Supporting Information

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

De Run Li, Anyu He and J. R. Falck*

Departments of Biochemistry and Pharmacology, University of Texas Southwestern Medical Center
Dallas, Texas 75390 USA
E-mail: j.falck@utsouthwestern.edu

I. General information

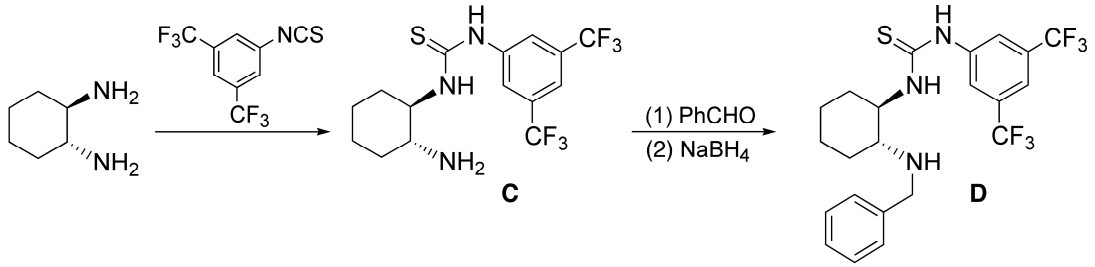
All reactions were maintained under an argon atmosphere. Anhydrous solvents were freshly distilled from sodium benzophenone ketyl, except for CH_2Cl_2, which was distilled from CaH_2. Extracts were dried over anhydrous Na_2SO_4 and then filtered prior to removal of all volatiles under reduced pressure. 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 (^{1}H) or 75/100 MHz (^{13}C). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform δ = 7.26 for ^{1}H NMR or δ = 77.23 for proton decoupled ^{13}C 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 ºC in toluene. Racemic alcohols were prepared by reduction of corresponding commercial ketones with $NaBH_4$ or $NaBH_4/CeCl_3$ (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

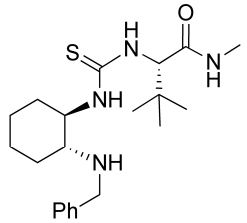


Catalysts C and D. 3,5-Bis(trifluoromethyl)phenyl isothiocyanate (1.3 mL, 7.1 mmol, 1.0 equiv) was added to a rt solution of (*R*,*R*)-cyclohexanediamine (970 mg, 8.51 mmol, 1.2 equiv) in anhydrous dichloromethane (20 mL). After 10 h, the reaction mixture was loaded onto a silica gel column and chromatographed using EtOAc/MeOH/NH_4OH (200:5:1→100:20:1) to give 1-((1*R*,2*R*)-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/NH_4OH, 100:5:1); $[\alpha]^{20}_D$ = +76.9 (*c* 1.7, $CHCl_3$); ^1H NMR ($CDCl_3$, 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, NH_2), 1.98-1.91 (m, 2H), 1.80-1.65 (m, 2H), 1.40-1.20 (m, 4H); ^{13}C NMR ($CDCl_3$, 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 $C_{15}H_{18}N_3SF_6$ $[MH]^+$ *m/z* 386.1125, found 386.1128.

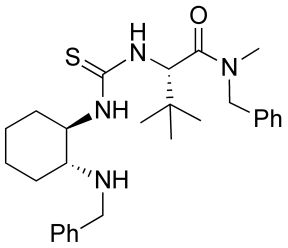
Benzaldehyde (0.578 mL, 5.45 mmol, 1.05 equiv) was added to a rt solution of catalyst **C** (2 g, 5.19 mmol, 1.0 equiv) in anhydrous MeOH (15 mL) under an argon atmosphere. After 4 h, the reaction mixture was cooled to 0 °C and $NaBH_4$ (211 mg, 5.71 mmol, 1.1 equiv) was added in portions. After another 20 min, saturated aq. NH_4Cl solution (50 mL) was added followed by conc. NH_4OH (2 mL). The resulting mixture was stirred for an additional 20 min, then extracted with dichloromethane (100 mL × 5). After drying over Na_2SO_4, the combined organic extracts were evaporated to dryness. The residue was purified by flash chromatography on silica gel using hexanes/EtOAc/MeOH/NH_4OH (400:100:5:1) to afford 1-((1*R*,2*R*)-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/CH_2Cl_2 as a white solid (75%), mp 140 °C-141 °C. TLC: R_f ~ 0.48 (CH_2Cl_2/MeOH, 9:1); ^1H NMR ($CDCl_3$, 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_{CF} ~ 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 $C_{22}H_{24}N_3SF_6$ $[MH]^+$ *m/z* 476.1595, found 476.1598.

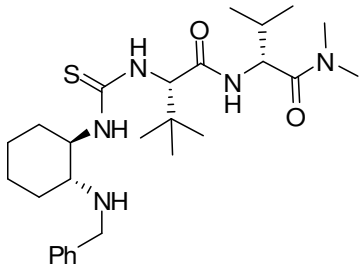


Catalyst G. Following the general procedure used to synthesize catalyst **D**, (*S*)-2-isothiocyanato-*N*,3,3-trimethylbutanamide was used to prepare catalyst **G** (54%). $[\alpha]^{20}_D$ = +54.9 (*c* 1.30, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.99 (br s, 1H), 7.39-7.20 (m, 5H), 5.94 (br s, 2H), 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 $C_{21}H_{35}N_4OS$ $[MH]^+$ *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, $CHCl_3$); ^{1}H 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

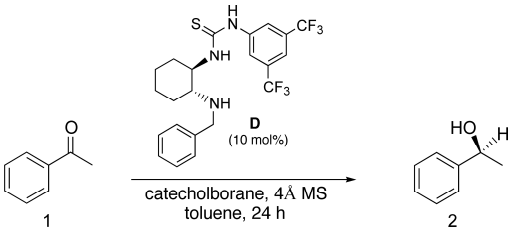
NMR ($CDCl_3$, 75 MHz) δ 183.1, 172.2, 140.1, 137.1, 128.7 (2C), 128.6 (2C), 128.4 (4C), 127.6, 127.2, 61.4, 61.1, 60.2, 51.7, 51.4, 36.3, 36.2, 32.7, 31.9, 26.9 (3C), 24.8, 24.6; HRMS (FAB, NBA) Calcd. for $C_{28}H_{41}N_4OS$ $[MH]^+$ *m/z* 481.3001, found 481.2998.



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, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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), 094 (d, *J* = 7.2 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 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 $C_{27}H_{46}N_5O_2S$ $[MH]^+$ *m/z* 504.3372, found 504.3374.

