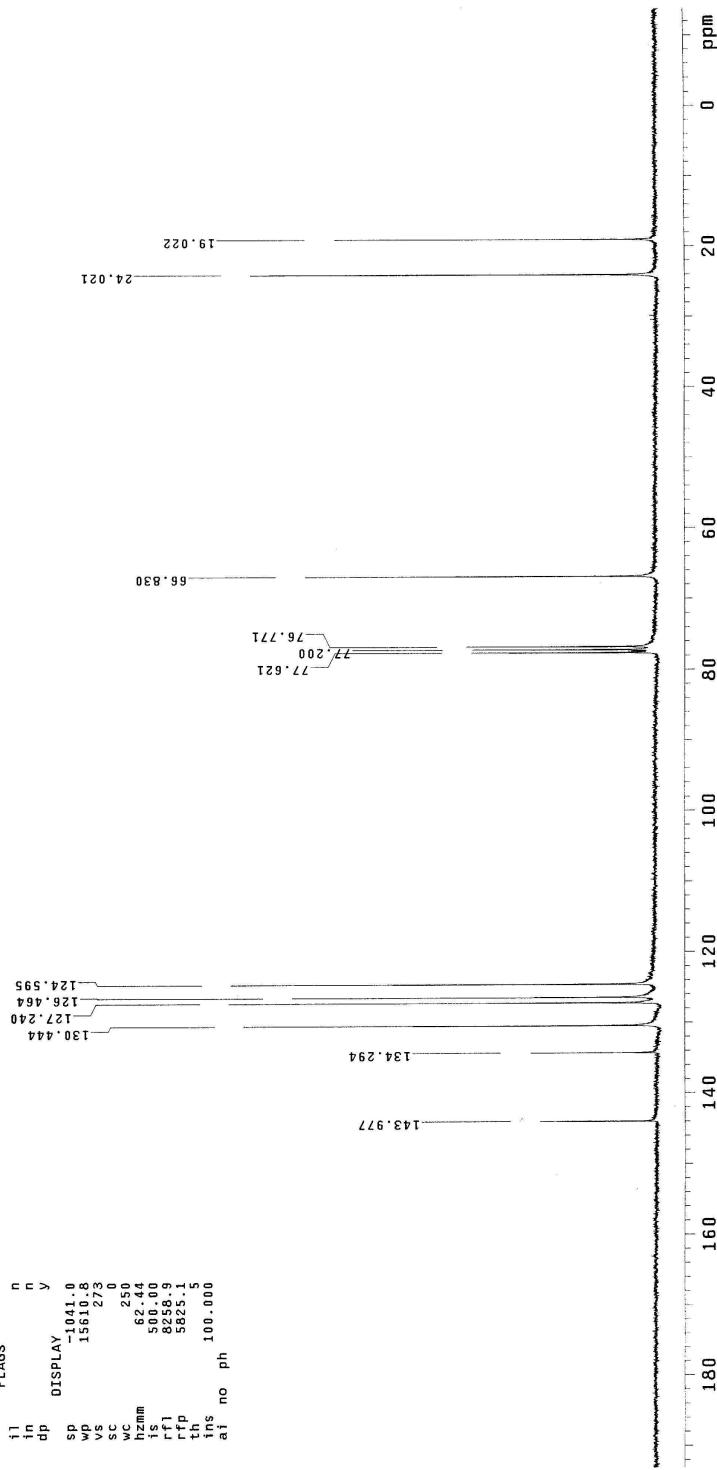
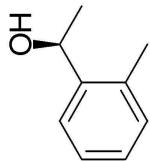


ed-VII-201-C13

```

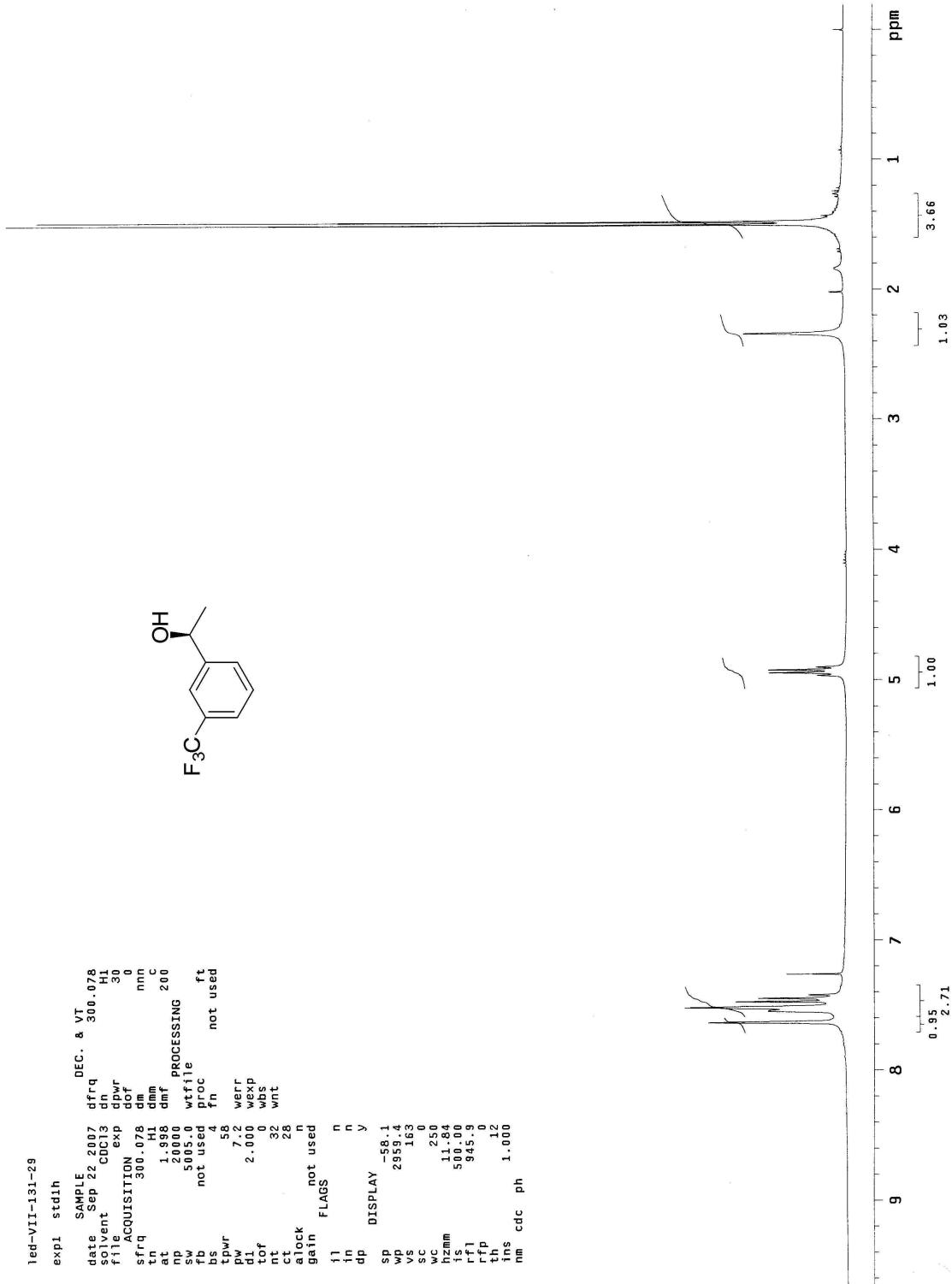
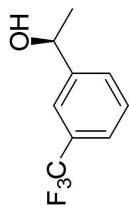
expt std3c          DEC. & VT
SAMPLE           dfrq   300.078
date Nov 21 2007 dn      H1
solvent C0C13    dpwr   37
file       exp     dof      0
ACQUISITION 75.482 dim     nyv
dfrq      C13    dim     w
at        1.000  dfrf   PROCESSING 10400
atp       4.000  lb      1.00
sw        2000.0  wftile  1.00
fb        not used
bs        4       proc   not used
bsp      56      fn     ft
tpwr     pw      4.4      not used
ddi      1.000  werr
dd2      1.000  wexp
dt        1000.0  ws
nt        1000.0  wnt
ct        892      n
alock    gain    not used
n
FLAGS          n
n
DISPLAY        n
y
sp      -1041.0
sp      156.0
wp      10.8
vs      2.3
sc      0
wc      62.4
cam    500.0
rr     88.9
rr     85.6
rr     58.9
rr     58.5
rr     5
is      100.000
is      no
is      no

```



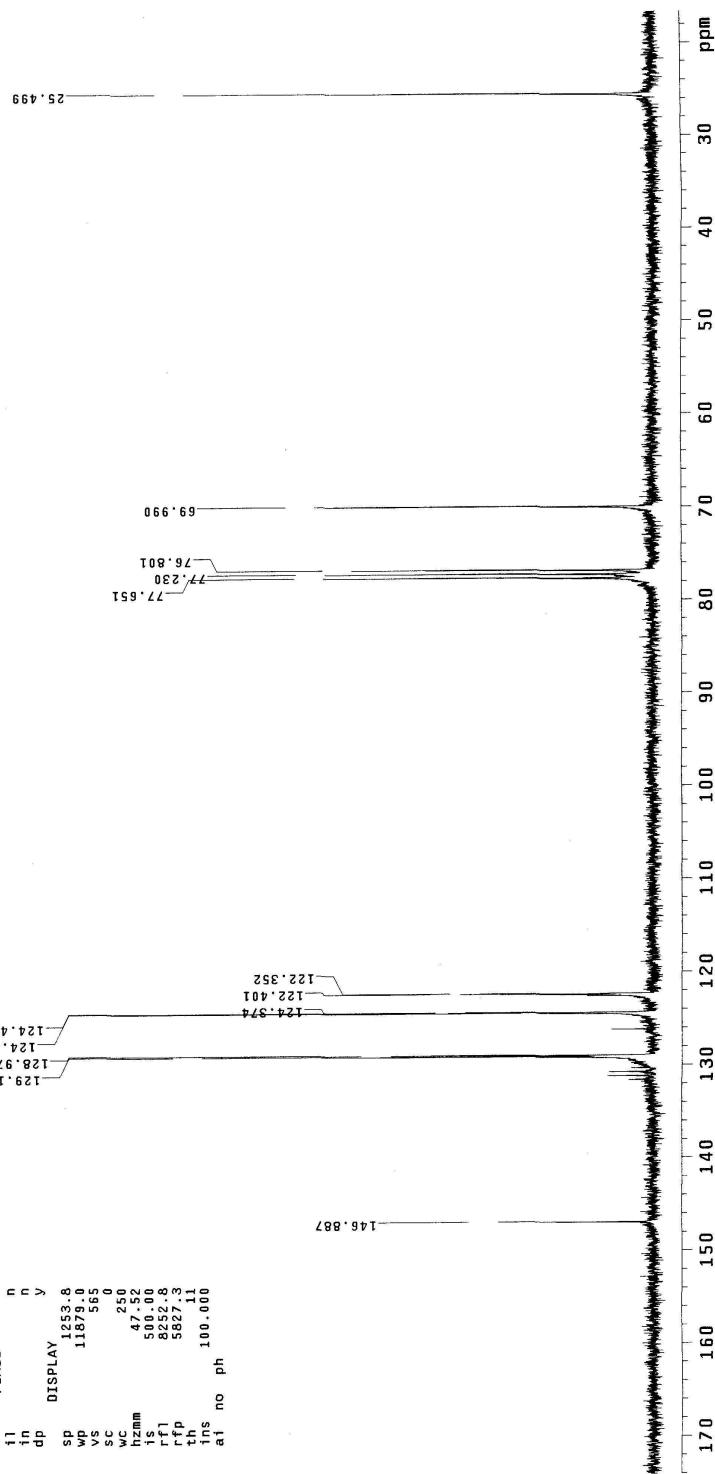
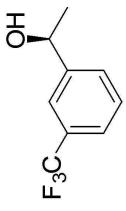
led-VII-131-29

```
expt stdih
SAMPLE Sep 22 2007 dfrq 300.078
date   2007      dn   H1
solvent CDCl3      dfrw 30
file    exp      dof 0
ACQUISITION exp      nm 0
dfrw 300.078
sirq      dn      dmm 0
tn       H1      dm 0
at        1.998      c 0
np       20000      PROCESSING 200
sw      5005.0      wfile
fb      not used      ft
fb      not used      f1
bs      4      not used
twr      58      not used
pw      7.2      werr
di      2.000      wexp
t0f      0      wbs
nt      32      wnt
ct      28
clock      not used
gain      n
FLAGS
ii      n
in      n
dp      y
DISPLAY ?-58.1
sp      -58.1
wn      2959.4
vs      163
sc      0
wc      250
h3mm      11.84
is      50.00
rf1      945.9
rf2      0
tb      12
ins      1.000
nm      cdc      ph
```



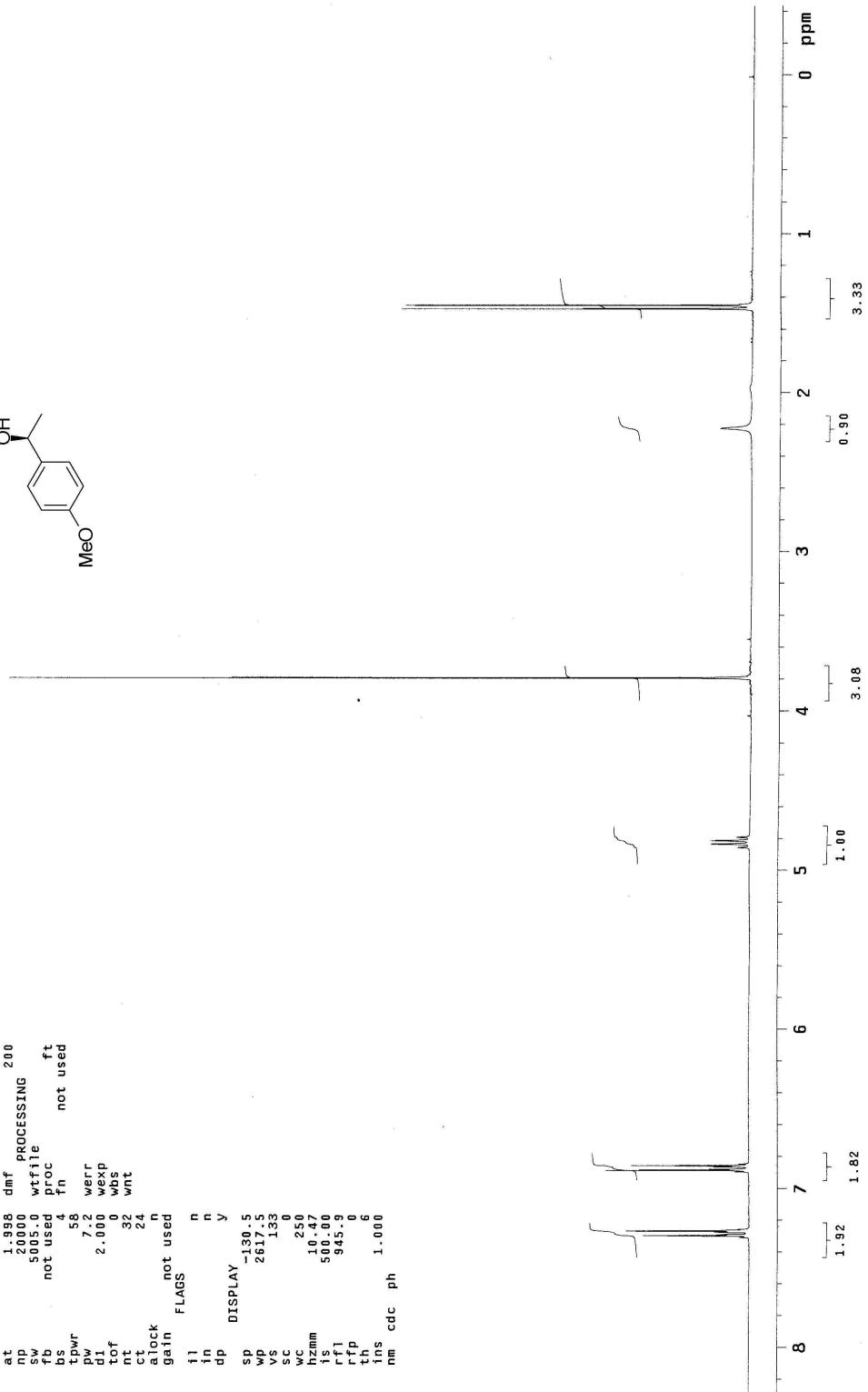
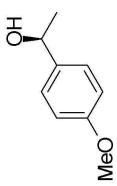
led-II-131-29-C13

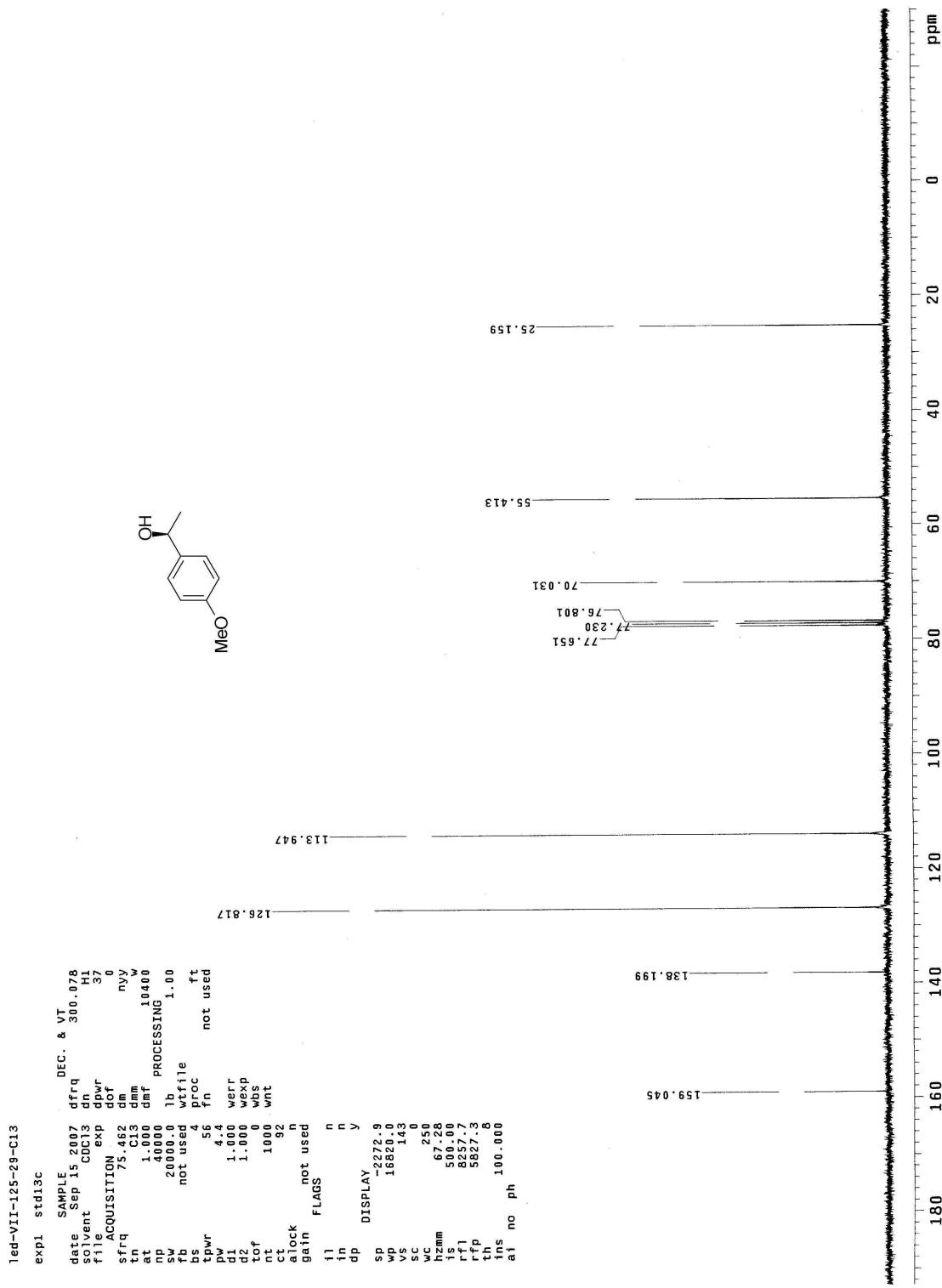
```
exp1 std13c
      SAMPLE DEC. & VT
      date Sep 22, 2007 dfrq 300.078
      solvent CDCl3
      file exp d1w
      $f1q 75.462 d1w
      tn C13 dim navy
      at 1.000 dmf yellow
      np 40000 lb PROCESSING
      sw 20000.0 1b wfile 1.00
      fb not used proc ft
      bs 4 f1n not used
      tpar 56 pw 4.4
      pw 4.4 d1 1.000 werr
      d2 1.000 wexp
      tof 0 whs
      nt 1000 wnt
      ct 448
      alock n
      gain not used
      flags n
      il n
      in n
      dp DISPLAY y
      sp 1253.8
      wp 11879.0
      vs 565
      sc 0
      wc 250
      hzmm 47.52
      is 500.00
      rfl 8252.8
      rfp 5827.3
      th 11
      ins 100.000
      ai no ph
```



led-VII-125-29

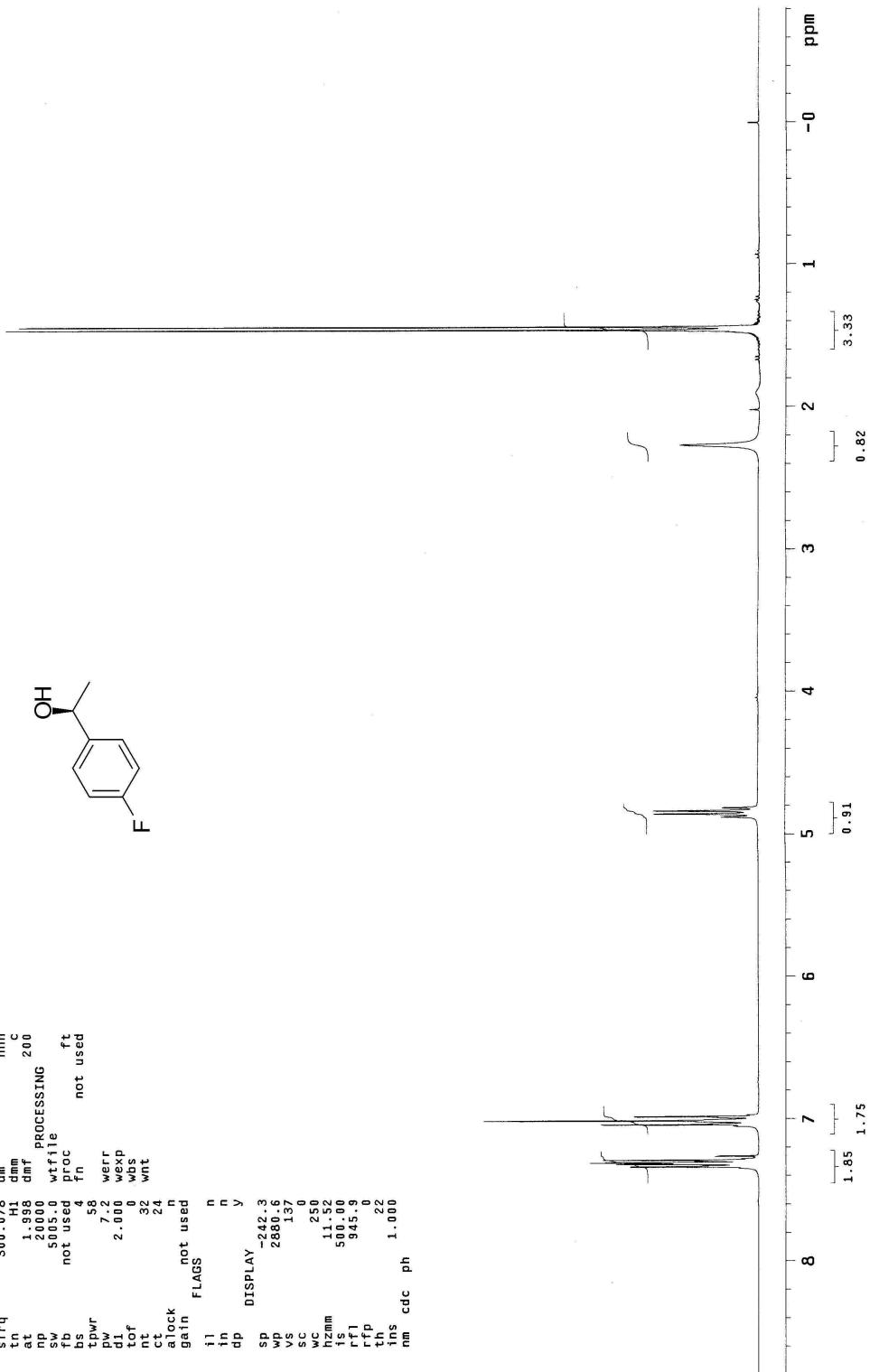
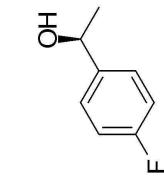
expt1 std1h
SAMPLE DEC. & VT
date Sep 15 2007 dfrq 300.078
solvent CDCl₃ dn H1
f1e exp 30
d1 pwr 0
d1e acq 0
sf1q 300.078 dm nnn
tn 1.995 dm c
at 1.995 dm
np 20000 PROCESSING 200
sw 50.000 wtime
fb not used proc ft
bs 4 fn not used
tpm 58
pw 7.2 werr
d1 2.000 wexp
t0r 0 wbs
nt 32 wnt
ct 24
alock
gain n
flags not used
ii n
in n
dp y
sp DISPLAY -130.5
wp 2617.5
vs 133
sc 0
wc 250
hzm 10.47
is 500.00
rf1 945.9
rf2 0
th 6
inc 1.000
nm cdc ph





led-VII-125-27

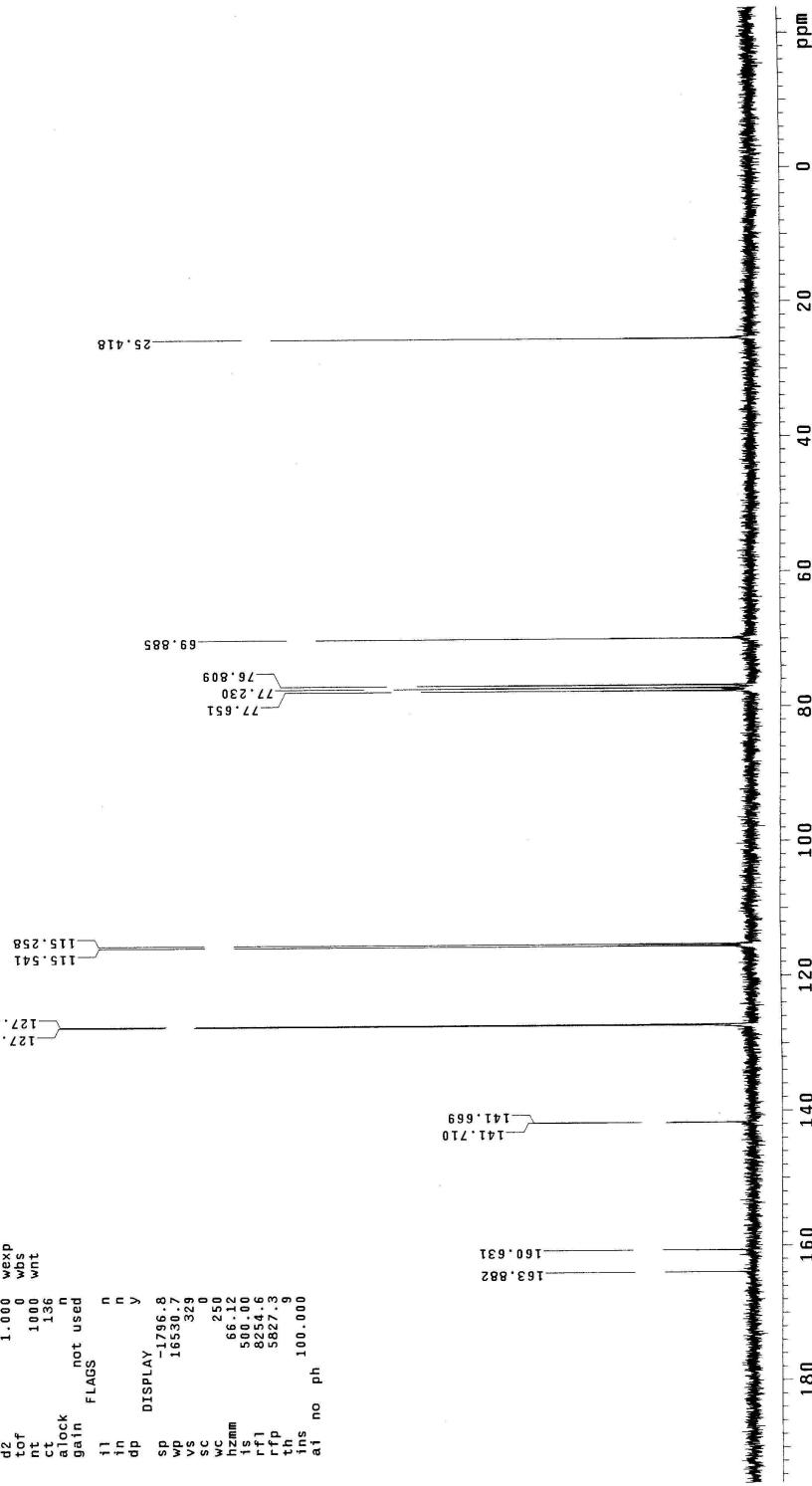
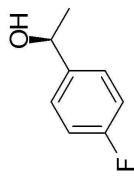
```
exp1 stdih
      SAMPLE   DEC. & VT
      date Sep 19 2007 dfrq 300.078
      solvent CDCl3 dn H1
      file      exp dppr
      ACQUISITION exp dpr
      sfrq 300.078 dm 0
      tn      1.938 dmm 0
      at      20.000 dmt 0
      np      500.0 wfile 200
      sw      500.0 wproc ft
      fb      not used
      bs      4 fn not used
      t1wr    58 werr
      pw      2.000 wexp
      d1      0 wbs
      nt      32 wnt
      ct      24
      alock   not used
      gain    FLAGS
      i1      n
      in      n
      dp      DISPLAY y
      sp      -242.3
      wp      2580.6
      vs      137
      sc      0
      wc      250
      hzmm 11.52
      is      50.00
      rf1    945.9
      rfp    0
      th      22
      nm      cdc ph 1.000
```



led-VII-128-27-C13

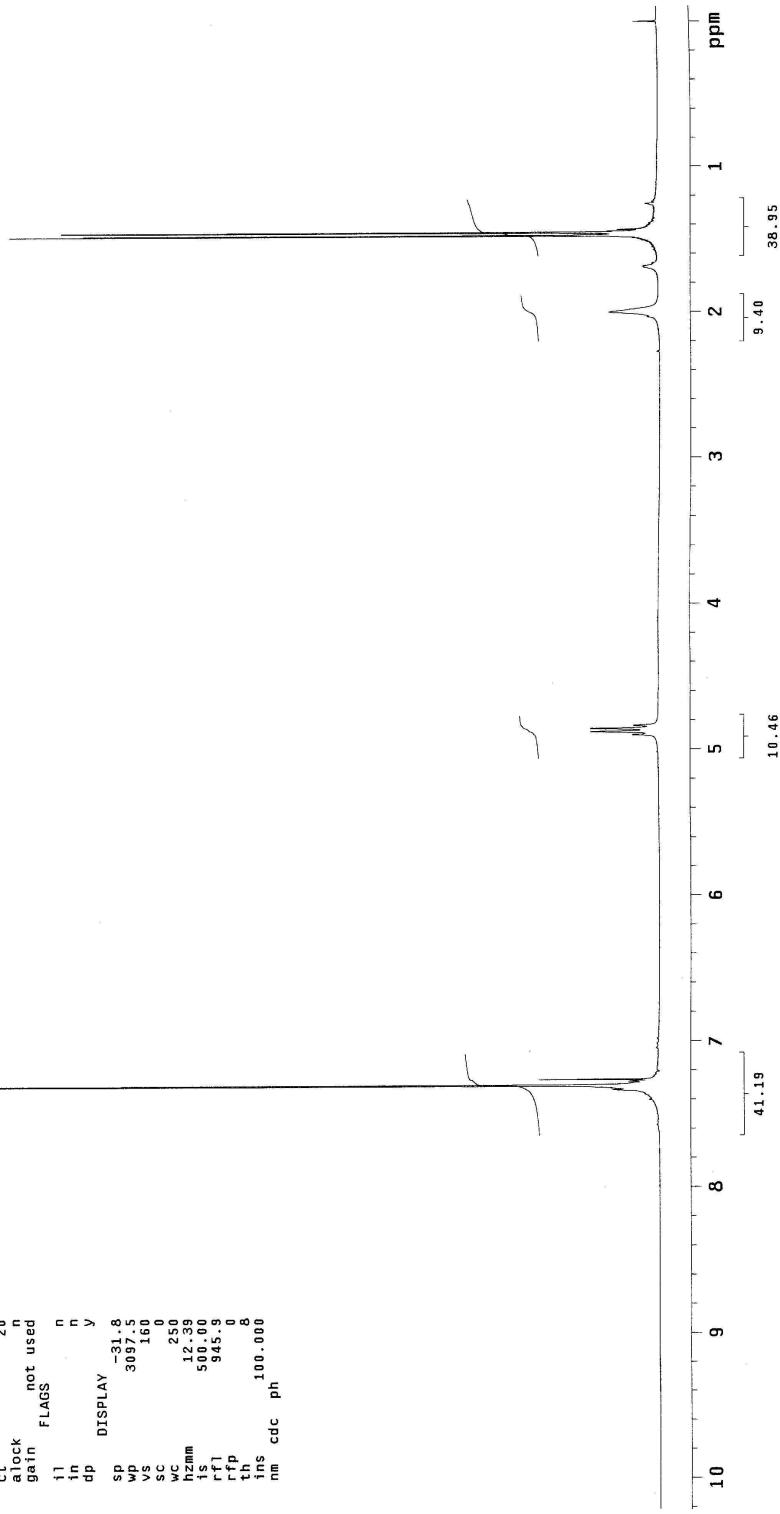
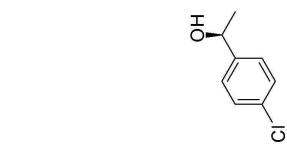
expl st113c

SAMPLE	19.2007	DEC. & VT	300.078
date	Sep 19 2007	dfrq	CDC13
solvent	dn	dpvr	H1
file	exp	dpr	37
sfrq	ACQUISITION	dm	0
tn	75.462	dm	ny
at	C13	dm	w
np	1.000	dm	10400
sw	4000.0	lb	PROCESSING
fb	20000.0	wtile	1.00
bs	not used	proc	ft
tpwr	4	fn	not used
pw	56	4.4	
d1	1.000	werr	
d2	1.000	wexp	
tof	1000	wbs	
nt	1000	wmt	
ct	136		
alock	n		
gain	not used		
FLAGS	n		
11	n		
in	n		
dp	DISPLAY	y	
sp	-1796.8		
wp	16530.7		
vs	329		
sc	0		
wc	250		
hzmm	66.12		
is	500.00		
rr1	8224.6		
rrp	5827.3		
th	100.00		
ins	9		
ai	no	ph	



led-VII-15g-29

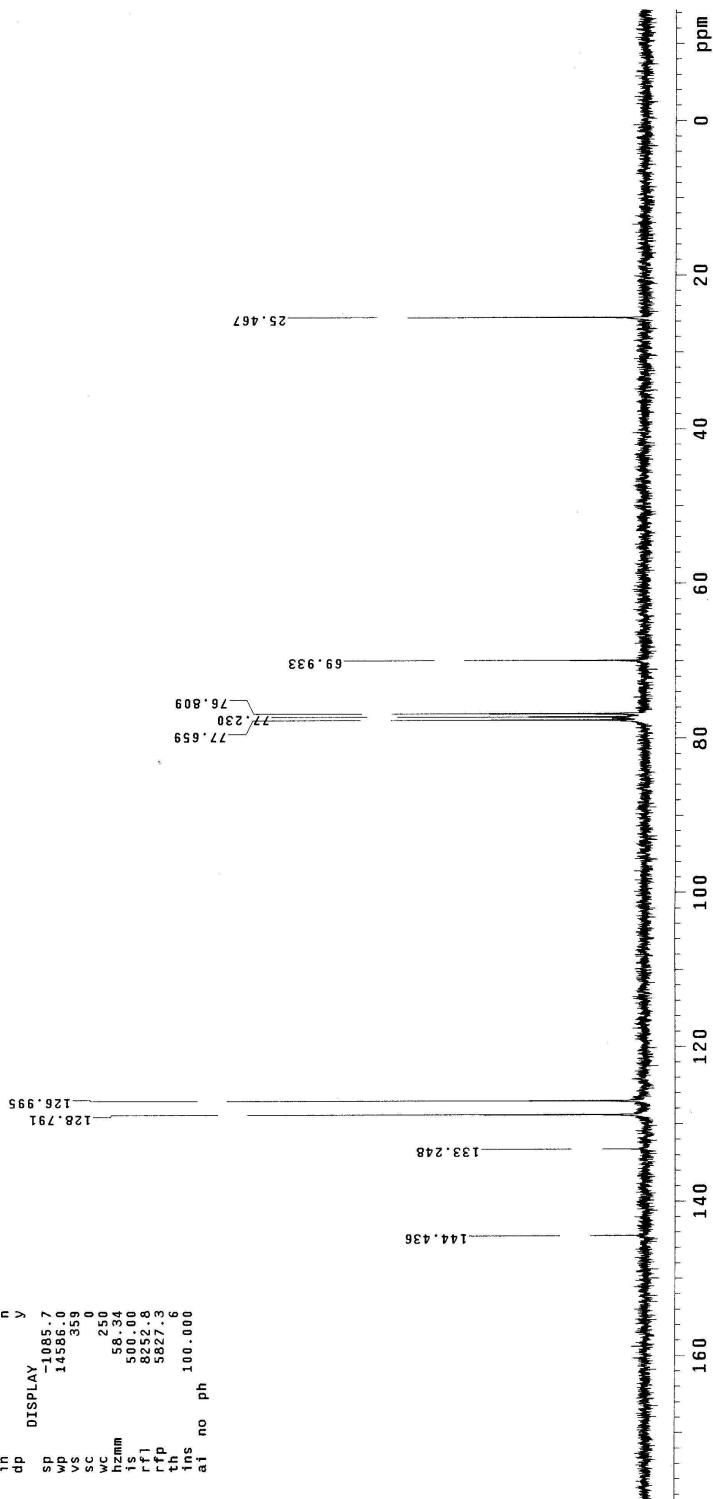
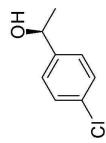
exp1 std1h
SAMPLE date Oct 9 2007 dfrq DEC. & VT
solvent CDCl₃ dfrq H1
f1frq exp dfrq H1
ACQUISITION 300.078 dfrq 300.078
t1 300.078 dfrq mm 0
sfrq 1.998 dmm c
at 1.998 dmf
np 20000.0 wfile
sw 5015.0 proc ft
fb not used fn not used
tpwr 58 pwerr
pw 7.2 wexp
d1 2.00 wbs
tof 0 wnt
nt 32
ct 20
a lock n
gain not used
FLAGS
i1 n
in n
dp DISPLAY y
sp -31.8
wp 309.5
vs 160
sc 0
wc 250
h2mm 12.39
1s 500.00
rf1 945.9
rfp 8
th ins 100.000
nm cdc ph 100.000