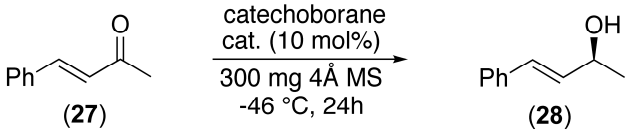
III. Reaction parameter optimization

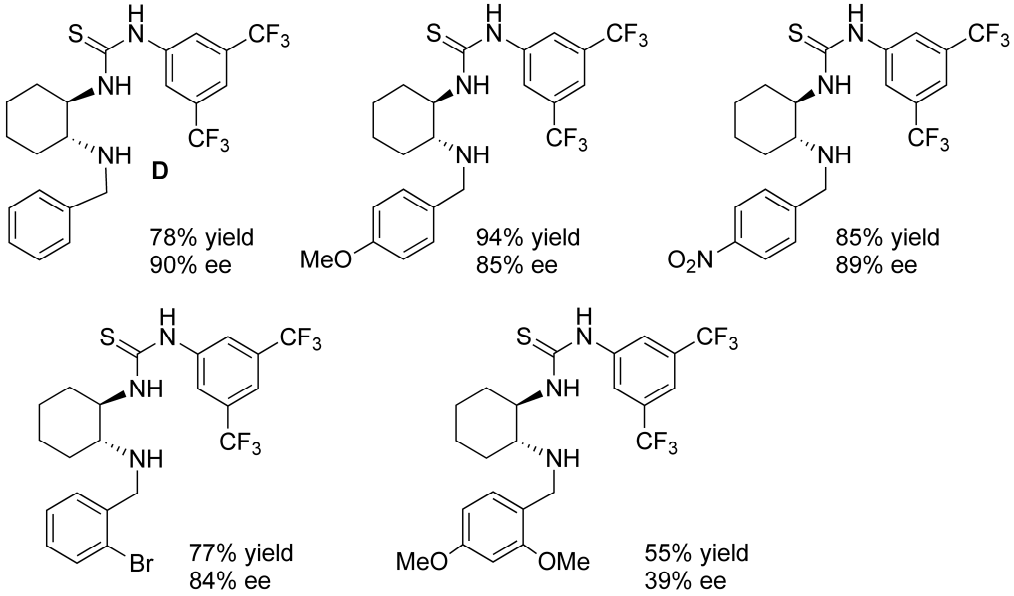
a. Temperature dependence



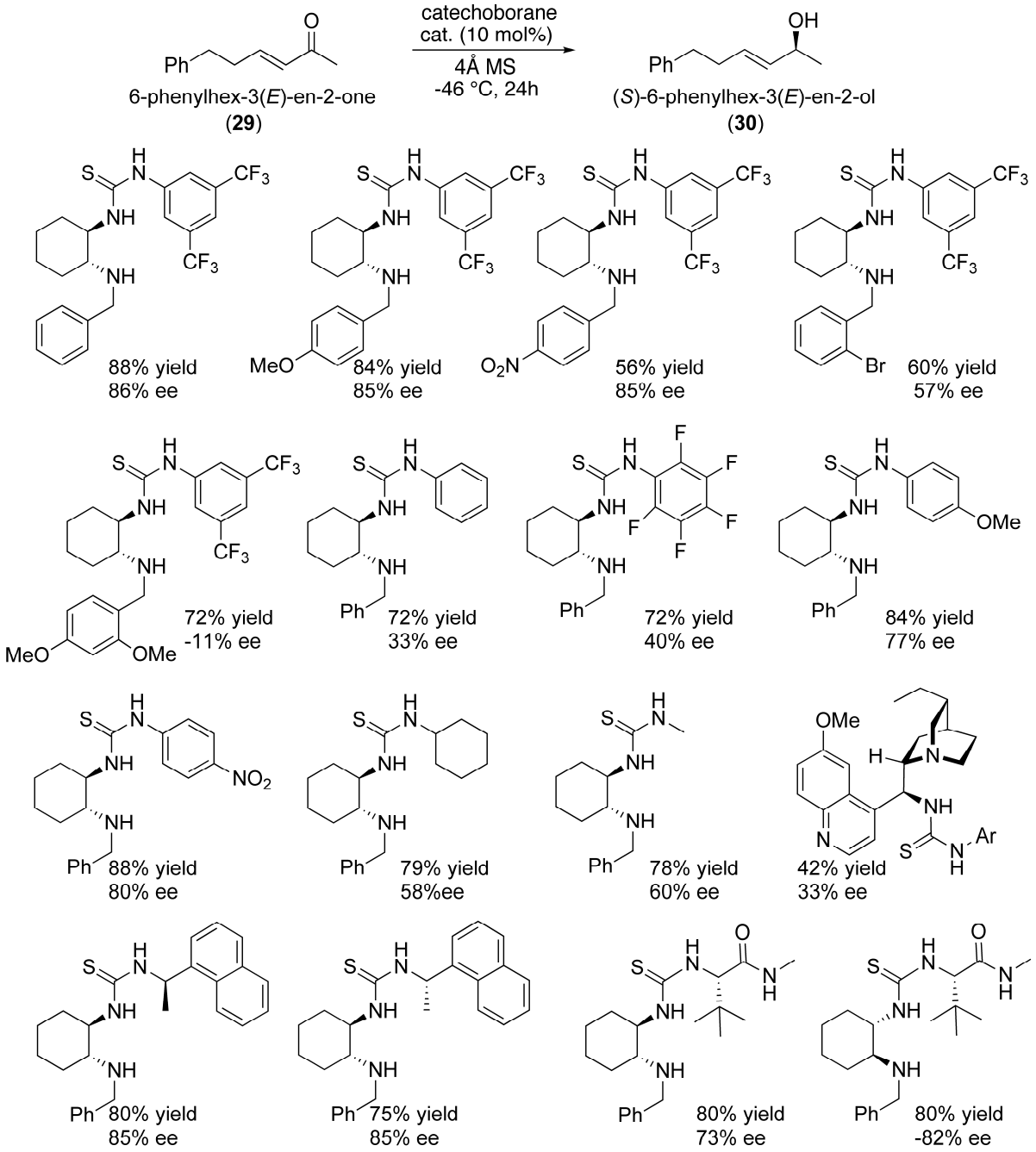
entry	temperature (°C)	yield (%)	ee (%)[a]
1	-78	24	73
2	-65	64	96
3	-59 ~ -62	80	97.7
4	-55	81	97.9
5	-46	88	98
6	-46 (in THF)	39	22
7	-46 (in CH_2Cl_2)	49	49
8	-41 ~ -42	76	97.3
9	-30	88	85
10	-20	60	85

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



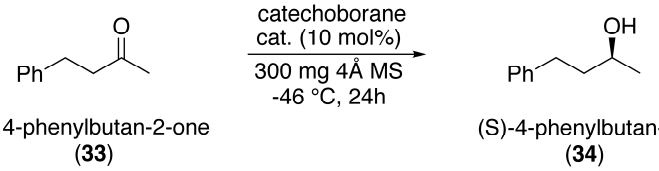


c. Catalyst survey for 6-phenylhex-3(*E*)-en-2-one (**29**)



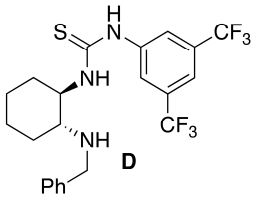
(A negative % ee indicates the *R*-enantiomer was produced)

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



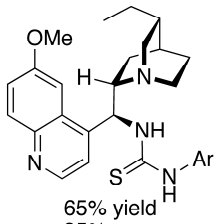
4-phenylbutan-2-one (**33**)

(S)-4-phenylbutan-2-ol (**34**)