led-VII-159-29-C13

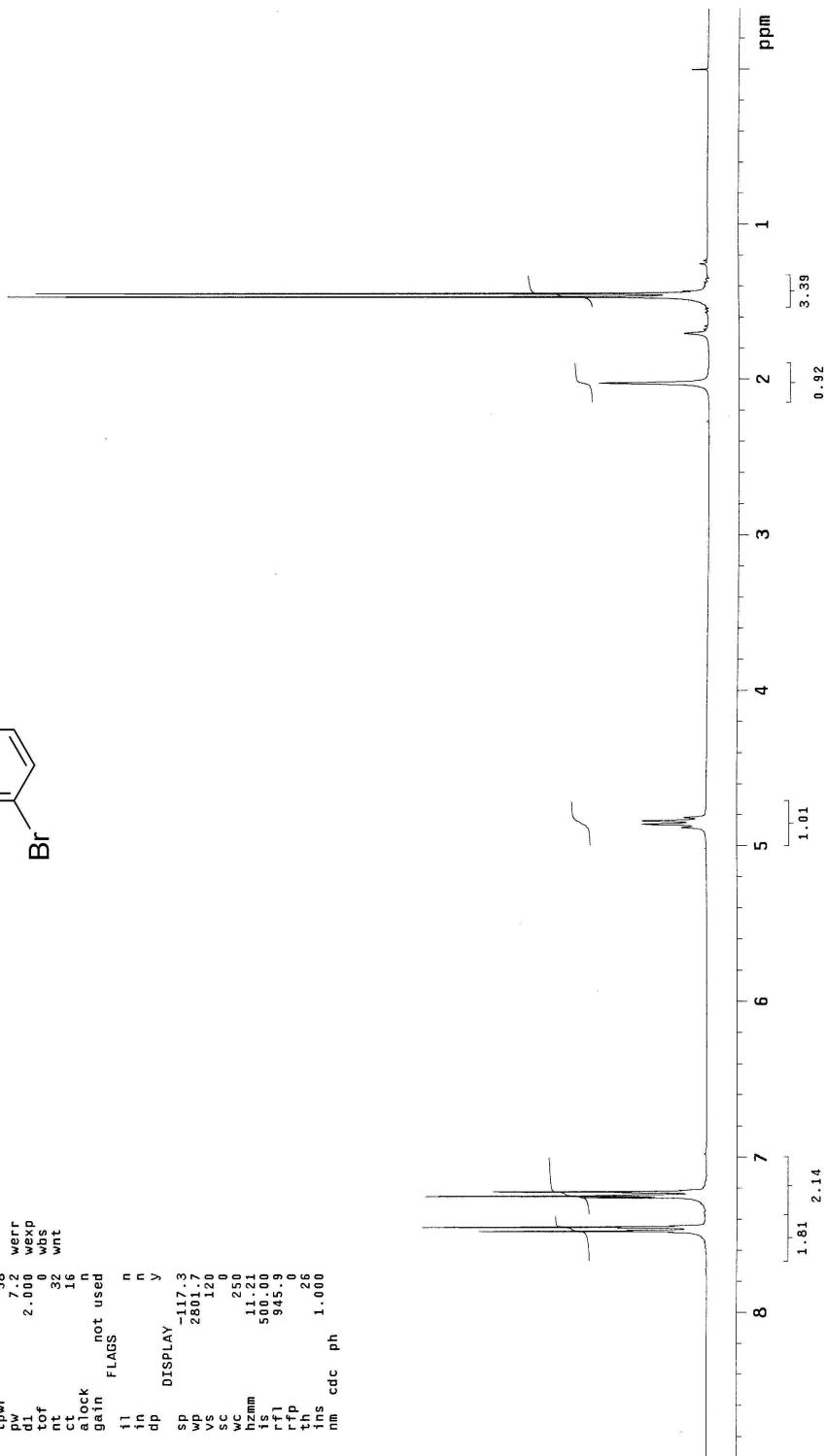
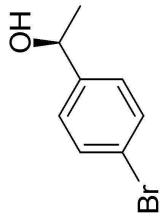
expi stdisc

SAMPLE	DEC.	&	VT
state Oct 9 2007	dfrq		300.078
solvent CDCl ₃	dn		H1
file exp	dpr		J37
sfrq	dof		nav
tn	C13		W
at 1.000	dmm		10400
np 20000.0	dmf		
sw 20000.0	lb		
fb not used	wfile		
bs 4	proc		
tpur	56	fn	ft
pw 4.4		not used	
d1 1.000	werr		
d2 1.000	wexp		
tof 0	wbs		
nt 1000	wnt		
ct 164			
alock n			
gain not used			
FLAGS			
i1 n			
in n			
dp y			
DISPLAY			
sp -1085.7			
wp 14586.0			
vs 359			
sc 0			
wc 250			
hzmm 58.34			
is 500.00			
rf1 852.8			
rfp 5827.3			
th 6			
ins 100.000			
ai no ph			



led-VII-161-59

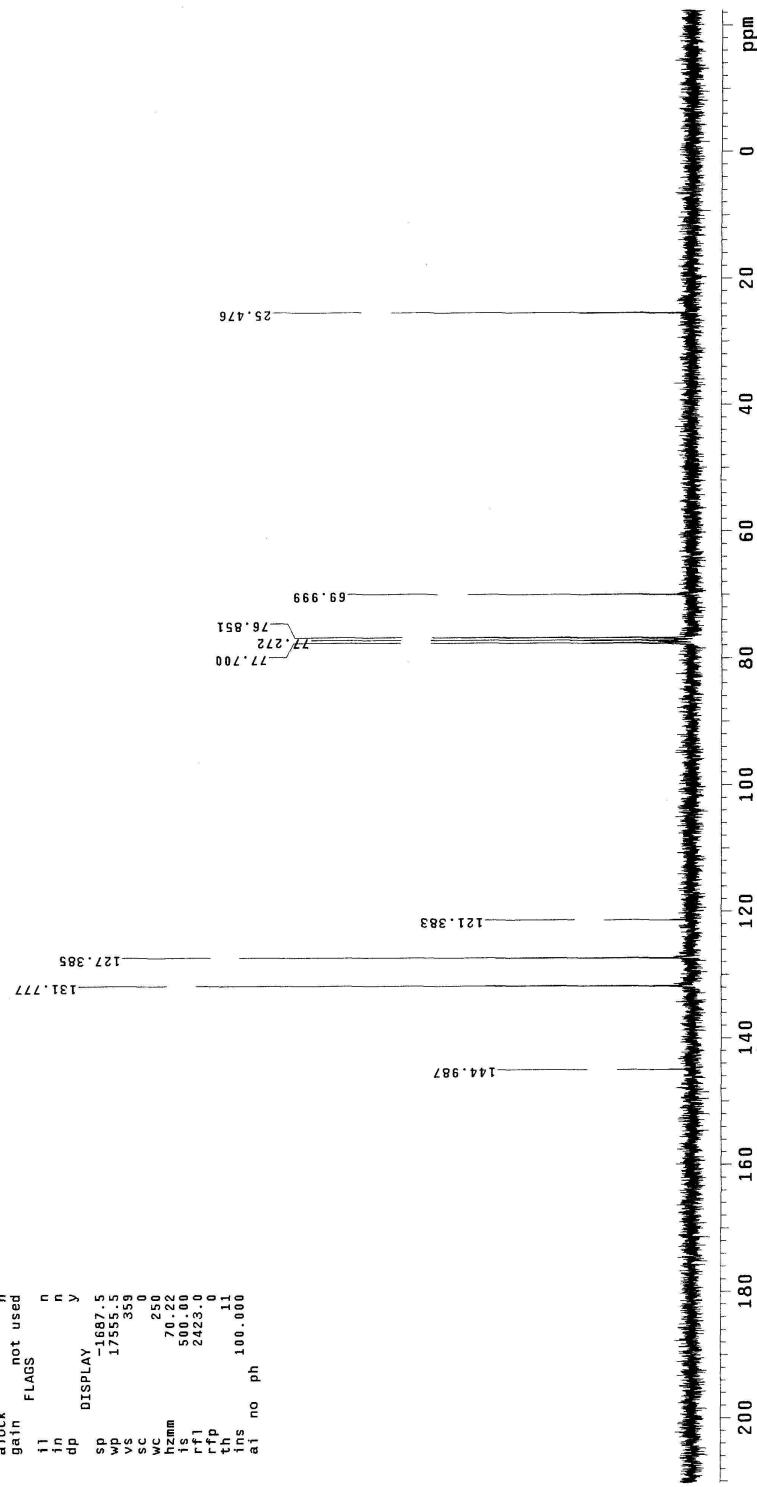
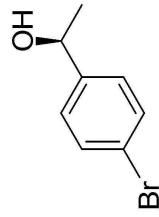
expt stdlh
SAMPLE Oct 9 2007 dfrq 300.078
solvent CDCl₃ dn H1
file exp dpwr 30
ACQUISITION exp dior 0
sfrq 300.078 dm nnn
tn H1 dmm c
at 1.998 dmf 200
np 20000 PROCESSING
sw 5005.0 wfile e
fb not used proc ft
bs 4 fn not used
twr 58 werr
pw 7.2 wexp
d1 2.000 wbs
t0f 0 wnt
nt 32
ct 16
clock not used
gain not used
FLAGS
ii n
in n
dp y
DISPLAY 2
sp -117.3
wp 2801.7
vs 120
sc 0
wc 250
h_{mm} 11.21
is 500.00
rf 945.9
rfp 0
tb 26
ins 1.000
nm cdc ph 1.000

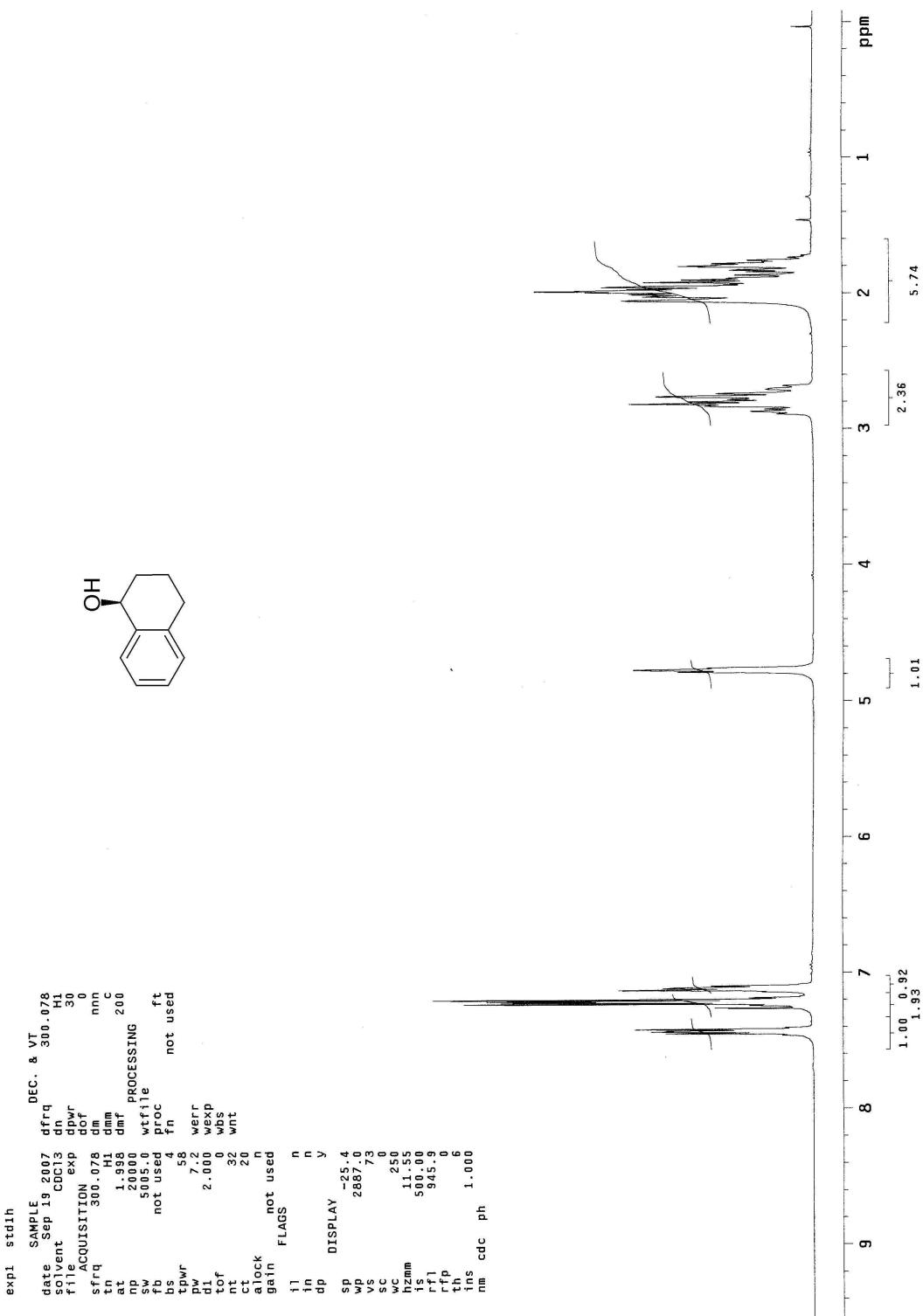


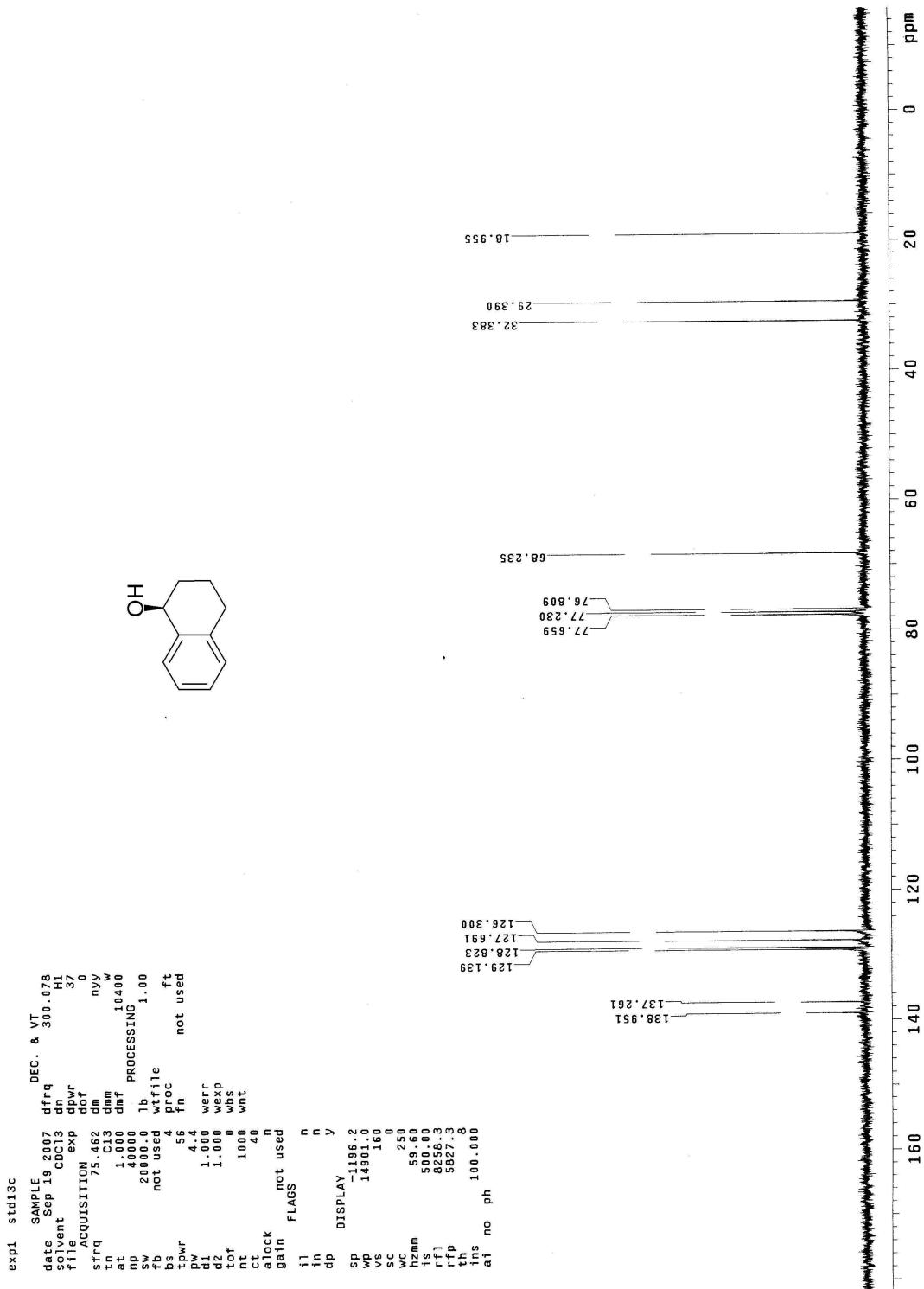
led-VII-61-59-C13

expi std13c

SAMPLE	DEC.	&	VT
date Oct 9 2007	dfrq		300.078
solvent CDCl ₃	din	H1	
file exp	dprf		37
sfrq	dfrf	0	
ACQUISITION 75.462	dim		
tn	C13		
at 1.000	dmm		
np 40000	dimf		W
sw 20000.0	lb	PROCESSING	10400
fb not used	wtrile		1.00
bs 4	proc		
tpwr 56	fn		
pw 4.4	not used		
d1 1.000	werf		
d2 1.000	wexp		
tof 0	wbs		
nt 1000	wnt		
ct 72			
alock			
gain	FLAGs	not used	
11	n		
in	n		
dp	DISPLAY	y	
sp	-1687.5		
wp	1755.5		
vs	359		
sc	0		
wc	250		
hzmn	70.22		
is	500.00		
rf1	242.0		
rfp	0		
th	11		
ins	100.00		
ai no	ph		

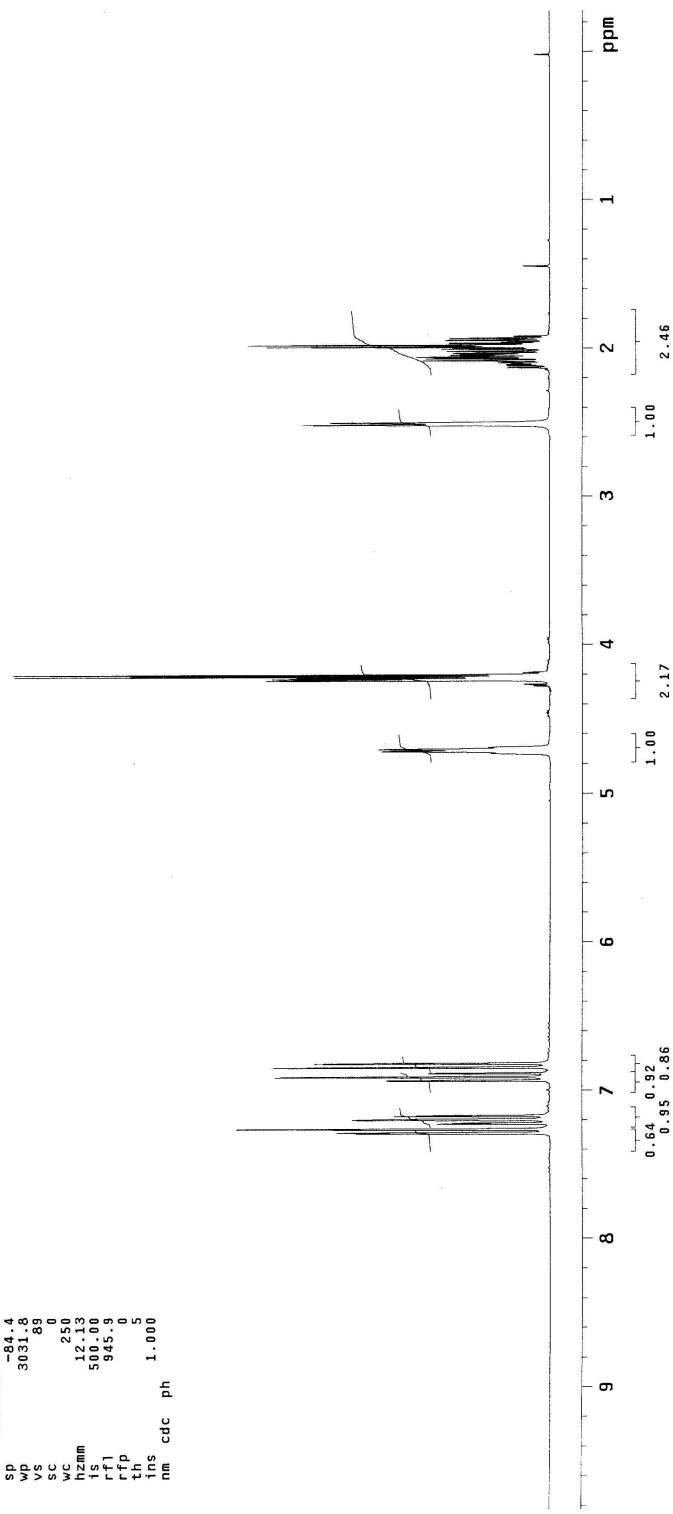
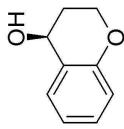


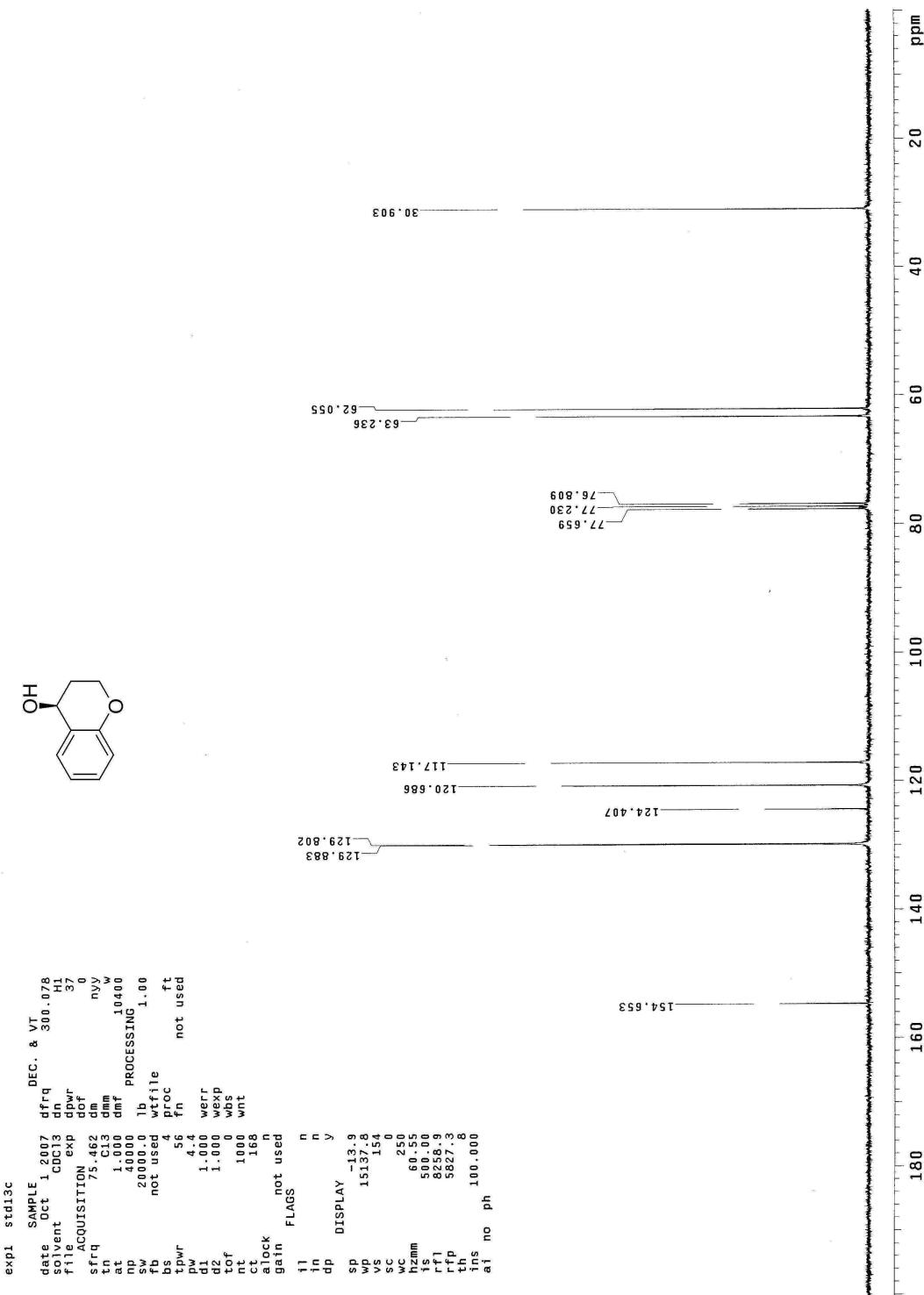




led-VII-140-29

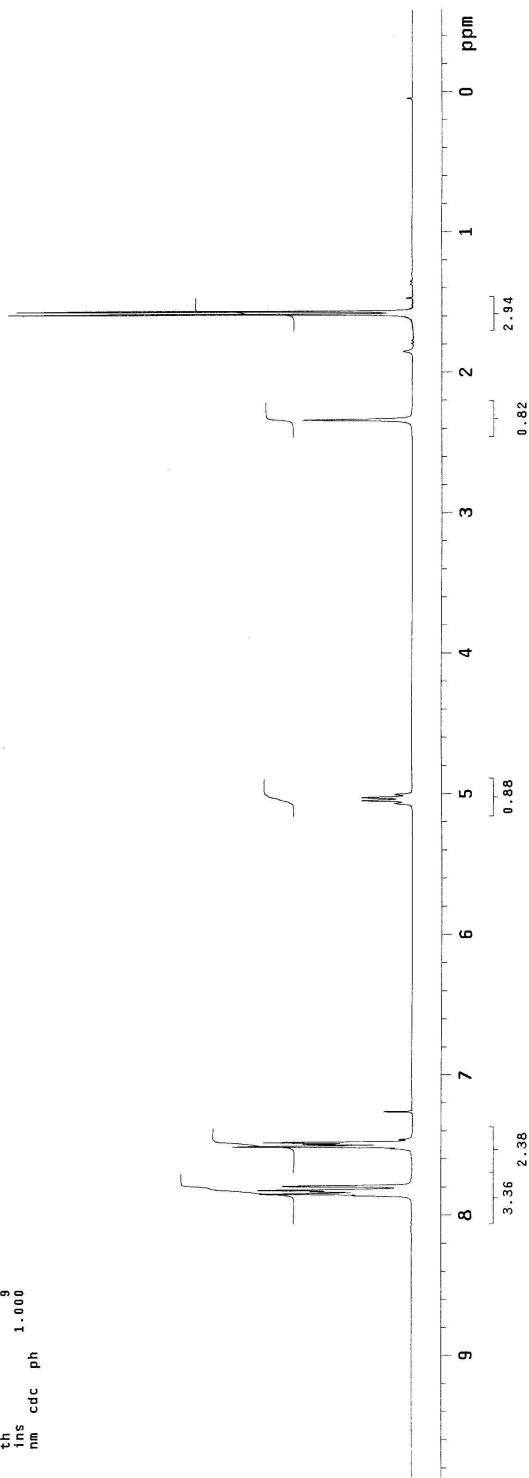
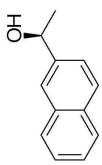
expt stdh
SAMPLE date Oct 1 2007 dfrq 300.078
file C0C13 dn H1
solvent sfrq 300.078 dpwr 30
ACQUISITION exp dof 0
tn 1.998 dmm nnn
at 20000 dmf c
np 5005.0 wtf1le PROCESSING 2.00
sw not used proc ft
fb not used fnc not used
bs tppwr 58
pw 7.2 werr
d1 2.000 wexp
t0f 32 wbs
nt 32 wnt
ct 24
alock n
gain not used
FLAGS
11 in n
in n
dp DISPLAY y
sp -84.4
vp 303.8
vs 8
sc 250
wc 12.13
Hzmm 500.00
r1 944.9
rfp 0
th 5
t1s 1.000
nm cdc ph





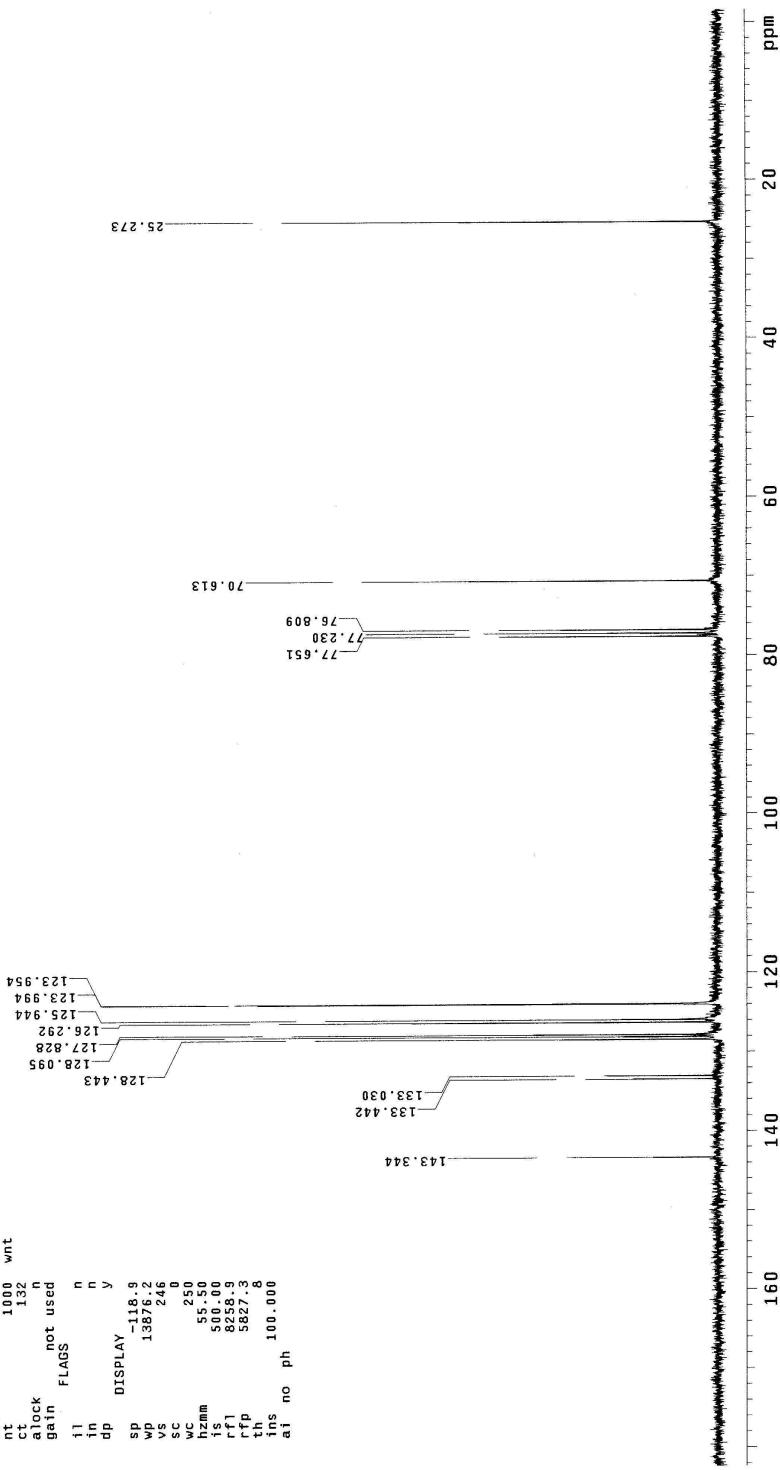
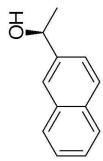
led-VII-1412-29

expi	stdlh
SAMPLE	DEC. & VT
date	Oct 1 2007
solvent	CDC ₃
file	ofrq
ACQUISITION	dtf
sfrq	300.078
tn	0
at	H1
np	1.998
sw	dmm
fb	20000
bs	5000.0
tpw	wf file
pw	not used
d1	4
tr	58
nt	7.2
ct	2.00
a	0
lock	32
gain	28
FLAGS	n
i1	n
in	n
dp	DISPLAY
sp	176.6
wp	-3135.9
vs	68
sc	250
wc	12.55
hz/mm	733.7
rs	945.9
rfp	0
th	9
nm	1.000
cdc	ph



led-VII-142-29-C13

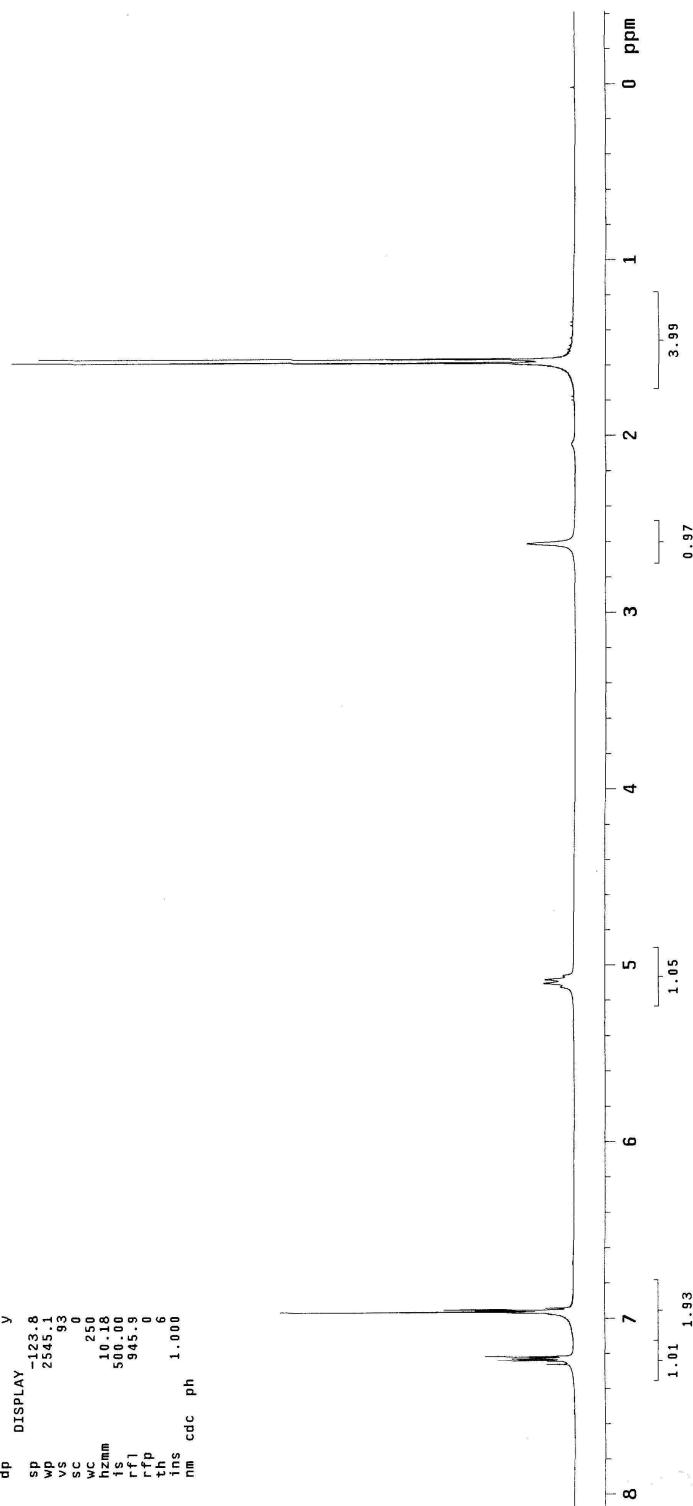
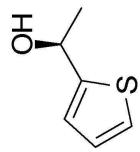
exp1	std3c		DEC.	&	VT
SAMPLE 1		date Oct 1, 2007	dfrq		300.078
	COC13	dn	dn	H1	
	solvent				37
	file		dprv		0
	exp		dof		nvy
	ACQUISITION		dim		
	sfq	75.462	C13	dmm	w
	tn		dmf		10.00
	np	1.000	PROCESSING		
	sw	40000.0	lb		1.00
	fb	20000.0	wtfile		
	bs	not used	4	proc	ft
	tpwr	56	fn		not used
	pw	4.4			
	d1	1.000	werr		
	d2	1.000	wexp		
	tov	1.000	wbs		
	nt	1000.0	wnt		
	ct	1.32			
	clock		h		
	gain		not used		
	FLASH				
	in	11	n		
	dp	n	n		
	DISPLAY		y		
	sp	-118.9			
	wp	13876.2			
	sc	246			
	wc				
	hzmm	250			
	is	55.000			
	rf1	500.000			
	rf2	1258.9			
	rf3	5827.3			
	th	8			
	ins				
	ai	100.000			
	rh				