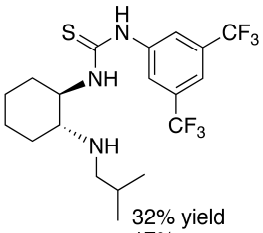


D

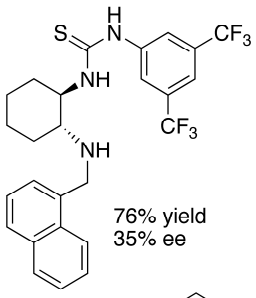
81% yield
47% ee



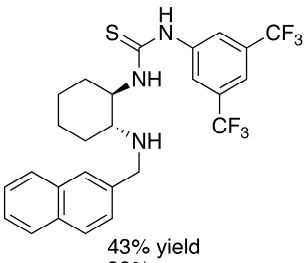
65% yield
35% ee



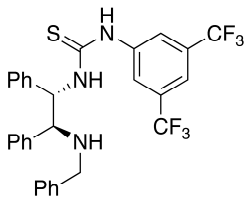
32% yield
17% ee



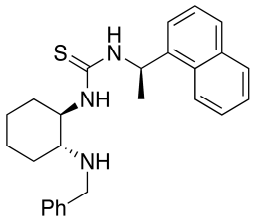
76% yield
35% ee



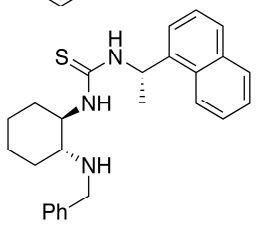
43% yield
39% ee



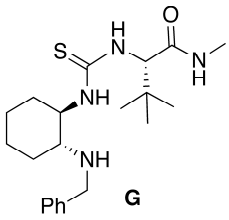
29% yield
4% ee



81% yield
45% ee

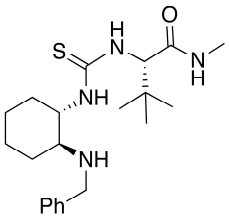


90% yield
60% ee

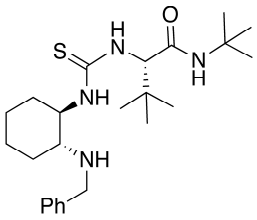


G

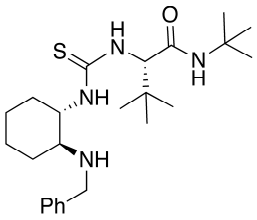
84% yield
63% ee



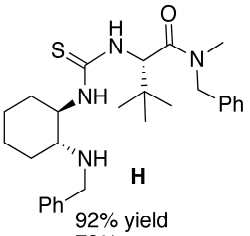
86% yield
-71% ee



62% yield
65% ee

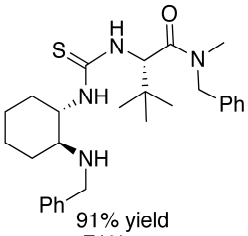


84% yield
-57% ee

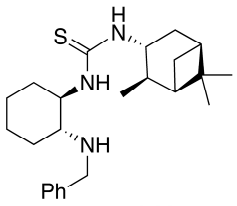


H

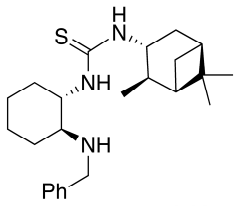
92% yield
79% ee



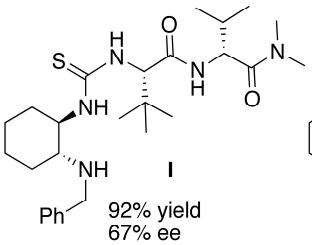
91% yield
-71% ee



82% yield
53% ee

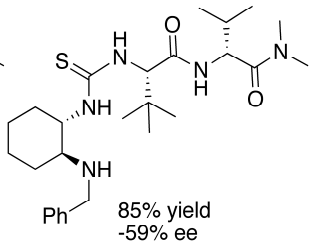


85% yield
-50% ee

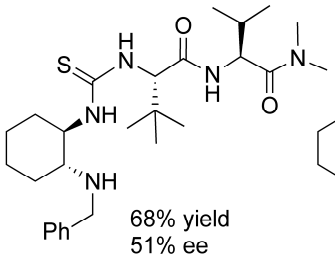


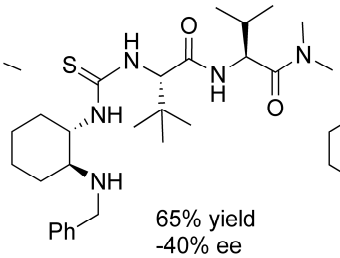
I

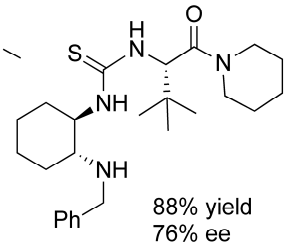
92% yield
67% ee

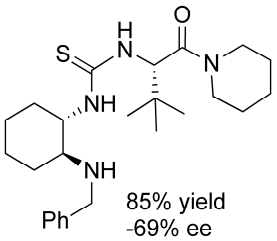


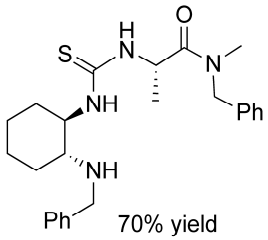
85% yield
-59% ee

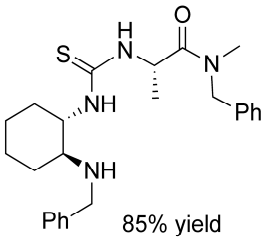

68% yield
51% ee

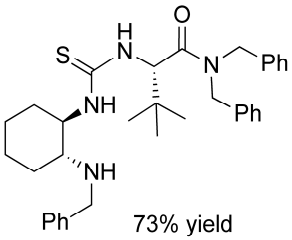

65% yield
-40% ee

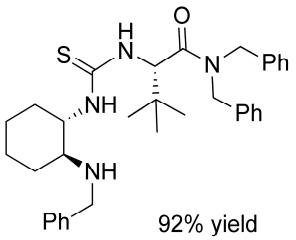

88% yield
76% ee

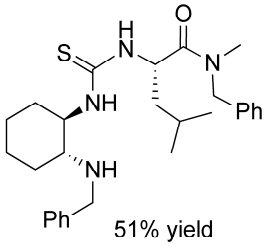

85% yield
-69% ee

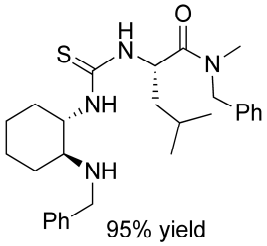

70% yield
19% ee

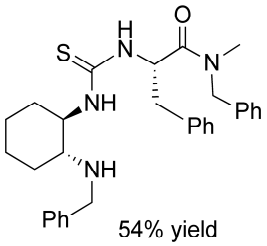

85% yield
-45% ee

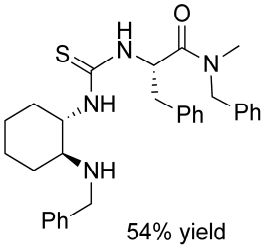

73% yield
55% ee


92% yield
-67% ee


51% yield
31% ee

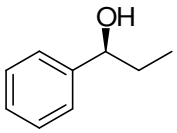

95% yield
-67% ee


54% yield
55% ee


54% yield
-47% ee

IV. General reduction procedure and examples

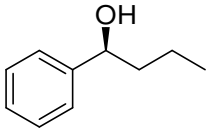
A mixture of catalyst **D** (12 mg, 0.025 mmol), 4Å molecular sieves (250 mg, freshly activated), and acetophenone (30 mg, 0.25 mmol) in toluene (0.7 mL) under an argon atmosphere was cooled to -78 °C. A solution of catecholborane (0.4 mL, 1.0 M in toluene, 0.4 mmol) was added slowly and the reaction 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 Et_2O (20 mL × 3), dried over Na_2SO_4 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, $CHCl_3$), lit.[4] (*R*)-1-phenylethanol, 96% ee, $[\alpha]_D$ = +42.92 (*c* 1.04, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 145.9, 128.6 (2C), 127.6, 125.5 (2C), 70.5, 25.3.



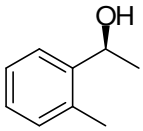
(*S*)-1-Phenylpropan-1-ol (4). 86% yield, 99% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 17.3 min, R_t (minor) = 14.4 min; major isomer $[\alpha]^{20}_D$ = -47.4 (*c* 1.48, $CHCl_3$), lit.[5] (*S*)-1-phenylpropan-1-ol, 98% ee, $[\alpha]^{25}_D$ = -48.4 (*c* 2.31, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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.

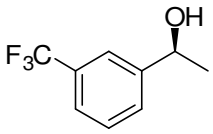
(5) Lutz, C.; Knochel, P. *J. Org. Chem.* **1997**, *62*, 7895–7898.



(*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, $CHCl_3$), lit.[6] (*R*)-1-phenylbutan-1-ol, 93% ee, $[\alpha]^{24}_D$ = +42 (*c* 0.28, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 144.7, 128.5 (2C), 127.6, 126.1 (2C), 76.1, 31.9, 10.2.



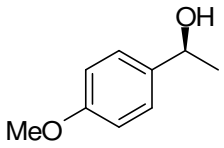
(*S*)-1-*o*-Tolylethanol (8). 71% yield, 95% ee. HPLC analysis: Chiralpak AD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 24.4 min, R_t (minor) = 21.8 min; major isomer $[\alpha]^{20}_D$ = -70.0 (*c* 1.0, $CHCl_3$), lit.[7] (*S*)-1-o-tolylethanol, 98% ee, $[\alpha]^{25}_D$ = -39.7 (*c* 0.56, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 143.9, 134.2, 130.4, 127.2, 126.4, 124.5, 66.8, 24.0, 19.0.



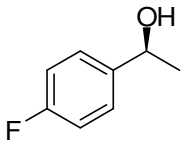
(*S*)-1-(3-(Trifluoromethyl)phenyl)ethanol (10). 92% yield, 96% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 14.0 min, R_t (minor) = 17.6 min; major isomer $[\alpha]^{20}_D$ = -31.0 (*c* 1.95, $CHCl_3$), lit.[8] (*S*)-1-(3-(trifluoromethyl)phenyl)ethanol, >99% ee, $[\alpha]^{20}_D$ = -27.9 (c 1.64 in CH_3OH); ^{1}H NMR ($CDCl_3$,

(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 ($CDCl_3$, 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.

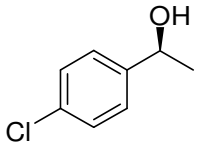


(*S*)-1-(4-Methoxyphenyl)ethanol (12). 80% yield, 97% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 21.2 min, R_t (minor) = 18.6 min; major isomer $[\alpha]^{20}_D$ = -52.3 (*c* 1.55, $CHCl_3$), lit.[4] (*R*)-1-(4-methoxyphenyl)ethanol, 92% ee, $[\alpha]_D$ = +40.64 (*c* 1.53, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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 ($CDCl_3$, 75 MHz) δ 159.0, 138.1, 126.8 (2C), 113.9 (2C), 70.0, 55.4, 25.1.

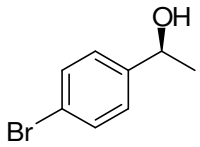


(*S*)-1-(4-Fluorophenyl)ethanol (14). 84% yield, 99% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 0.6 mL/min, 254 nm, R_t (major) = 36.4 min, R_t (minor) = 43.0 min; major isomer $[\alpha]^{20}_D$ = -44.8 (*c* 1.40, $CHCl_3$), lit.[9] (*S*)-1-(4-fluorophenyl)ethanol, 97% ee, $[\alpha]_D$ = -47.4 (*c* 0.0576, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.34-7.26 (m, 2H), 7.05-7.6.98 (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 ($CDCl_3$, 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.

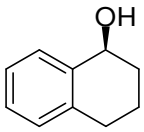
(9) Carter, M. B.; Schiøtt, B.; Gutiérrez, A.; Buchwald, S. L. *J. Am. Chem. Soc.* **1994**, *116*, 11667.



(*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, $CHCl_3$), lit.[10] (*S*)-1-(4-chlorophenyl)ethanol, 96% ee, $[\alpha]^{27}_D$ = -45.0 (*c* 0.90, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 144.4, 133.2, 128.7 (2C), 126.9 (2C), 69.9, 25.4.



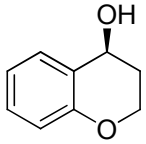
(*S*)-1-(4-Bromophenyl)ethanol (18). 95% yield, 99% ee. HPLC analysis: Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm; R_t (major) = 24.5 min, R_t (minor) = 28.9 min); major isomer $[\alpha]^{20}_D$ = -36.9 (*c* 2.40, $CHCl_3$), lit.[10] (*S*)-1-(4-bromophenyl)ethanol, 98% ee, $[\alpha]^{27}_D$ = -37.3 (*c* 1.1, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 144.9, 131.7 (2C), 127.3 (2C), 121.3, 69.9, 25.4.



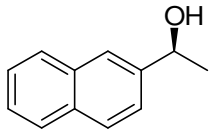
(*S*)-1,2,3,4-Tetrahydronaphthalen-1-ol (20). 86% yield, 99% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 16.2 min, R_t (minor) = 18.6 min; major isomer $[\alpha]^{20}_D$ = +38.9 (*c* 1.45, $CHCl_3$), lit.[7] (*S*)-1,2,3,4-tetrahydronaphthalen-1-ol, 91% ee, $[\alpha]^{25}_D$ = +31.2 (*c* 0.54, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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.

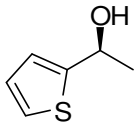


(*S*)-Chroman-4-ol (22). 95% yield, 98% ee. HPLC analysis: Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 16.1 min, R_t (minor) = 21.2 min; major isomer $[\alpha]^{20}_D$ = -62.0 (*c* 1.8, $CHCl_3$), lit.[11] (*R*)-chroman-4-ol, >99% ee, $[\alpha]^{20}_D$ = +65 (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.27 (dd, *J* = 7.5, 1.5Hz, 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.

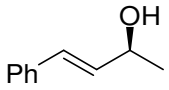


(*S*)-1-(Naphthalen-2-yl)ethanol (24). 93% yield, 98% ee. HPLC analysis: Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 25.7 min, R_t (minor) = 33.7 min); major isomer $[\alpha]^{20}_D$ = -50.0 (*c* 2.0, $CHCl_3$), lit.[7] (*S*)-1-(naphthalen-2-yl)ethanol, 94% ee, $[\alpha]^{25}_D$ = -40.6 (*c* 0.8, $CHCl_3$); ^{1}H 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.

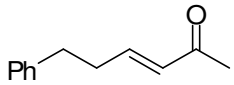
(11) Wettergren, J.; Bogevig, A.; Portier, M.; Adolfssona, H. *Adv. Synth. Catal.* **2006**, *348*, 1277–1282.



(*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, $CHCl_3$), lit.[12] (*S*)-1-(thiophen-2-yl)ethanol, 99% ee, $[\alpha]^{24}_D$ = -26.0 (*c* 1.02, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 150.0, 126.7, 124.5, 123.3, 66.2, 25.3.

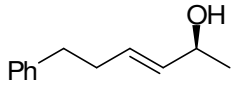


(*S*)-4-Phenylbut-3(*E*)-en-2-ol (28). 78% yield, 90% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 22.8 min, R_t (minor) = 16.0 min; major isomer $[\alpha]^{20}_D$ = -28.6 (*c* 1.4, $CHCl_3$), lit.[4] (*S*)-4-phenylbut-3(*E*)-en-2-ol, 92% ee, $[\alpha]^{25}_D$ = +16.4 (*c* 0.9, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 136.8, 133.7, 129.5, 128.7 (2C), 127.7, 126.5 (2C), 69.0, 23.5.

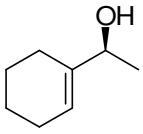


6-Phenylhex-3(*E*)-en-2-one (29). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32-7.17 (m, 5H), 6.82 (dt, *J* = 16.2 , 6.6 Hz, 1H), 6.09 (d, *J* = 16.2 Hz, 1H), 2.79 (t, *J* = 8.4 Hz, 2H), 2.59-2.51 (m, 2H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 198.6, 147.1, 140.7, 131.7, 128.6 (2C), 128.4 (2C), 126.3, 34.4, 34.1, 26.9; ES-MS $[MH]^+$ *m/z* 175.1.

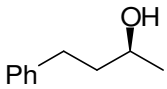
(12) Ohkuma, T.; Koizumi, M.; Yoshida, M.; Noyori, R. *Org. Lett.* **2000**, *2*, 1749–1751.



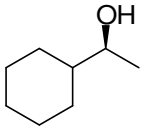
(*S*)-6-Phenylhex-3(*E*)-en-2-ol (30). 88% yield, 86% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm, R_t (major) = 20.5 min, R_t (minor) = 13.0 min; major isomer $[\alpha]^{20}_D$ = -8.8 (*c* 1.95, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32-7.19 (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); ^{13}C NMR ($CDCl_3$, 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, $CHCl_3$), lit.[7] (*S*)-1-cyclohexenylethanol, 91% ee, $[\alpha]^{25}_D$ = -9.4 (*c* 1.5, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 141.4, 121.7, 72.4, 25.0, 23.8, 22.8, 22.7, 21.7.



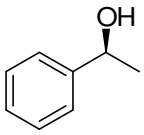
(*S*)-4-Phenylbutan-2-ol (34). 92% yield, 79% ee. HPLC analysis: Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm, R_t (major) = 15.5 min, R_t (minor) = 11.1 min; major isomer $[\alpha]^{20}_D$ = +13.8 (*c* 1.70, $CHCl_3$), lit.[4] (*R*)-4-phenylbutan-2-ol, 88% ee, $[\alpha]^{22}_D$ = -17.32 (*c* 1.6, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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, $CHCl_3$), lit.[13] (*R*)-1-cyclohexylethanol, 67% ee, $[\alpha]_D$ = -1.90 (*c* 0.75, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 72.3, 45.2, 28.8, 28.5, 26.6, 26.3, 26.2, 20.5.

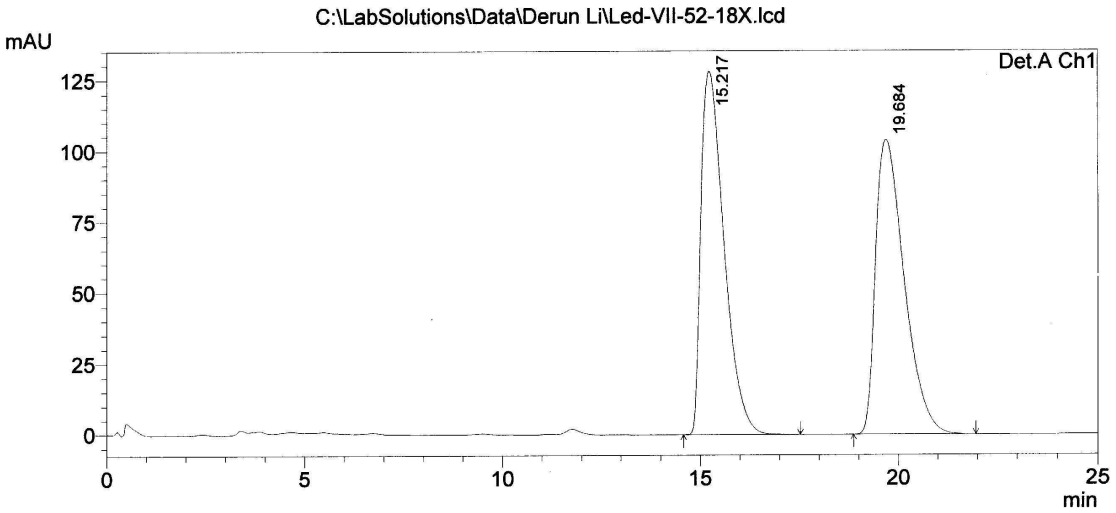
(13) Gamble, M. P.; Smith, A. R. C.; Wills, M. *J. Org. Chem*. **1998**, *63*, 6068–6071.

V. Chiral HPLC analyses



(*S*)-1-Phenylethanol (2)

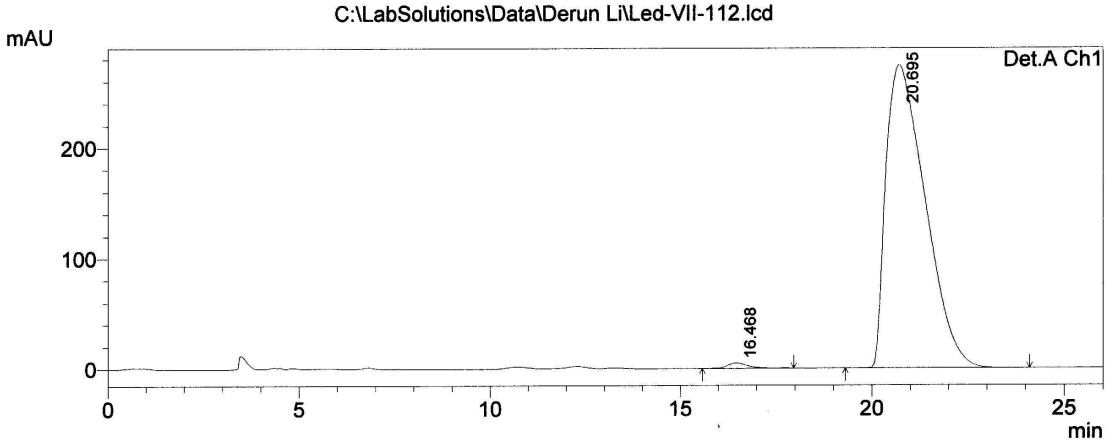
[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

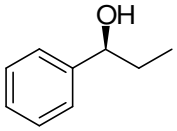
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.217	5195482	128317	49.887	55.218
2	19.684	5219004	104065	50.113	44.782
Total		10414486	232382	100.000	100.000



PeakTable

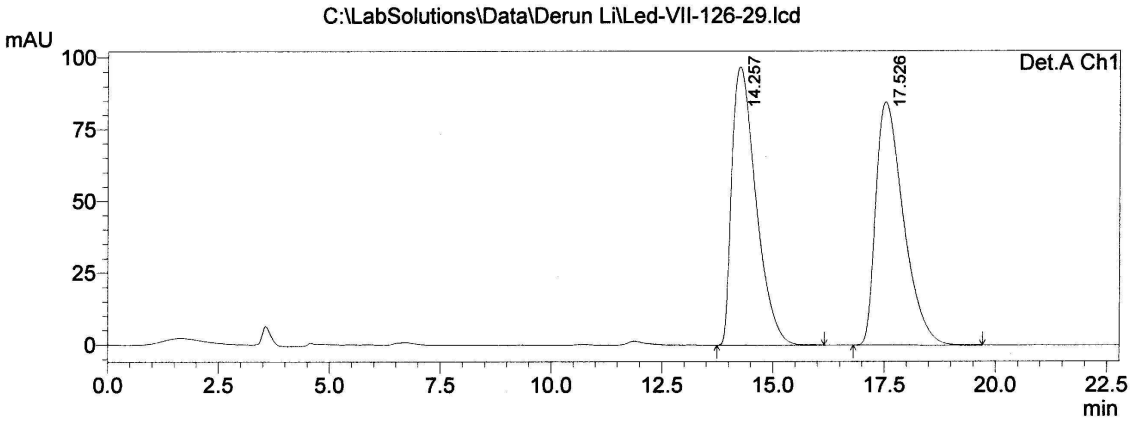
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.468	175384	4913	0.888	1.760
2	20.695	19582790	274225	99.112	98.240
Total		19758174	279138	100.000	100.000



(*S*)-1-Phenylpropan-1-ol (4)

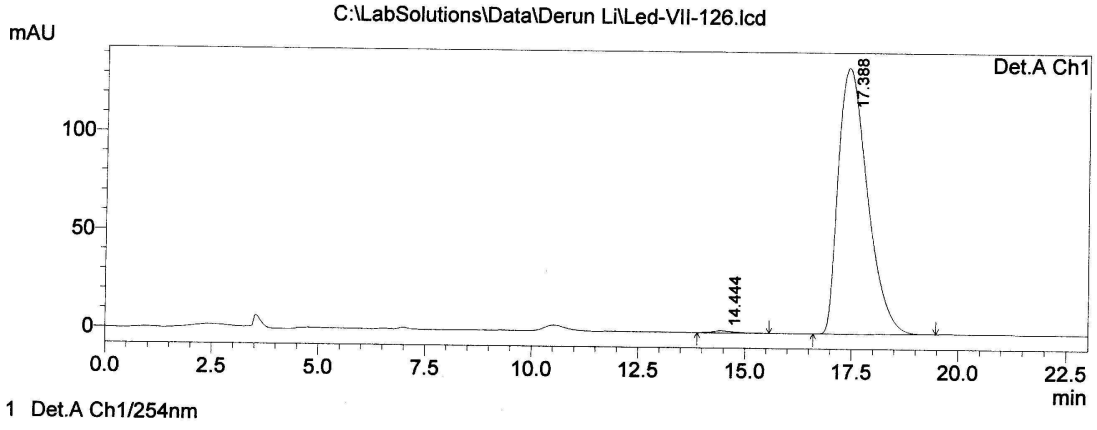
[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

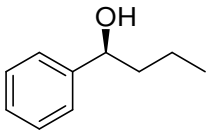
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.257	3663280	96891	49.987	53.365
2	17.526	3665159	84671	50.013	46.635
Total		7328440	181561	100.000	100.000