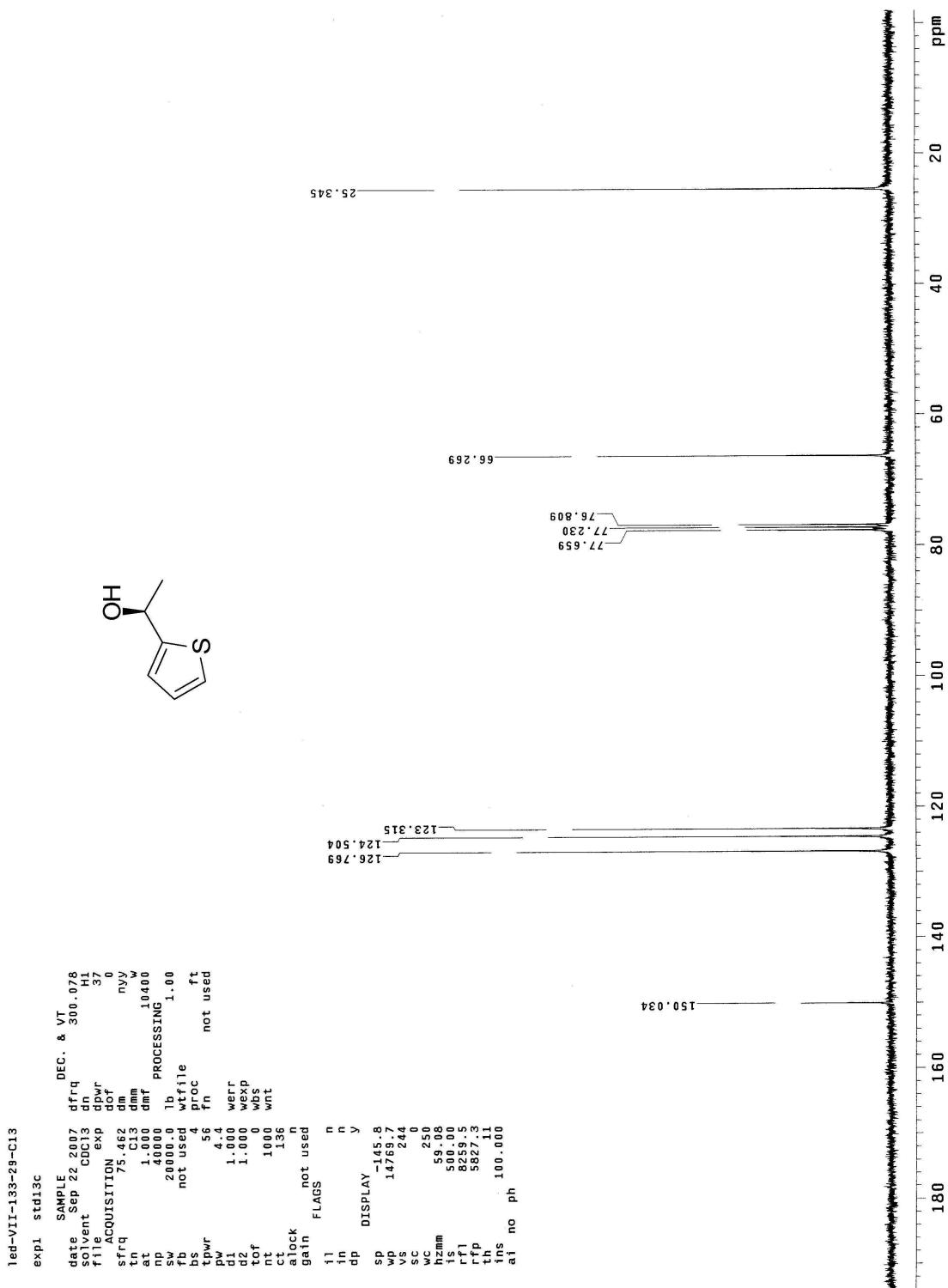


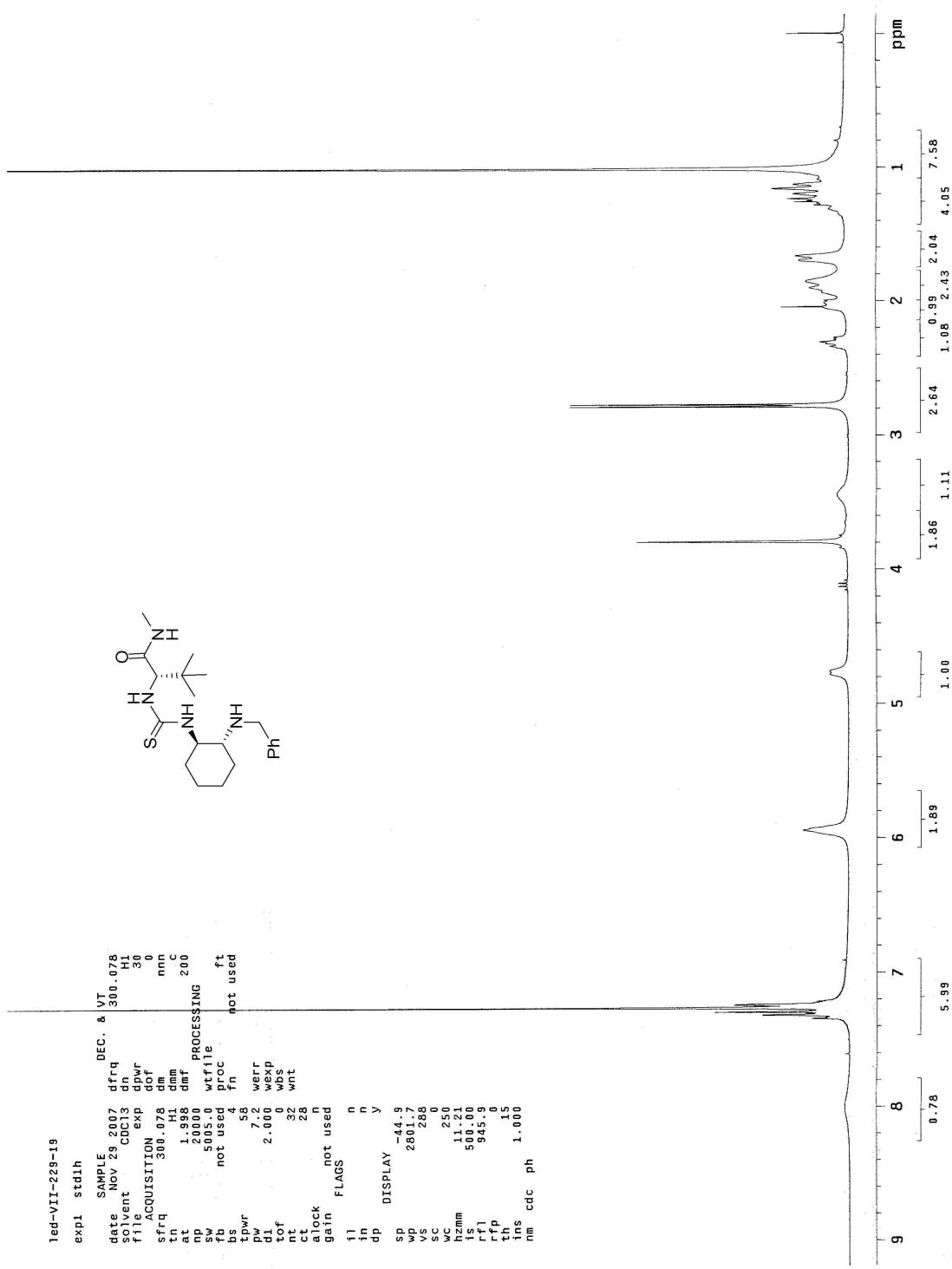
led-VII-133-29

expt stdth

SAMPLE	dfrq	DEC. & VT
date Sep 22 2007	CDC13	300.078
solvent	din	H1
file	exp	30
sfrq	dfrw	0
ACQUISITION	dof	nnn
sfrq	din	c
trn	H1	200
at	dmm	
1.998	dmf	
np	PROCCESSING	
sw	5005.0	
fb	wfile	
not used	proc	
bs	ft	
fn	not used	
trwv	58	
pw	7.2	werr
di	2.000	wexp
tcf	0	wbs
nt	32	wnt
ct	20	
alock		
gain	n	
not used		
1)	FLAGS	
in	n	
dp	n	
DISPLAY	y	
SP	-123.8	
wp	2545.1	
wp	93	
sc	0	
sc	250	
hc	10.18	
hs	500.00	
r1	945.9	
rf1	0	
fp	6	
th	1.000	
ns		
nm		
cdc		
ph		





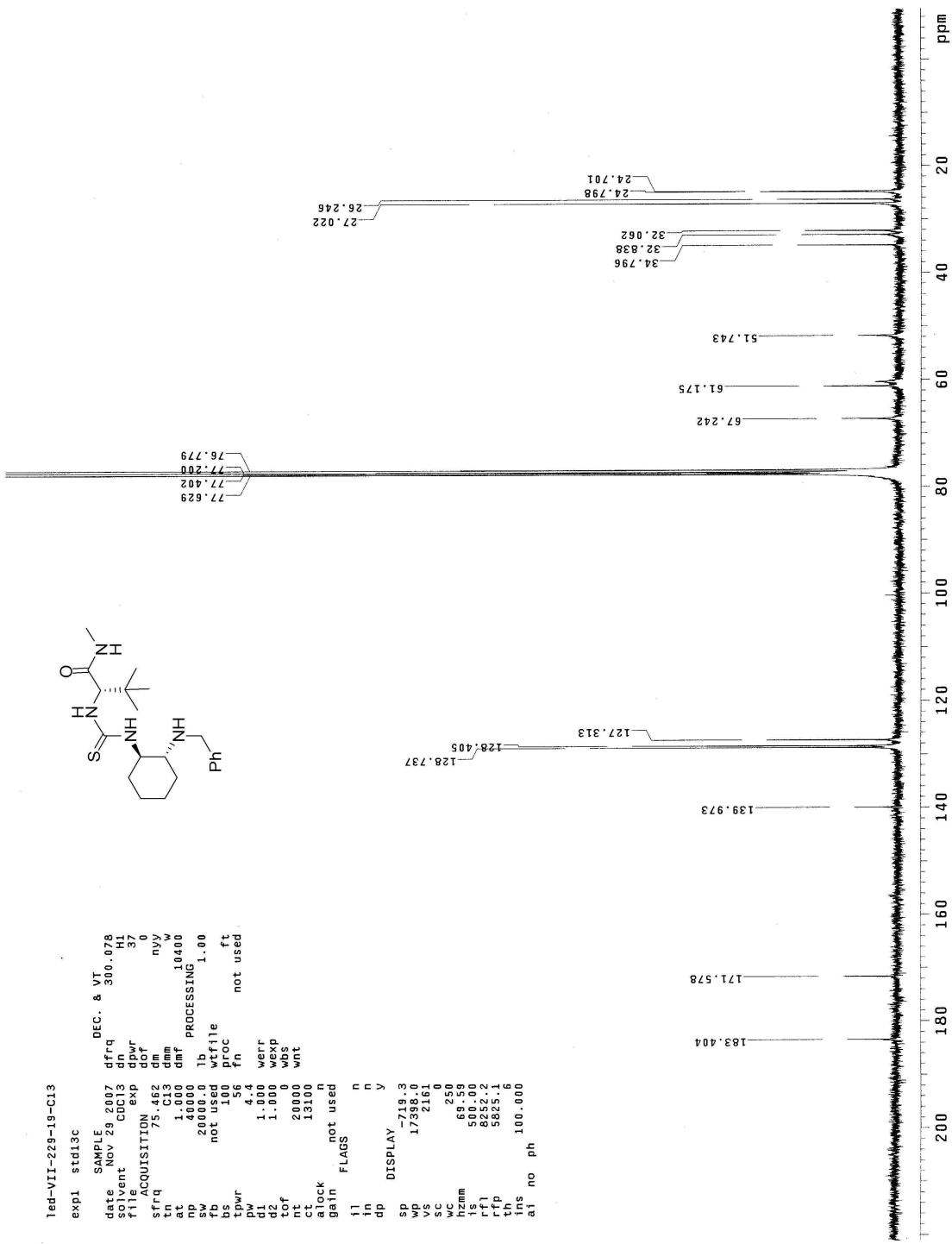
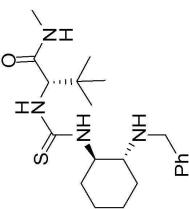


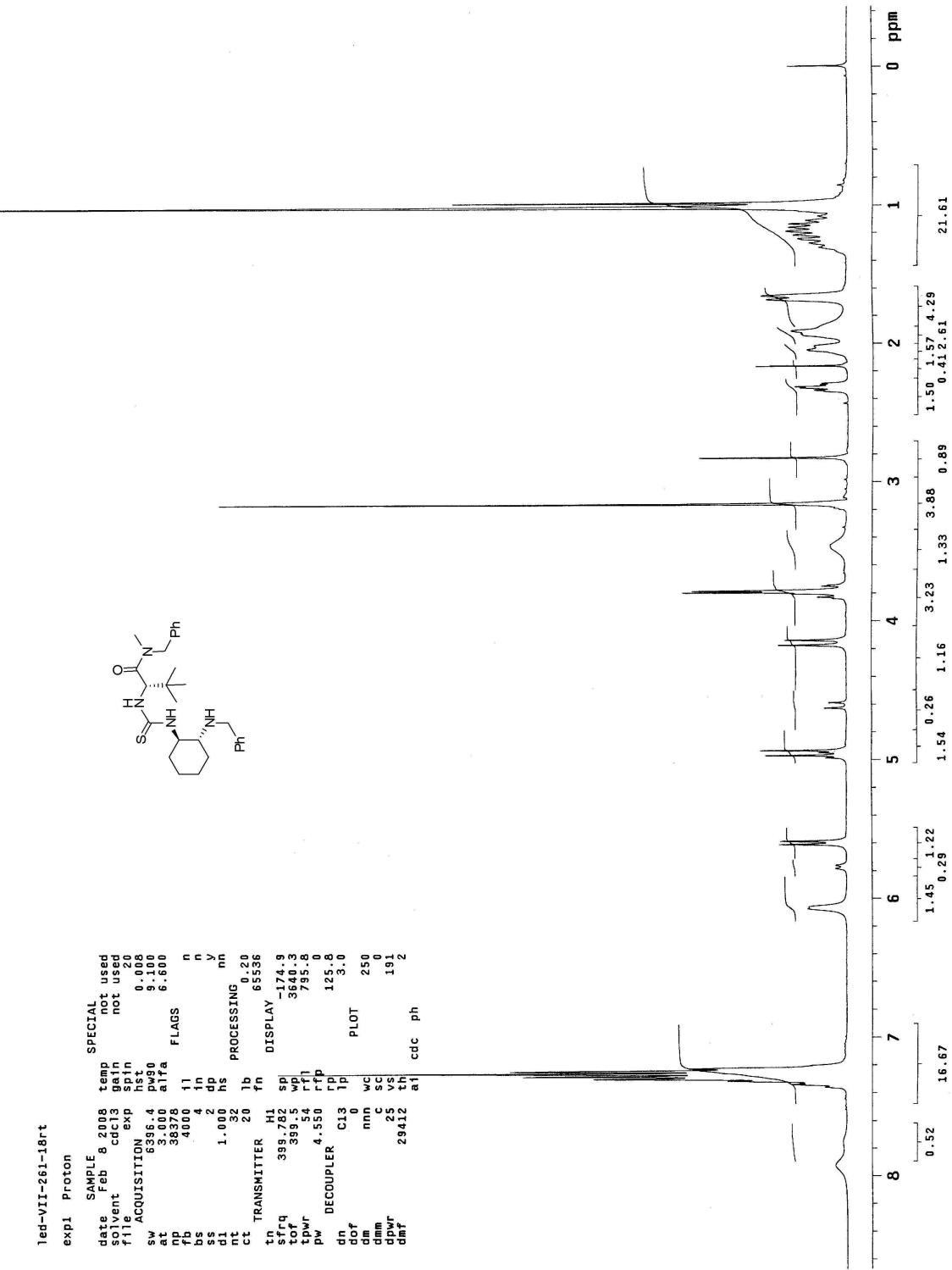
```

led-VII-229-15-C13
exp1 std13c

SAMPLE date Nov 29 2007
          solvent CDC13
          file exp
ACQUISITION exp
           sfrq 71.62
           tn C13
           at 1.000
           np 40000
           sw 20000.0
           fs not used
           fb 100
           pw 5.6
           pw 4.4
           d1 1.000
           d2 1.000
           tof 0
           nt 20000
           ct 131.00
           alock n
           gain not used
           flags n
           il n
           in n
           dp y
DISPLAY sp -719.3
           wp vs 2161.0
           sc wc 1798.0
           hznm is 69.59
           rrf1 rp 8525.2
           rrpf 6

```



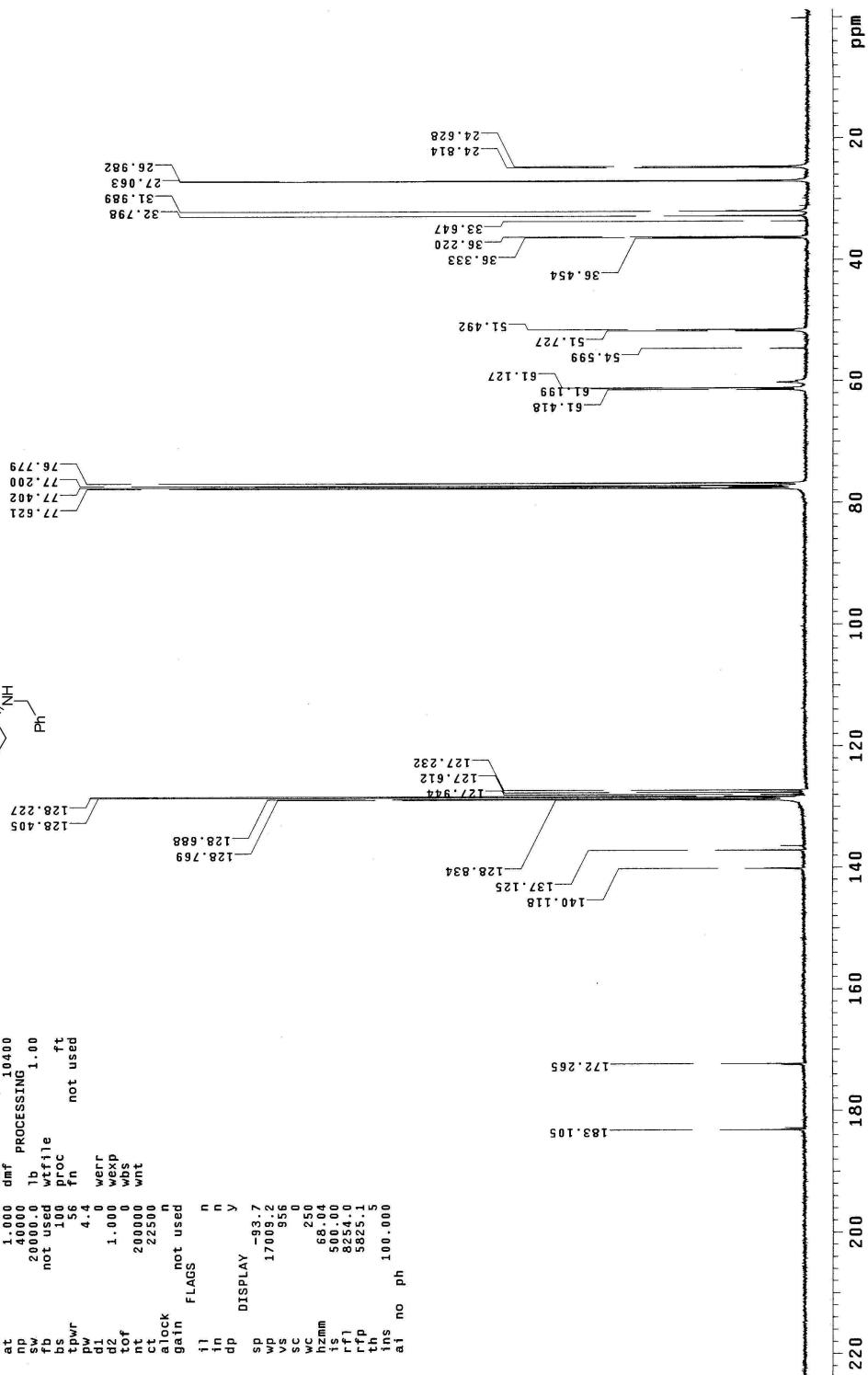
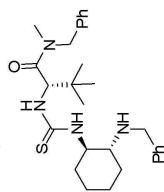