PeakTable

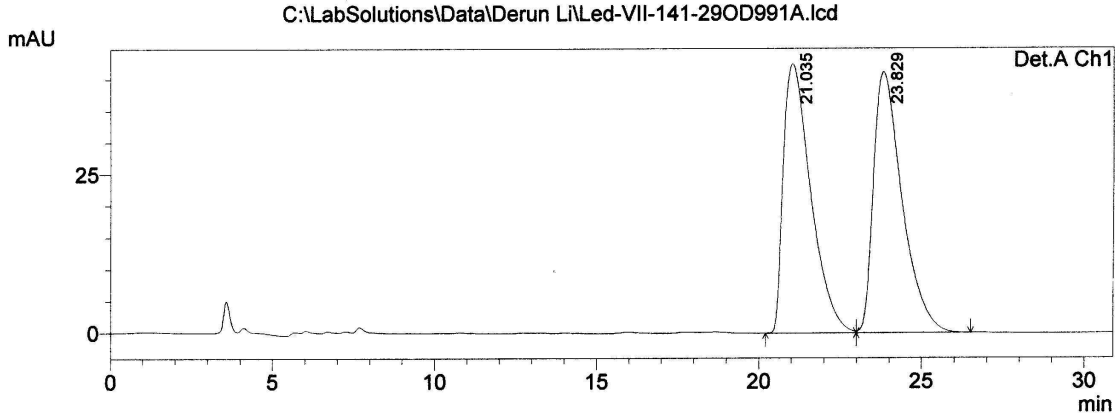
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.444	32400	1026	0.501	0.752
2	17.388	6438129	135380	99.499	99.248
Total		6470530	136406	100.000	100.000



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

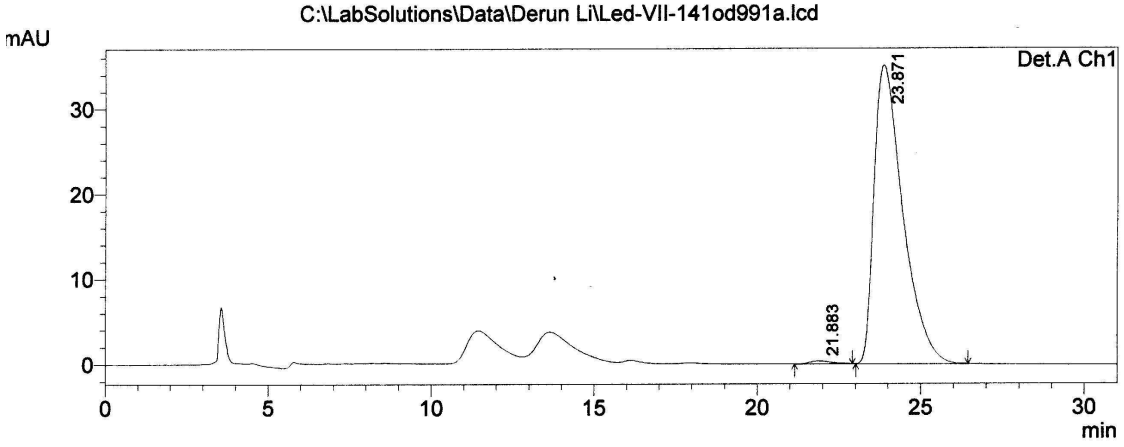
[Chiralcel OD column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

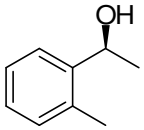
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.035	2502206	42447	49.845	50.752
2	23.829	2517729	41190	50.155	49.248
Total		5019935	83637	100.000	100.000



PeakTable

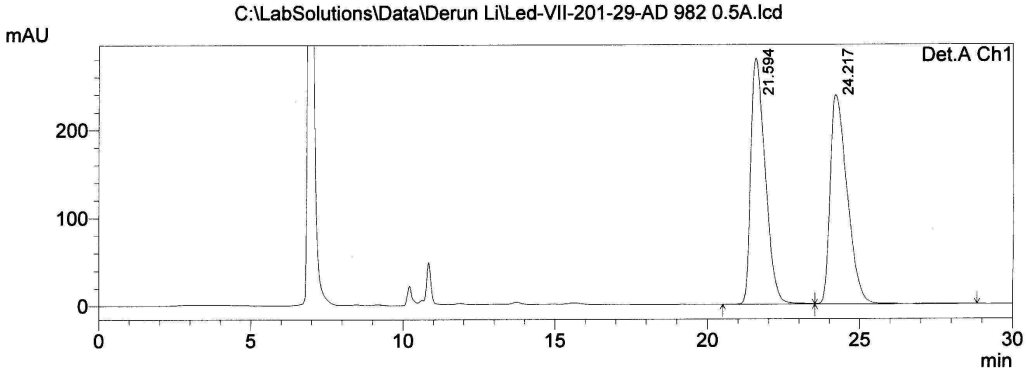
Detector A Ch1 254nm

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)

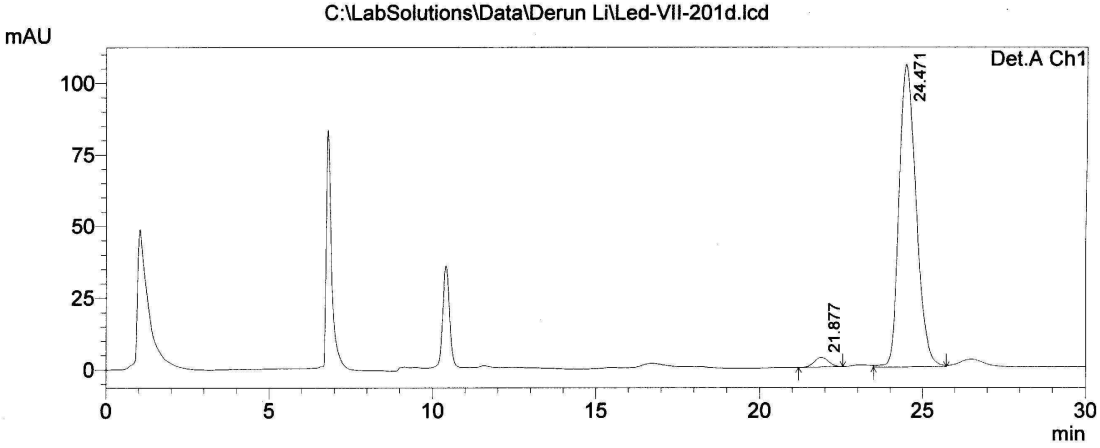
[Chiralpak AD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 245nm

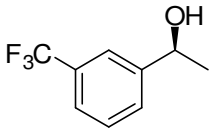
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.594	9263298	279274	49.986	54.041
2	24.217	9268335	237510	50.014	45.959
Total		18531633	516784	100.000	100.000



PeakTable

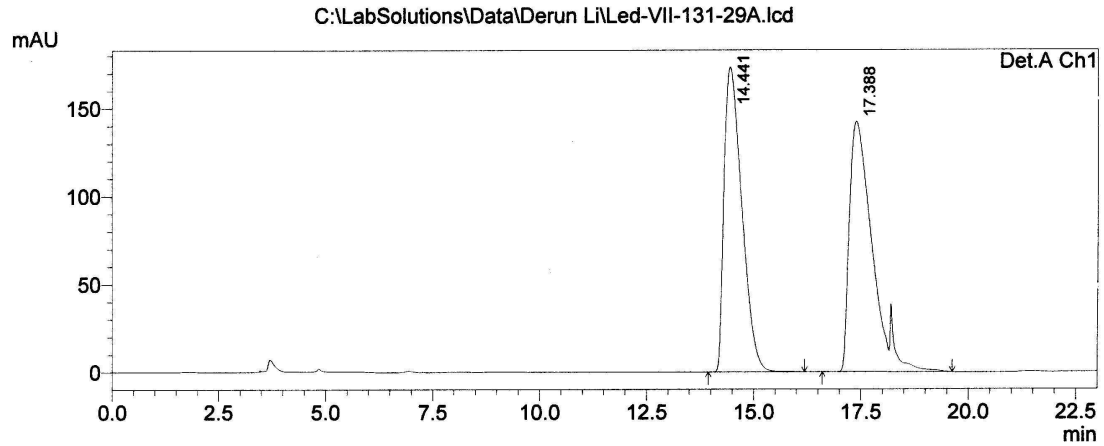
Detector A Ch1 245nm

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% *i*PrOH/hexane, 1.0 mL/min, 254 nm]

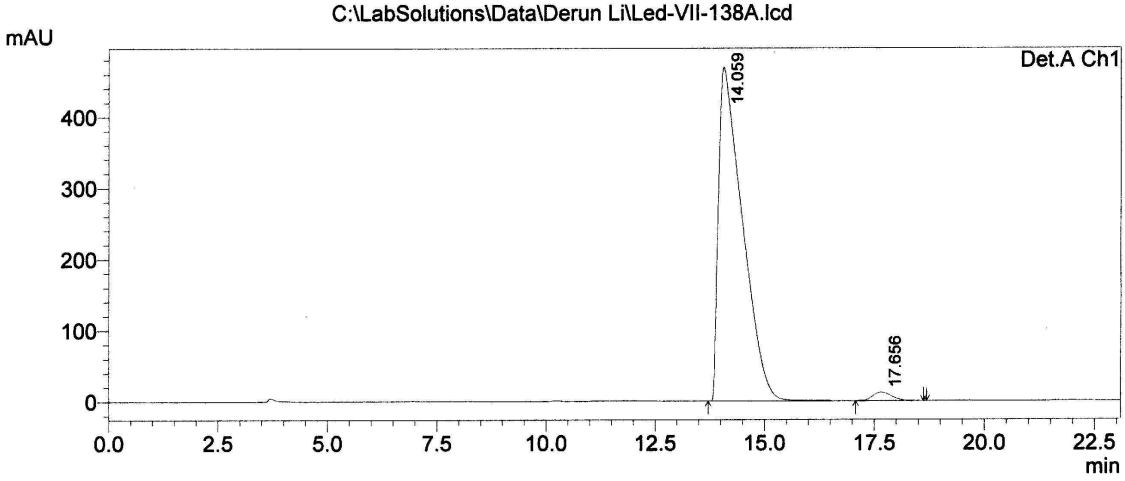


1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm

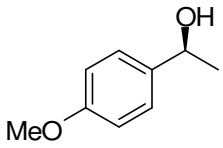
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.441	5024407	173407	48.499	54.921
2	17.388	5335511	142332	51.501	45.079
Total		10359918	315739	100.000	100.000