led-VII-261-18

```

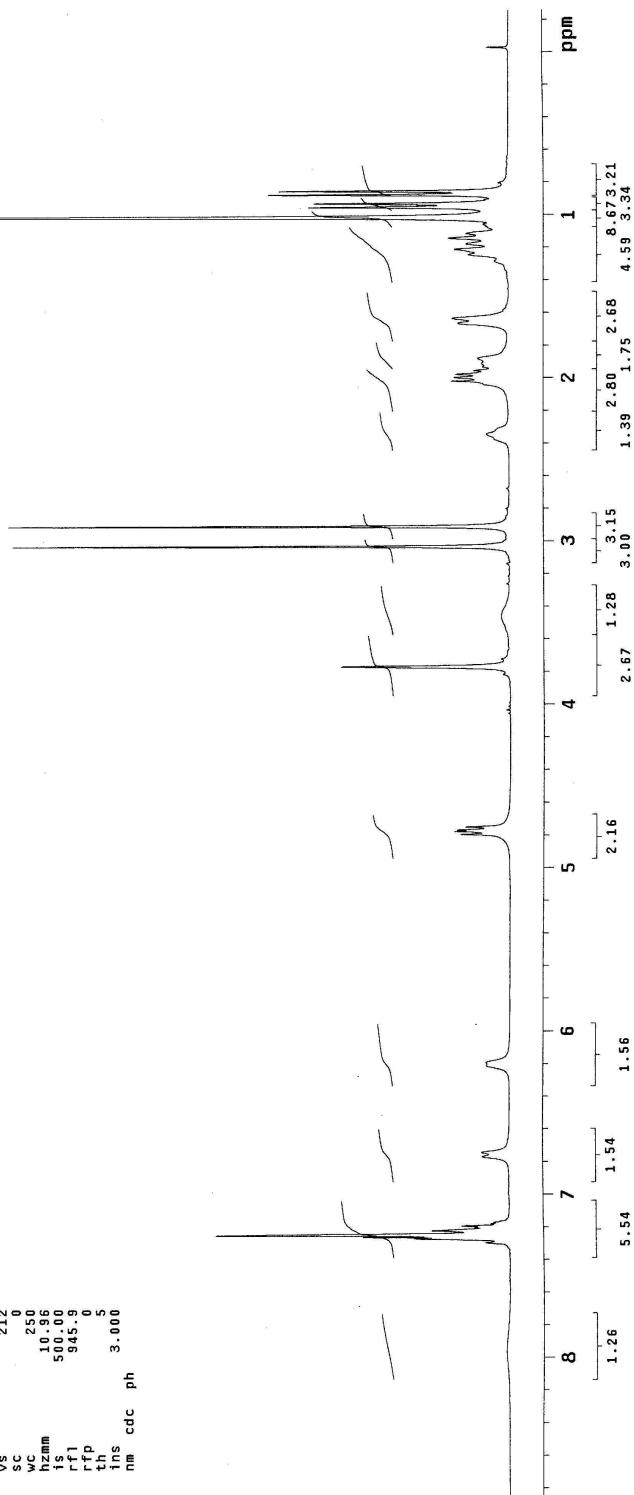
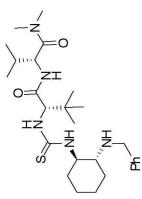
expt  std13c
      SAMPLE          DEC. & VT
      date  Feb 13 2008    dfrq   300.078
      solvent   CDCl3    dn     H1
      file    exp      dpr
      ACQUISITION  exp      dof   0
      sfrq    75.462    dmm   nyy
      tn      C13      dmf   w
      at      1.000    dmf  10400
      np      40000    lb  PROCESSING 1.00
      sw      20000.0   not used
      fb      1b       wfile
      bs      100      proc
      tpwr   56       fn   not used
      pw      4.4      werr
      d1      0
      d2      1.000    wexp
      tof      0       wbs
      nt      200000  wnt
      ct      22500
      alock   not used
      gain    FLAGS
      i1      n
      in      n
      dp      DISPLAY
      sp      -93.7
      sd      17009.2
      wp      vs     95.6
      sc      sc      0
      wc      wc      250
      hzmm   is      68.04
      is      500.00
      rrf1   8234.0
      rfp    5825.1
      th      ins    100.000
      ai      no     ph

```



led-VII-27-18

expt	stdth
SAMPLE	9 2008
date	Feb 9 2008
file	CDCl ₃
solvent	exp
ACQUISITION	dec. & vt
sfreq	300.078
tn	30
at	H1
pw	1.598
np	20000
tdw	500.50
fb	wf file
bs	4
tppr	not used
pw	58
d1	proc
tr	7.2
nt	2.000
ct	werr
dt	0
nt	32
ct	wexp
lock	32
gain	0
FLAGS	not used
l1	n
in	n
dp	y
DISPLAY	-77.9
sp	2740.3
wp	212
sc	0
wc	250
hzam	10.16
is	500.00
rfl	945.9
rfp	0
th	5
ins	3.000
nm	cdc ph

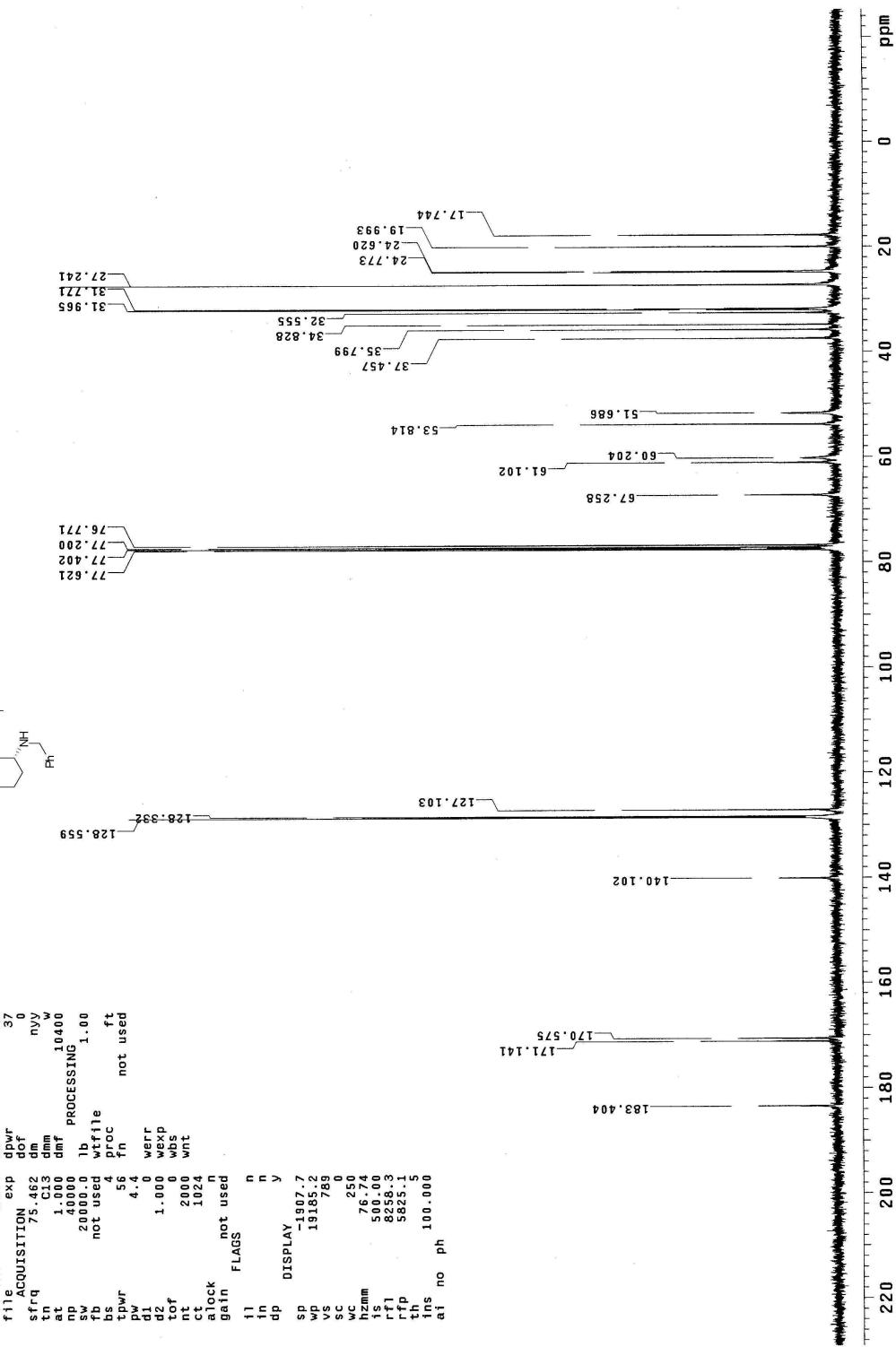
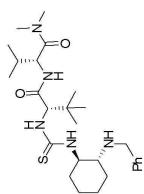


led-vII-271-18-C13

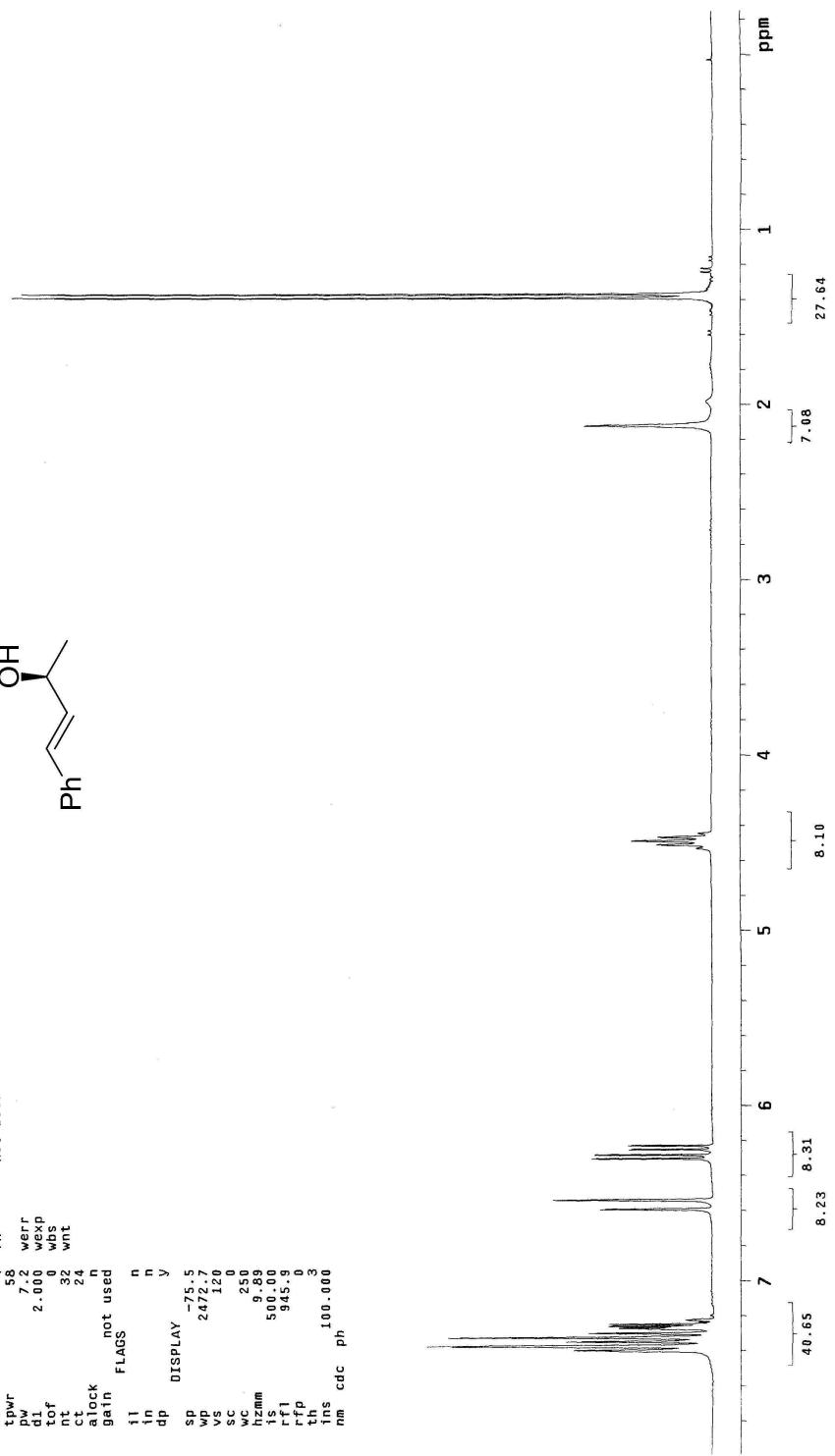
```

expt1 std13c
      SAMPLE          DEC. & VT
      date   Feb 9 2008 dfrq 300.076
      solvent    CCl4            H1
      file       EXP             37
      dphr        0
      dof         0
      dmw        m
      ACQUISITION 75.462
      sfrq        0.000
      tn          1.000
      dmf        0.0400
      PROCESSING 1.000
      at          4.000
      nw          2000.0
      lb          1.000
      not used
      wfile        proc
      not used
      fn          f
      pn          ft
      not used

      tpwrf      4.4
      pwrf        0.0
      wexp        0.0
      wbs         0.0
      wnt         0.0
      bs          4
      ct          1024
      b1          n
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      b1441       5
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      b
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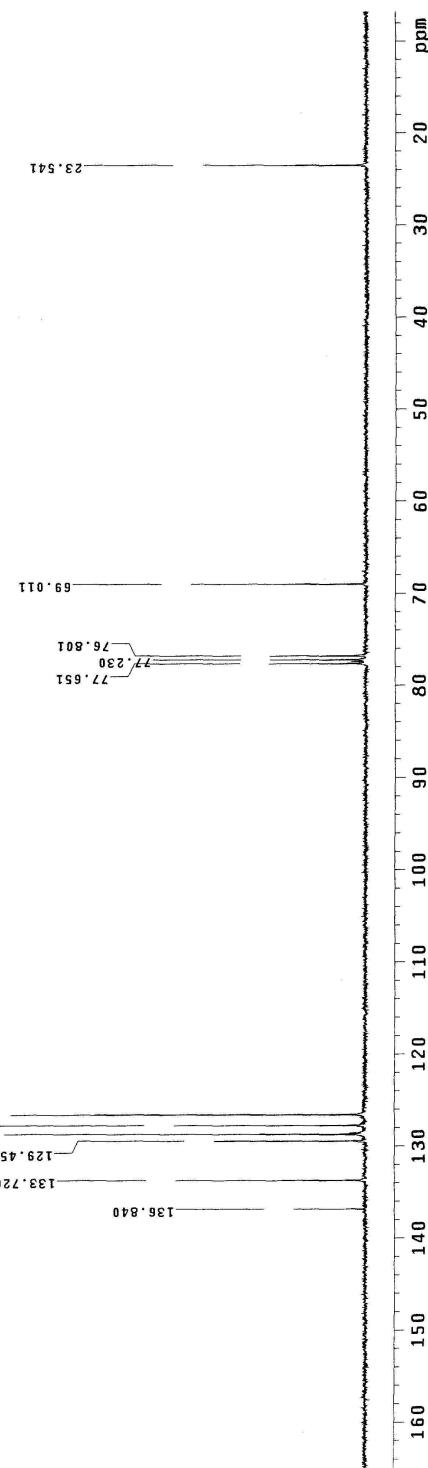
led-VII-103-r
 expt stdth
 SAMPLE Sep 14 2007 DEC. & VT
 solvent CDCl₃ dfrq 300.078 H1
 file d1n 30
 ACQUISITION exp d1w 0
 sfrq 300.078 d1r nnn
 tn 1.078 d1m c
 at 1.998 d1m
 np 2.000 PROCESSING 200
 sw 5.05.0 wf file
 fb not used proc ft
 bs not used fm not used
 tpr 5.8
 pw 7.2 werr
 dl 2.00 wexp
 tor 0 whs
 nt 32 wht
 ct 24
 a/clock n
 gain n
 flags n
 i1 n
 in n
 dp n
 DISPLAY y
 Sp -75.5
 wp 2472.7
 ys 120
 sc 0
 wc 250
 hzmm 9.89
 is 500.0
 rrf1 945.9
 rfp 0
 th 3
 ins 100.000
 nm cdc ph

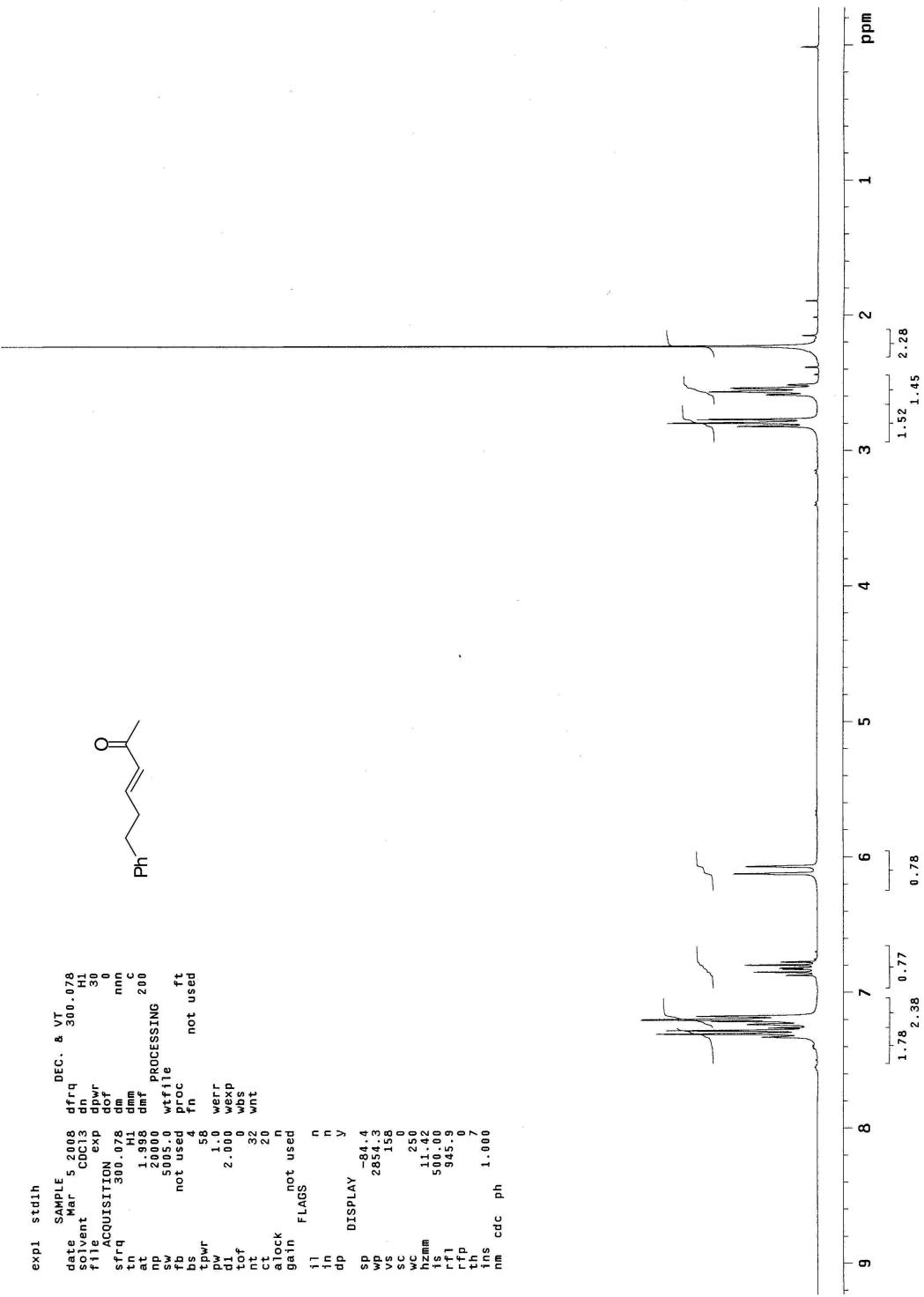


led-VII-100-r-C13

expt std13c

SAMPLE DEC. & VT
date Sep 14, 2007 dfrq 300.078
solvent CDCl₃ dn H1
file exp dpr 37
ACQUISITION 75.462 dpr 0
sf,q dpr nyy
tn Cl3 dpr w
at 1.000 dpr 10.000
np 4000 PROCESSING 1.00
sw 2000.0 1b
fb not used
bs 4 wrfile
tpr 56 proc
pw 4.4 ft
dw 1.000 warr
d2 1.000 wexp
t0f 0.0 whs
nt 1000 wnt
ct 112
alock n
gain not used
FLAGS
i1 n
in n
dp y
DISPLAY
sp 511.6
wp 11931.5
vs 87
sc 0
wc 250
hzmm 47.73
is 500.00
rf1 8258.9
rfp 5827.3
th 5
ins 100.000
ai no ph



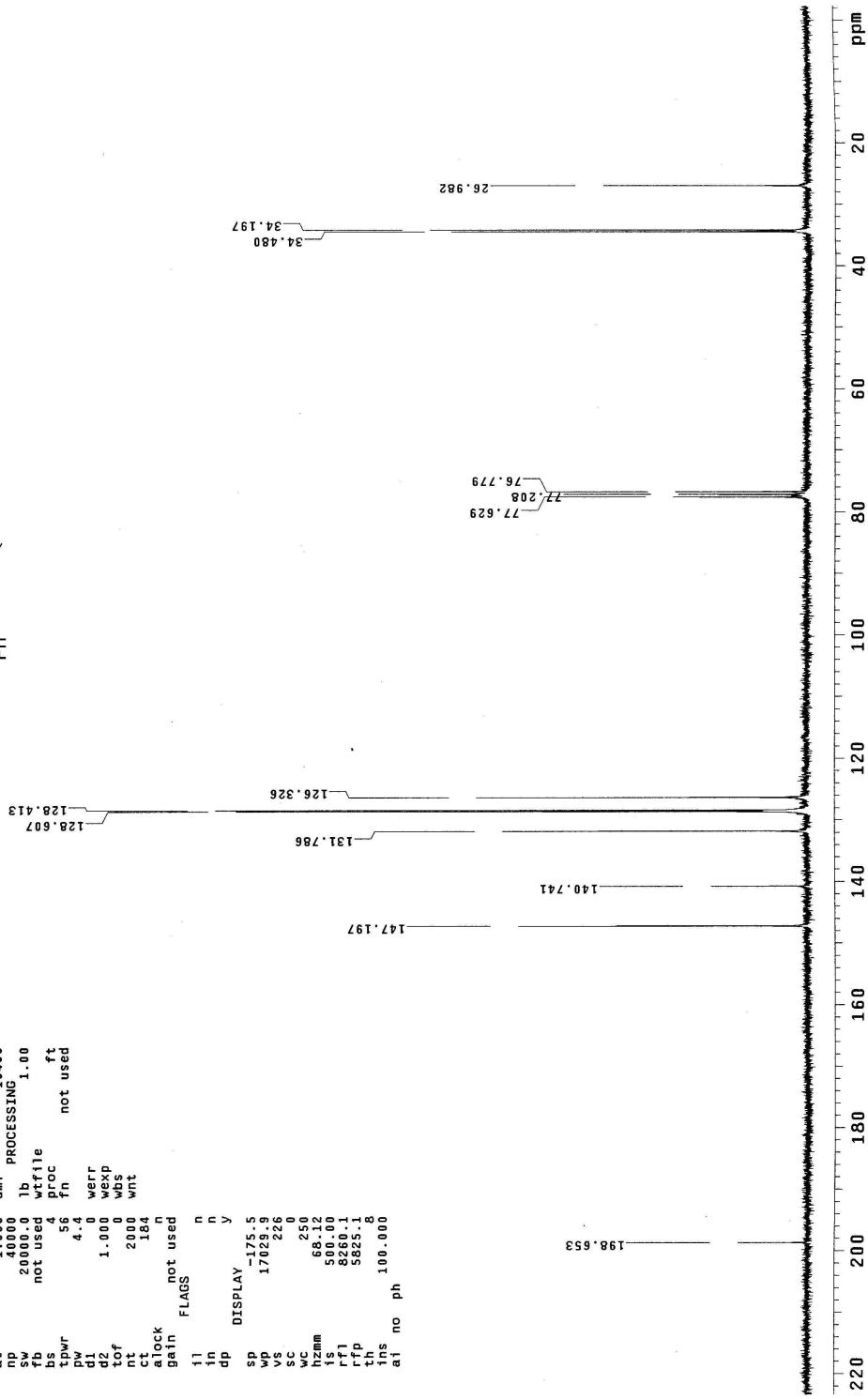


1ed-VII-153-18-C13

```

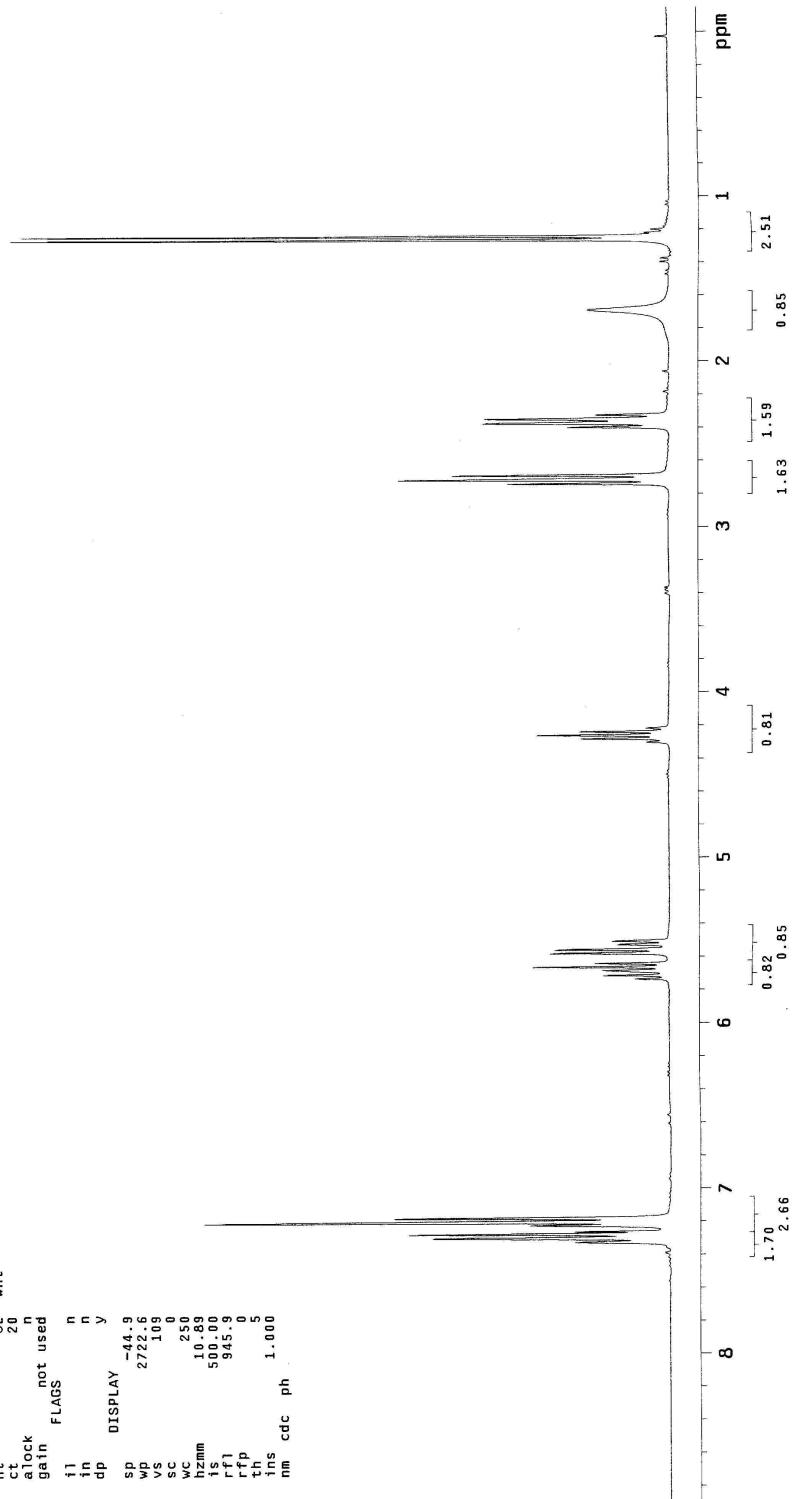
exp1 std13c
      SAMPLE   DEC. & VT
      date Mar 5 2008 dfrq 300.078
      solvent CDCl3 dn H1
      file      exp dpvr 37
      ACQUISITION dof 0
      sfrq 75.462 dim nyw
      tn      C13 w
      at      1.000 dmf 10400
      np      40000 PROCESSING 1.0400
      sw      20000.0 lb 1.00
      fb      not used wfile 1.00
      bs      4 proc ft
      tppr 56 fn not used
      pw      4.4 werr
      d1      0
      d2      1.000 wexp
      tof     0 wbs
      rt      2000 wnt
      ct      184
      alock
      gain  not used
      flags
      11    n
      in    n
      dp    DISPLAY y
      SP    -175.5
      wp    17029.9
      vs    226
      sc    0
      wc    250
      Hzmm 68.12
      fs    500.00
      rrf1 8200.00
      rfp  5825.1
      th    100.000
      ai    no ph

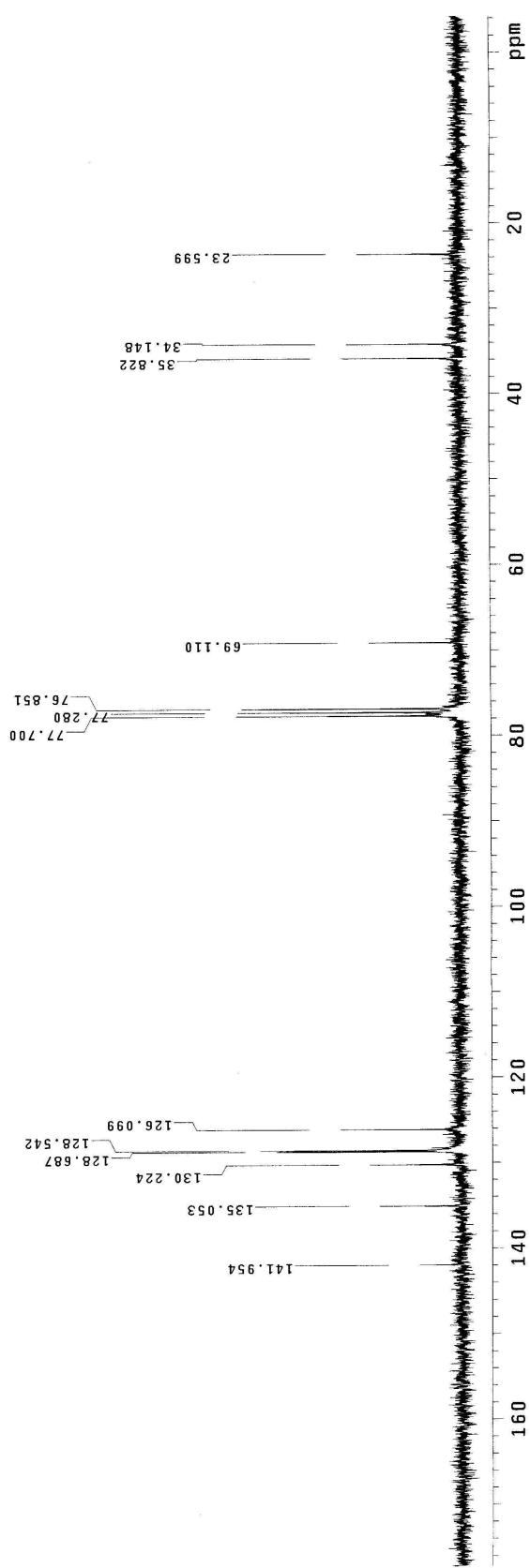
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led-VII-154

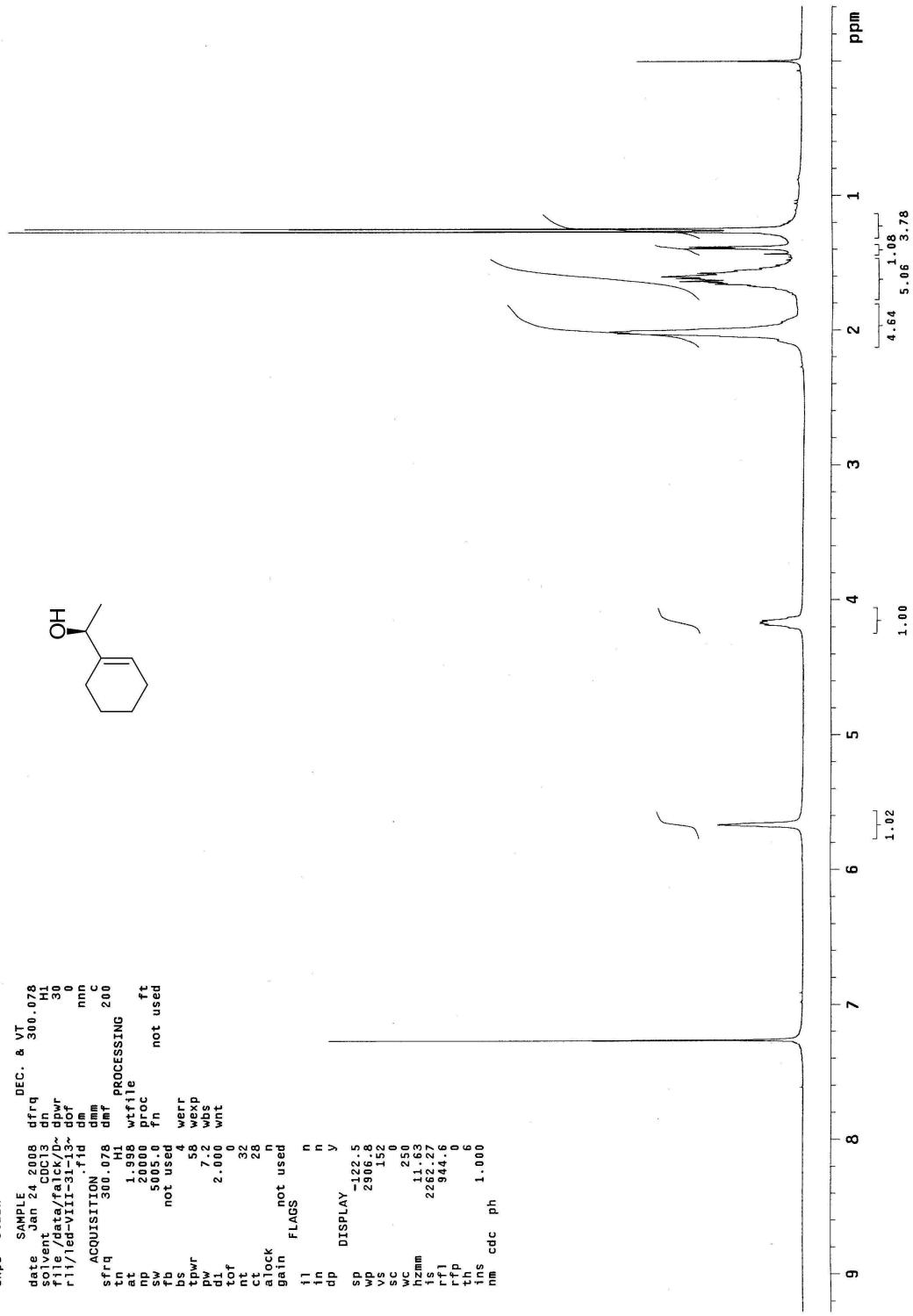
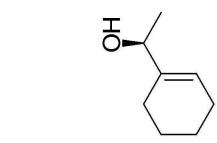
```
exp1 std1h
SAMPLE 8-2007 dfrq 300.078
date Oct 8-2007 DEC. & VT
sovent CC13 dn H
rfexp dfrq 300.078 dn H
rfw dfr 300.078 dn H
ACQUISITION exp dfr 300.078 dn H
sf1q 300.078 dm 0
tn 1.998 dm 0
at 2.000 dm 0
rp 2.000 fm 0
sw 5015.0 wtf11e PROCESSING 200
sb 5015.0 not used
fb not used proc not used
ts 4 fm not used
tpwr 58 werr
fw 1.0 werr
d1 2.000 wexp
tref 0 wbs
rt 32 wnt
ct 20
alock n
gain not used
flags
f1 n
fin n
cp y
DISPLAY -44.9
sp 2722.6
vp 2722.6
vs 109
sc 0
vc 250
hzmin 10.89
ts 500.00
rf1 945.9
rfp 0
fh 5
ins 1.000
nm cdc ph 1.000
```