PeakTable

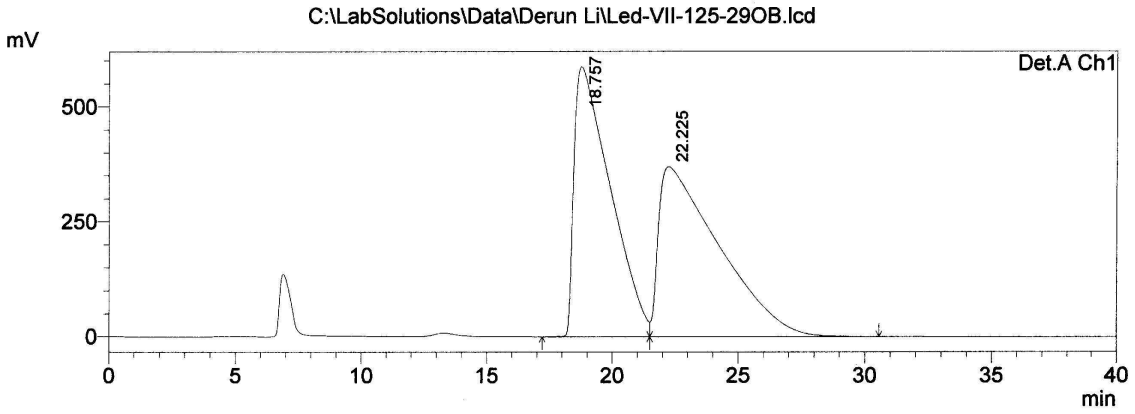
Detector A Ch1 254nm

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)

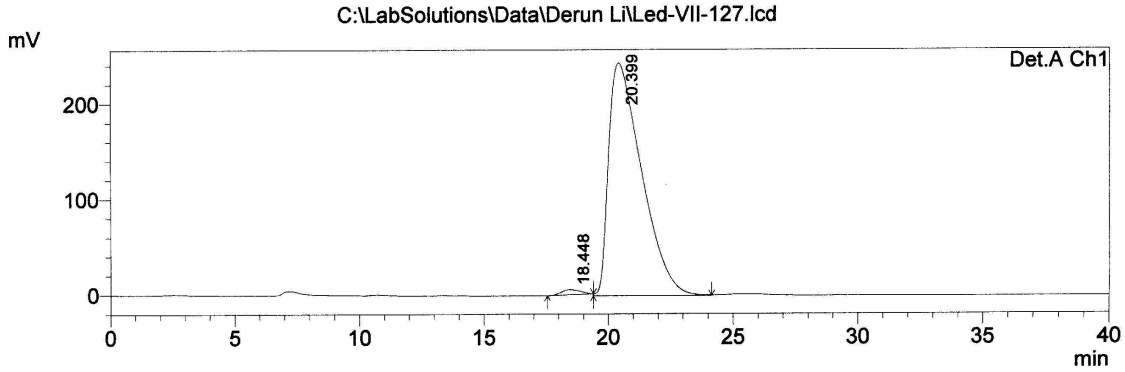
[Chiralcel OB column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

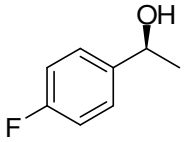
Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.757	58901696	587336	48.893	61.400
2	22.225	61568771	369231	51.107	38.600
Total		120470468	956567	100.000	100.000



PeakTable

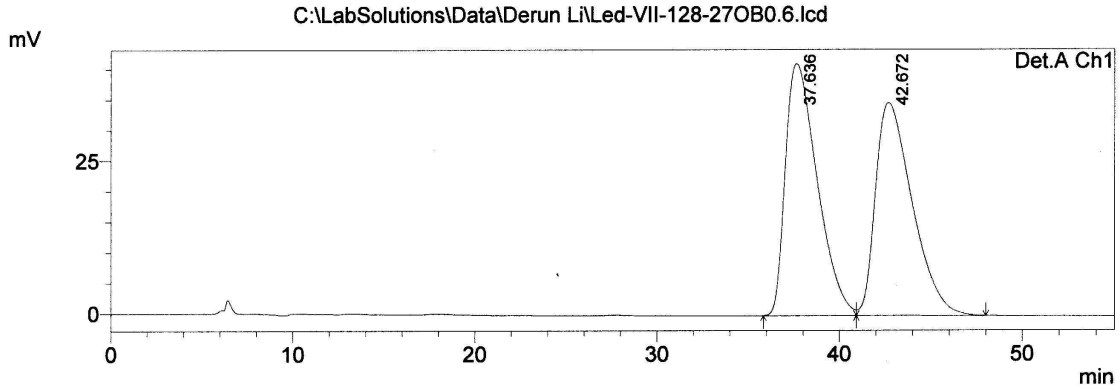
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.448	279574	5313	1.230	2.135
2	20.399	22451339	243501	98.770	97.865
Total		22730914	248814	100.000	100.000



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

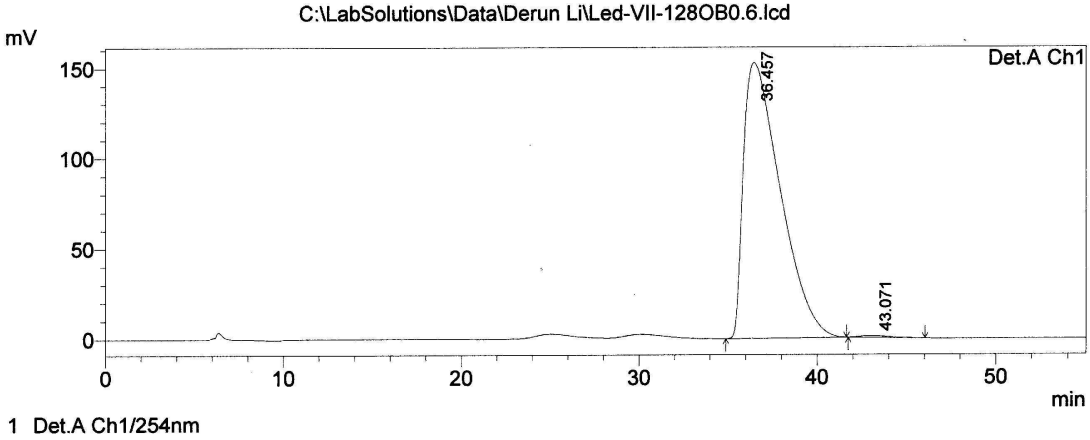
[Chiralcel OB column (250 mm × 4.6 mm), 1% *i*PrOH/hexane, 0.6 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

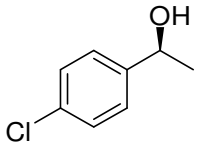
Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.636	5012167	41185	50.687	54.167
2	42.672	4876271	34848	49.313	45.833
Total		9888439	76033	100.000	100.000



PeakTable

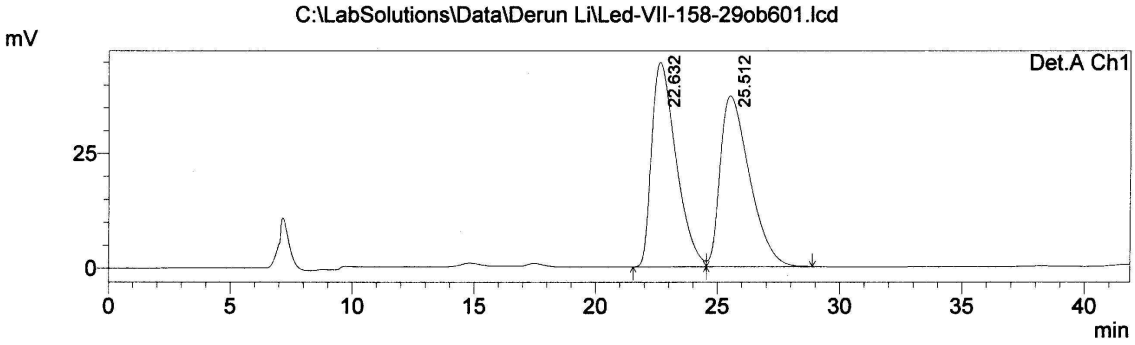
Detector A Ch1 254nm

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)

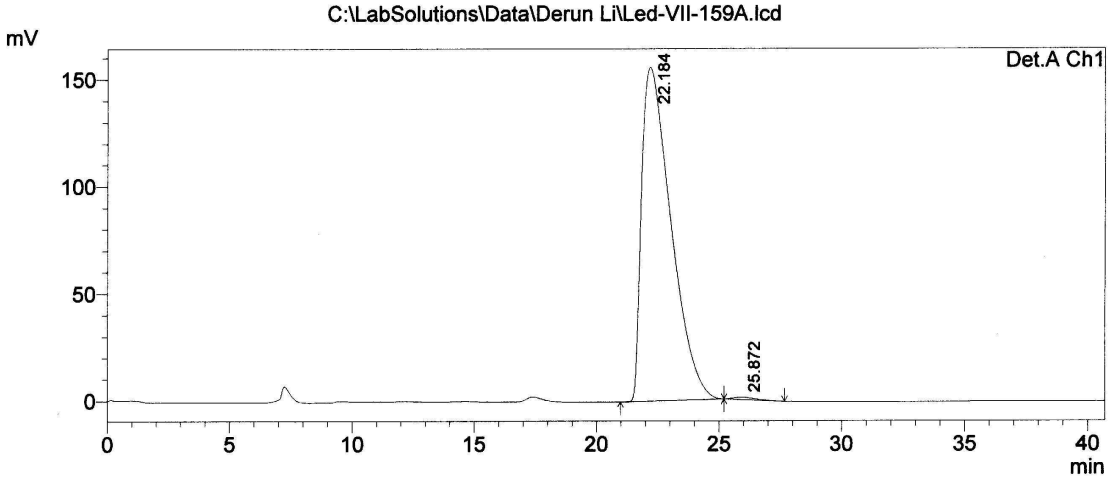
[Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

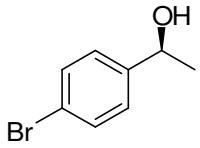
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.632	3112787	44596	49.903	54.482
2	25.512	3124831	37258	50.097	45.518
Total		6237618	81854	100.000	100.000