led-VIII-31-13

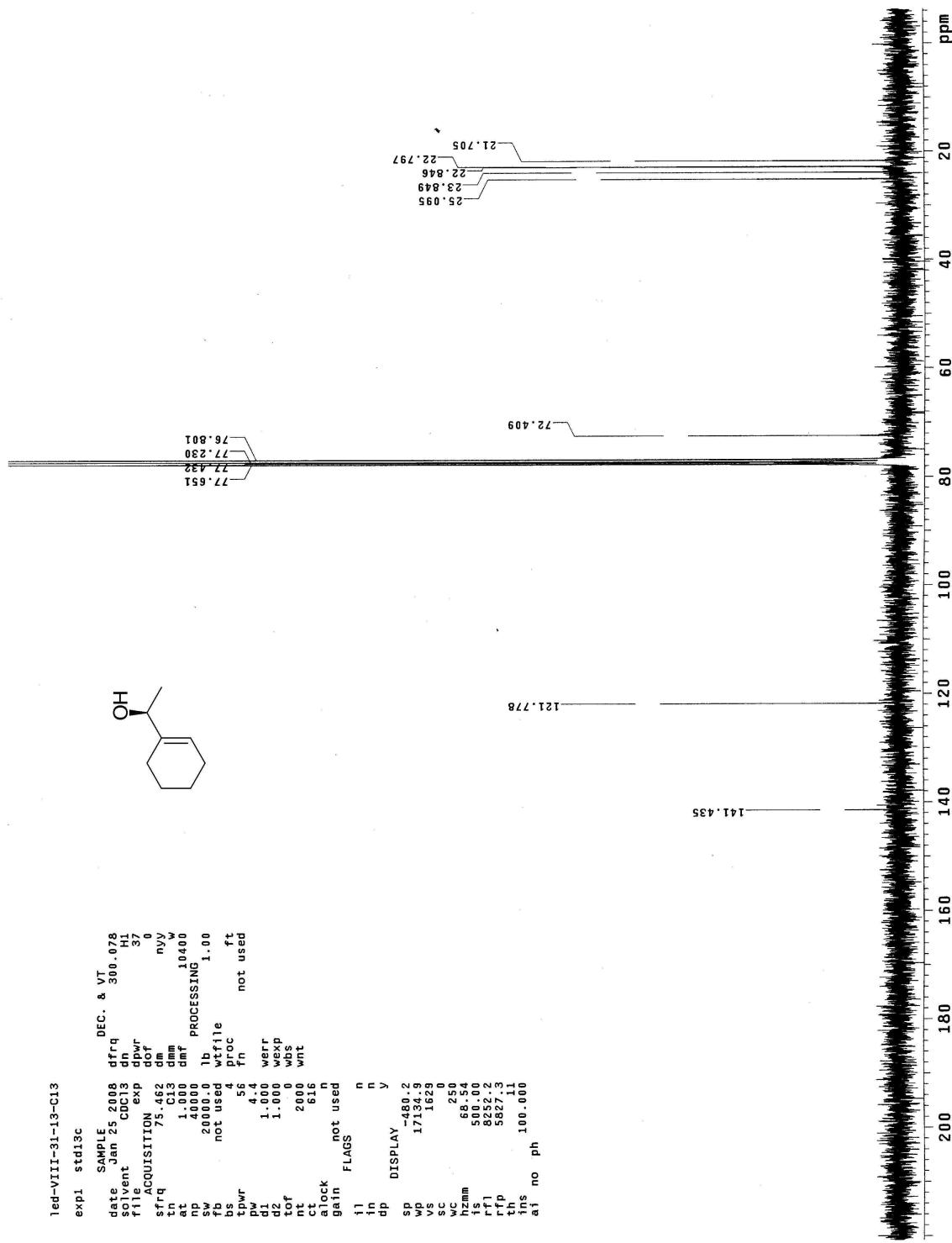
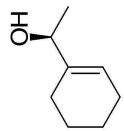
exp1 stdih
SAMPLE dfrq DEC. & VT
date Jan 24 2008 300.078
solvent CDCl₃ H1
file /date/fdrck/D-pwr
r11/led-VIII-31-13-
of
ACQUISITION .fid dn
dmm mn
dmf c
sfrq 300.078 PROCESSING 2.00
tn tn wtfile
at 1.998
np 20000 ft
sw 500.0 proc
fb not used fn not used
bs 4 werr
tppw 58 wexp
pw 7.2 wbs
d1 2.000 wnt
t0f nt
rt 32
ctt 28
clock n
gain not used n
FLAGS



```

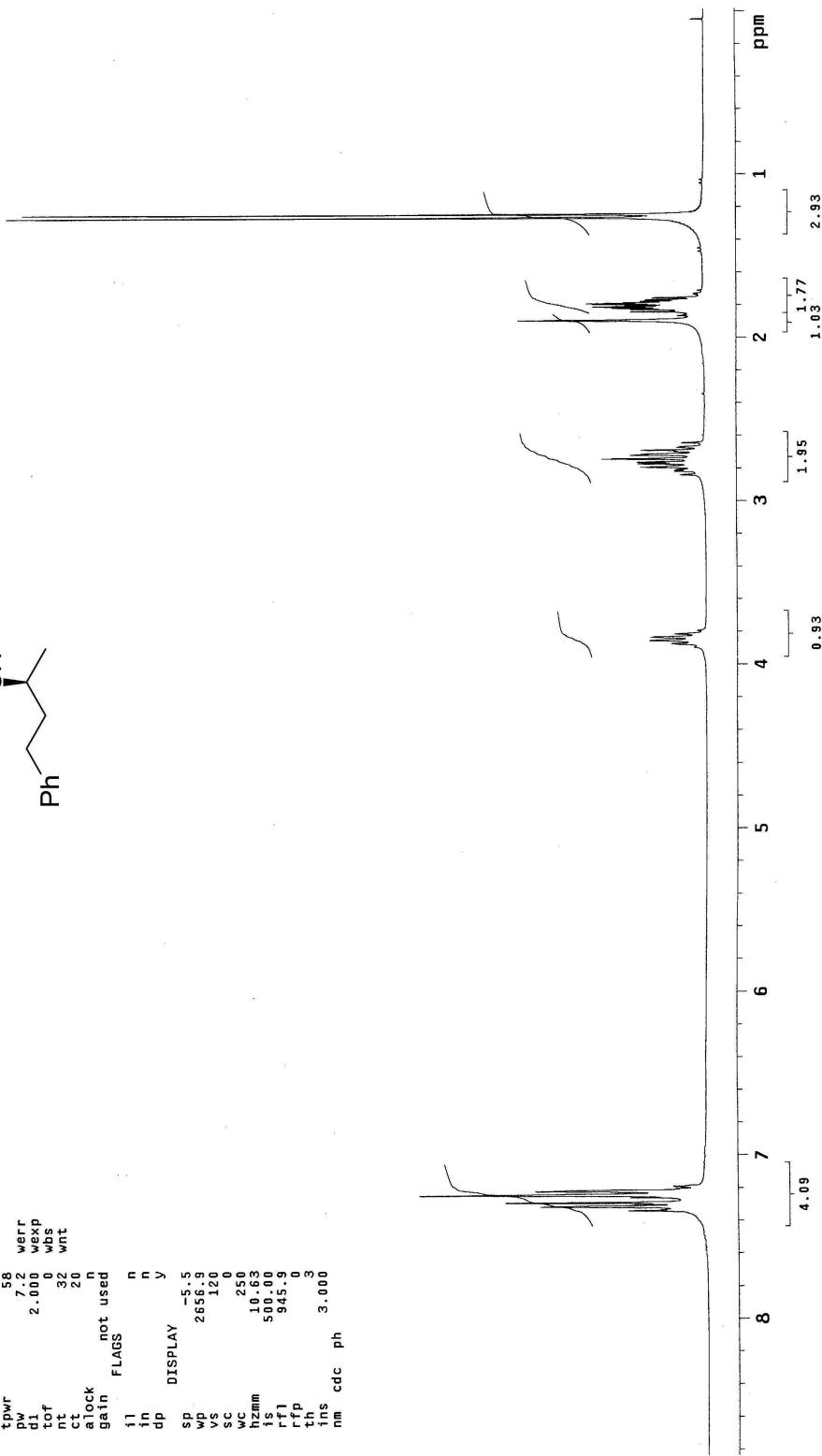
1ed-VIII-31-13-C13
exp1 std13c
      SAMPLE   VT
      date Jan 25 300 .07-H
      solvent CDC13 3
      exp dprw n
      srfrq 75.4612 nuy
      tnm 1.0000 PROCESSING 10400
      atp 0.0000 lb 1.0000
      sp 2000.00 wtrfile
      not used
      bs 4 proc
      tpwr 56 not used
      pw 4
      dd 1.000 wer
      dd 1.000 wexp
      tor 0 wbs
      nt 2000.0 wnt
      ct 616
      alock n
      gain not used
      FLAGS
      in n
      in n
      dp y
      DISPLAY
      sp -1480.2
      sp 17134.9
      vs 1625.0
      sc 0
      wc 250.0
      hzmm 68.54
      is 500.00
      rfp 852.2
      rfp 5827.3
      th 11
      ins 100.000
      ai no ph

```



led-VII-97-18

```
expl stdlh
      SAMPLE   DEC. & VT
      date Feb 9 2008 dfrq 300.078
      solvent CDCl3 dn H1
      file      exp dpwr 30
      ACQUISITION dof 0
      streq 300.078 dm nnn
      tn      H1 dmm c
      at      1.998 dmf 200
      np      20000 PROCESSING
      sw      5005.0 wtime
      fb      not used proc ft
      bs      4 fn not used
      tppr    58
      pw      7.2 werr
      d1      2.000 wexp
      tof     0 wbs
      nt      32 wnt
      ct      20
      alock   n
      gain    not used
      i1      n
      in      n
      dp      DISPLAY -5.5
      sp      2656.3
      wp      120
      vs      0
      sc      250
      wc      10.63
      hzmm   500.00
      is      945.9
      rrf1   0
      rfp    3
      th      3
      ins    3.000
      nm cdc ph 3.000
```

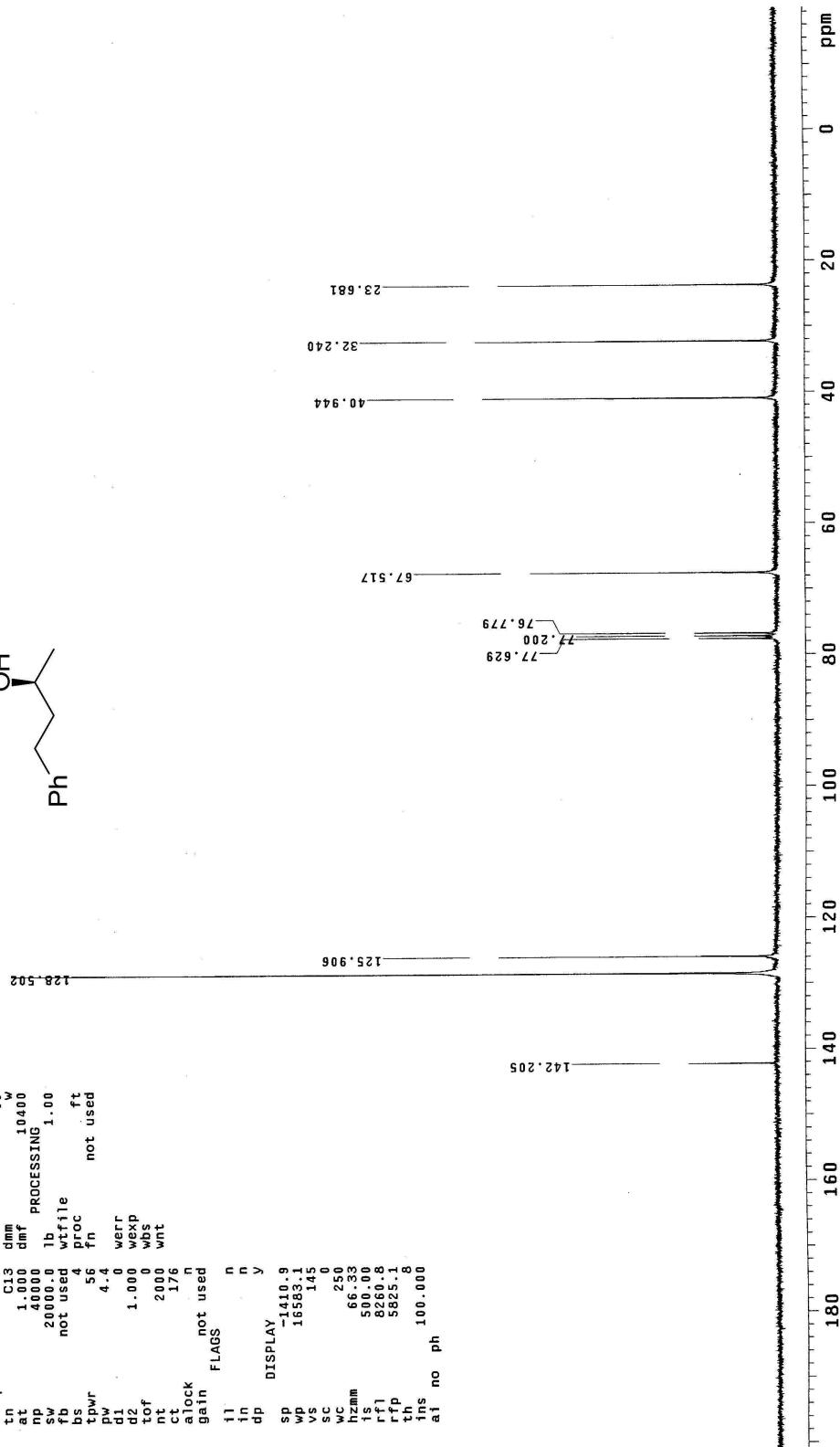
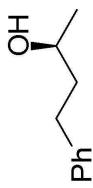


13C OBSERVE

```

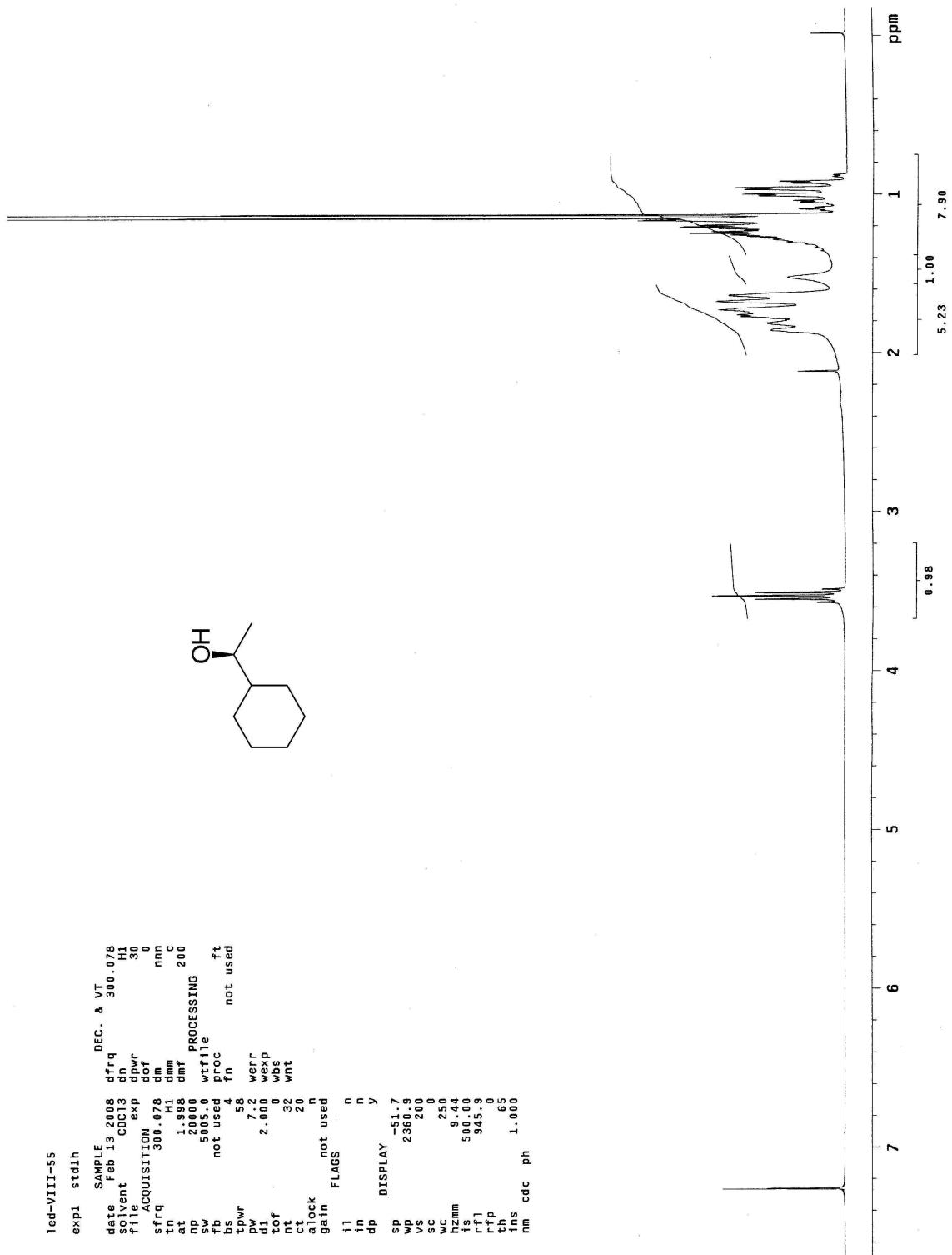
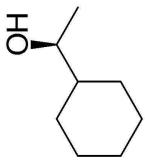
expt std13c
SAMPLE
date Feb 9 2008 dfrq 300.075
solvent CDCl3 dfrq 300.075
file exp dfrq 300.075
ACQUISITION 75.462 dfrq 300.075
sfrq 1.000 dfrq 300.075
tn 40000 dfrq 300.075
np 40000 dfrq 300.075
sw 20000.0 lb 1.00
fb not used wfile 1.00
bs 4 proc ft
tpur 56 fn not used
pw 4.4 werr
d1 1.000 wexp
d2 1.000 wbs
t0f 2000 wnt
nt 176
ct
alock
gain not used
FLAGS
il n
in n
dp y
DISPLAY -1410.9
sp 16583.1
wp 145
vs 0
sc 250
wc 66.33
hzmm 500.0
r1 8260.8
rfp 5822.1
th 100.000
ins no ph
ai no ph

```



led-VIII-55

exp1	stdth	SAMPLE	DEC.	VT
date	Feb 13 2008	dfrq	300.0	0.078
solvent	CDCl ₃	din	H1	
file		exp	30	
sfrq		dprw	0	
ACQUISITION	300.078	dof	nnn	
tn		dim	c	
at	1.998	dmm		
np	200.00	dmf		
sw	5005.0	PROCESSING	200	
fb	not used	w11e		
bs	4	proc		
tpwr	58	fn	ft	
pw	7.2			
d1	2.000	werr		
tof	0	wep		
nt	32	wbs		
ct	20	wnt		
clock				
gain		not used	n	
FLAGS				
i1	n			
in	n			
dp	y			
DISPLAY	-51.7			
sp	2300.9			
wp	200			
vs	0			
sc	250			
wc	9.44			
hzmm	500.00			
is	945.9			
rfl	0			
rfp				
th	65			
ins	1.000			
nm	cdc ph			



led-VIII-55-C13

```
exp1 st13c
      SAMPLE   DEC. & VT
      date    Feb 13 2008 dfrq  300.078
      solvent CDCl3   H1
      file     exp    37
      ACQUISITION dpr
      sfq      exp    0
      th       dmr   nyy
      at       1.000  dmnm w
      np       4000.0 dmf  PROCESSING 1.00
      sw       20000.0 1b
      not used write
      fb       proc   ft
      bs       56    fn  not used
      tpwr   4.4
      pw      0.0
      d1      werr
      d2      1.000 wexp
      t0f     0.0
      nt      1000  wht
      ct      40
      alock   not used
      gain    not used
      flags
      i1      n
      in      n
      dp      y
      DISPLAY
      sp      -141.9
      wp      13087.6
      vs      234
      sc      0
      wc      250
      hzmm   52.35
      1s      500.00
      rfi    8253.4
      rfp    582.1
      ins    14
      ai     no
      no     ph
      100.000
```