PeakTable

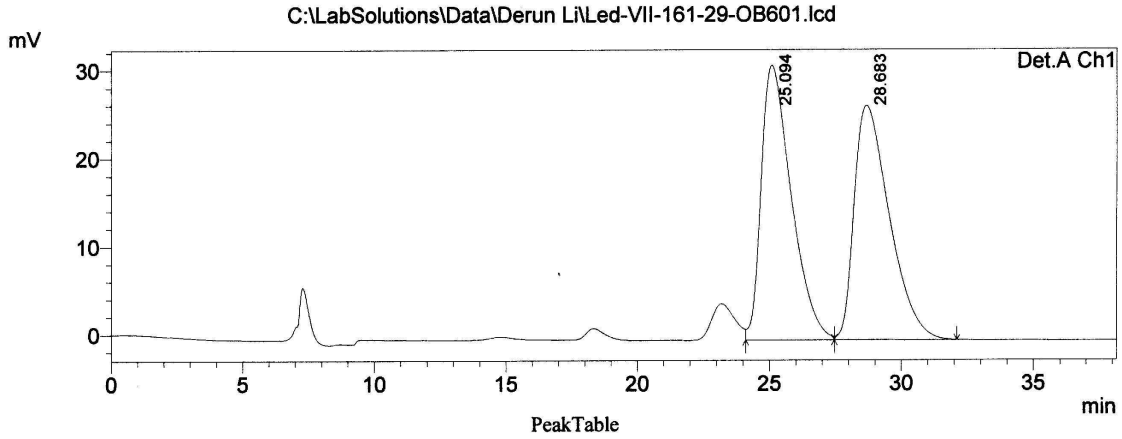
Detector A Ch1 254nm

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)

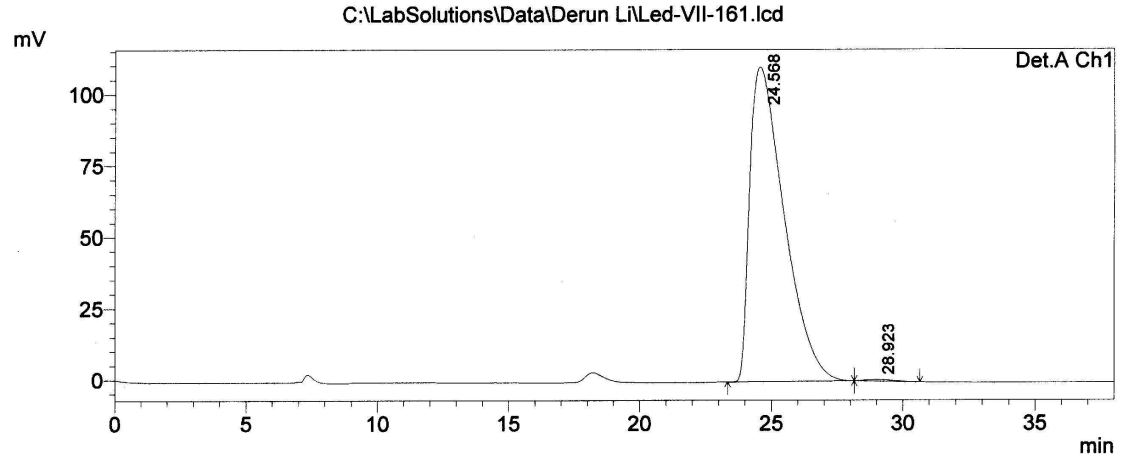
[Chiralcel OB column (250 mm × 4.6 mm), hexane/EtOH (60:1), 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.094	2478207	31209	50.075	53.949
2	28.683	2470749	26639	49.925	46.051
Total		4948956	57848	100.000	100.000

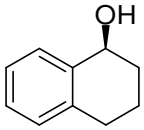


1 Det.A Ch1/254nm

PeakTable

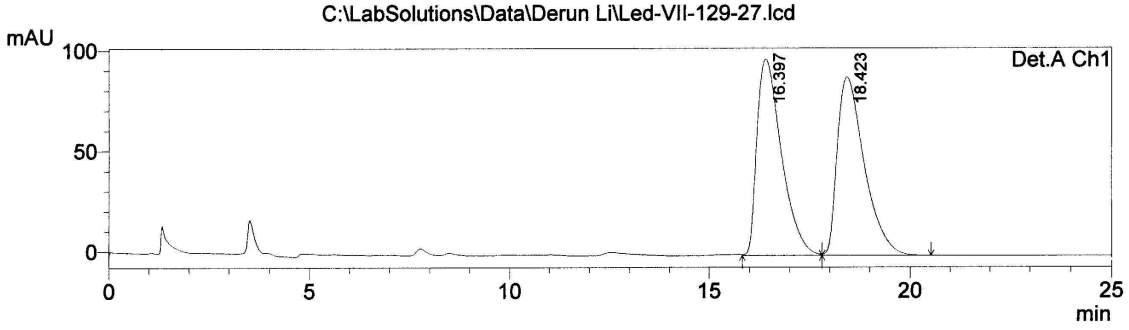
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.568	10029031	110179	99.591	99.486
2	28.923	41200	569	0.409	0.514
Total		10070230	110749	100.000	100.000



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

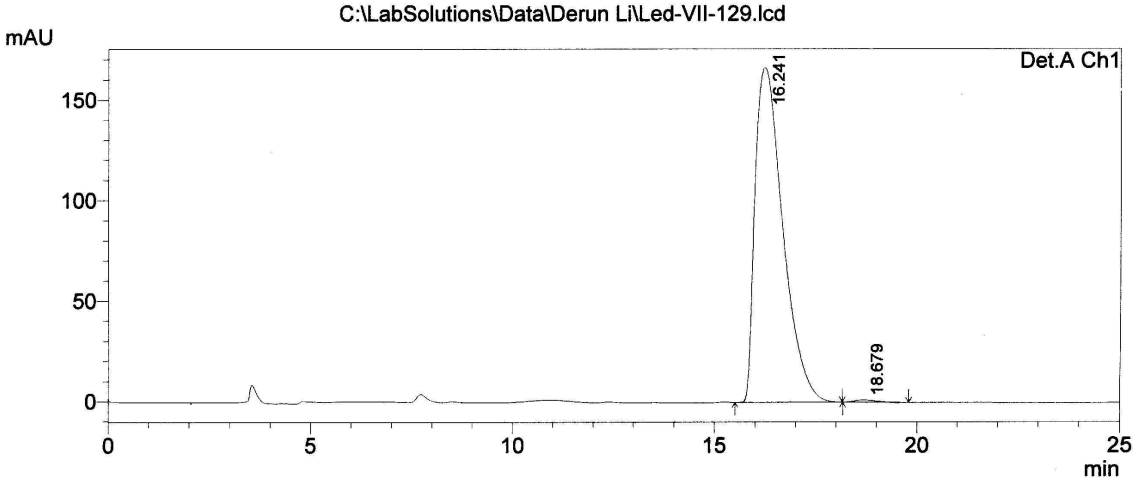
[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

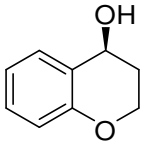
Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.397	4169825	97648	50.025	52.388
2	18.423	4165710	88747	49.975	47.612
Total		8335535	186395	100.000	100.000



PeakTable

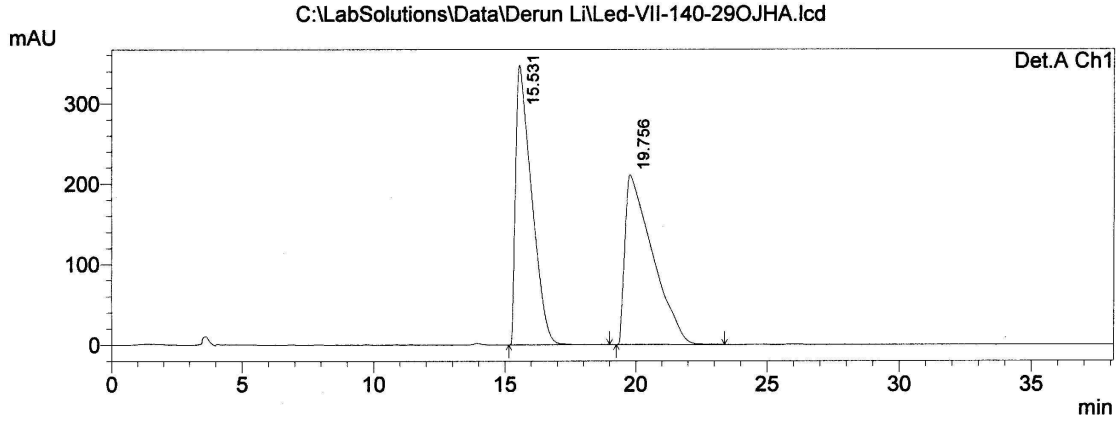
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.241	7963948	166589	99.443	99.305
2	18.679	44593	1165	0.557	0.695
Total		8008541	167754	100.000	100.000



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

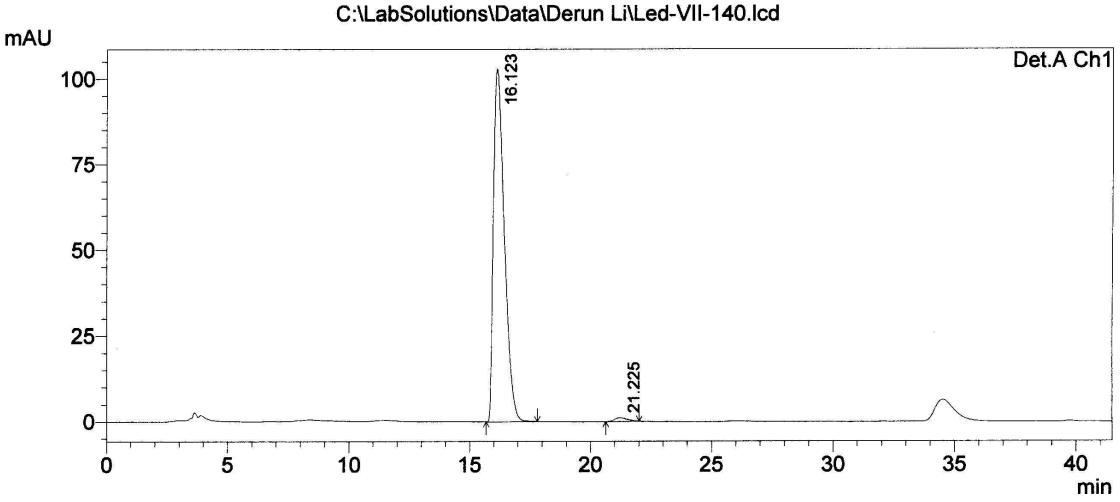
[Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

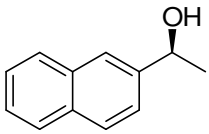
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.531	14775439	347688	48.862	62.245
2	19.756	15463959	210895	51.138	37.755
Total		30239399	558583	100.000	100.000



PeakTable

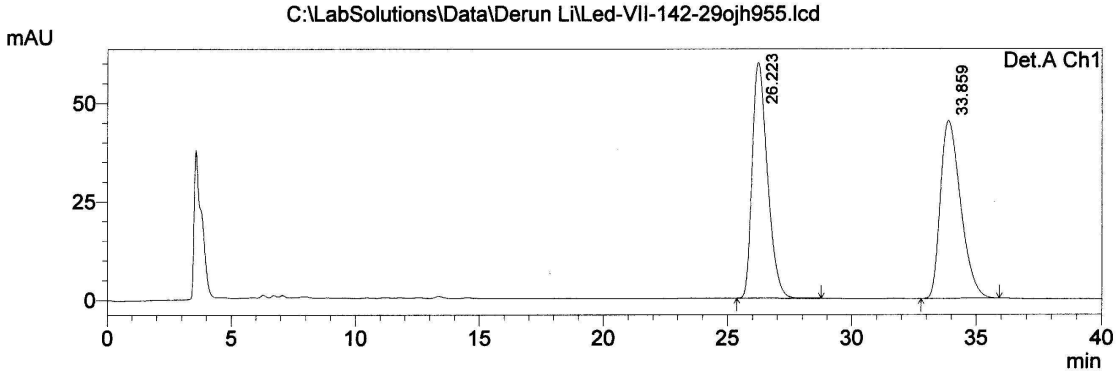
Detector A Ch1 254nm

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)

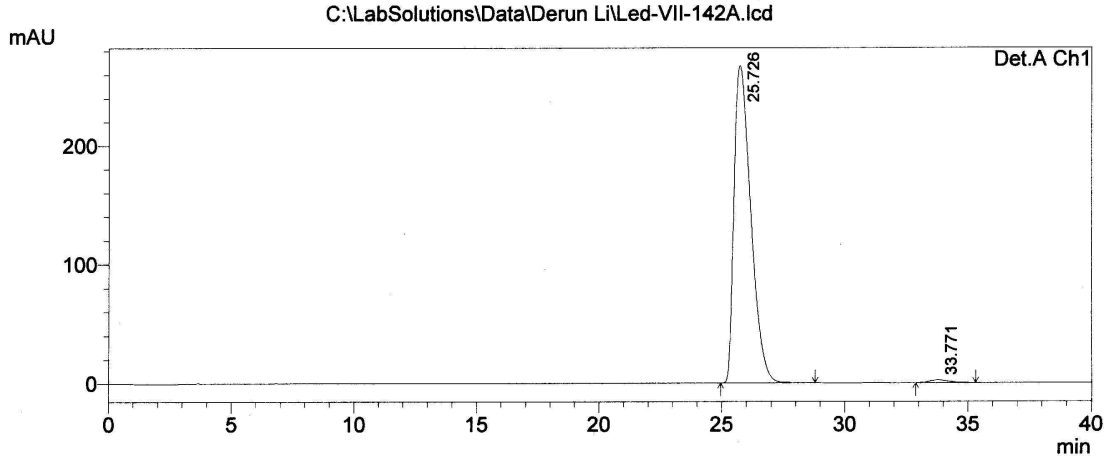
[Chiralcel OJ-H column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



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

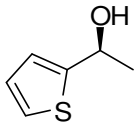


1 Det.A Ch1/254nm

PeakTable

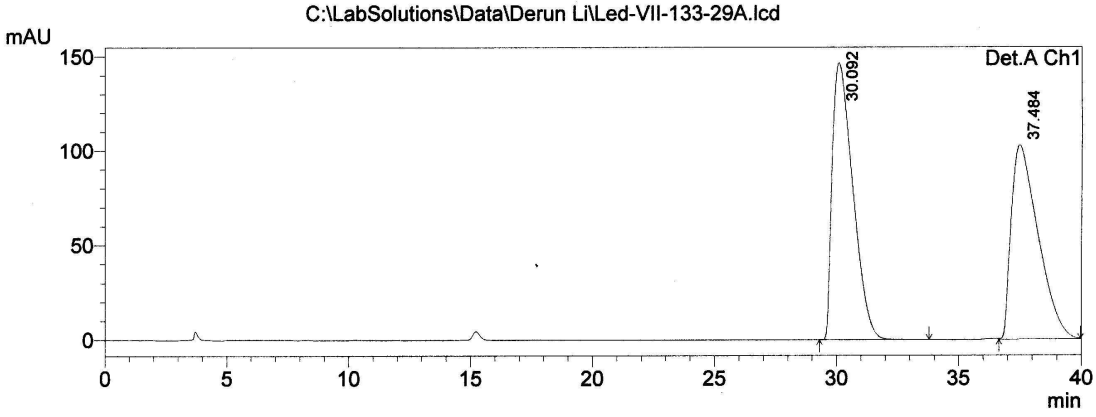
Detector A Ch1 254nm

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)

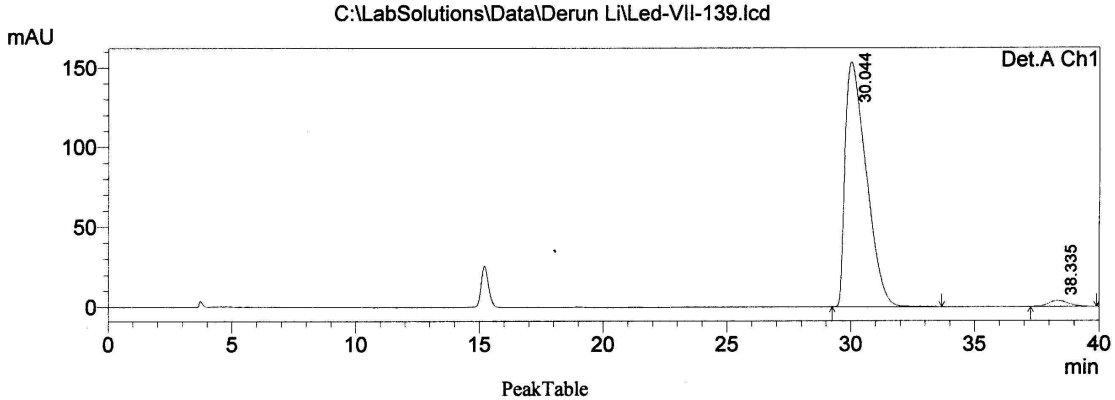
[Chiralcel OD column (250 mm × 4.6 mm), 2% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

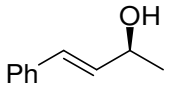
Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.092	8637146	146707	52.589	58.809
2	37.484	7786764	102755	47.411	41.191
Total		16423910	249462	100.000	100.000



PeakTable

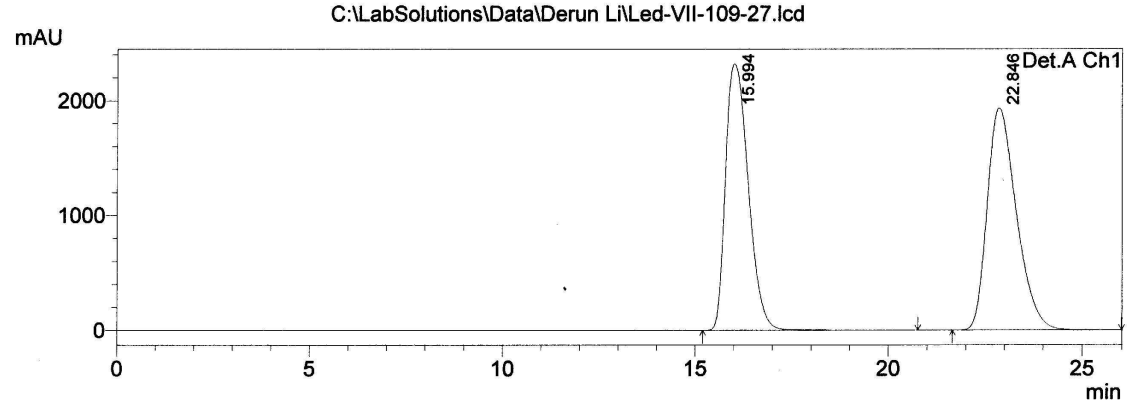
Detector A Ch1 254nm

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)

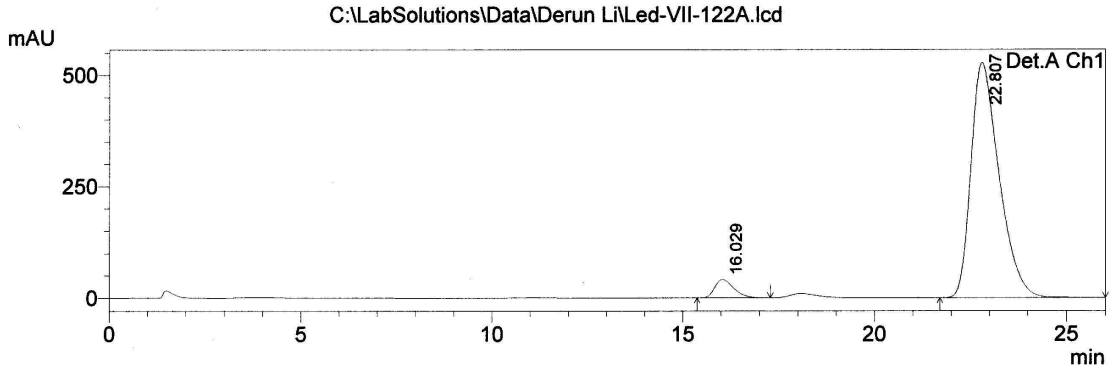
[Chiralcel OD column (250 mm × 4.6 mm), 10% *i*PrOH/hexane, 0.5 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

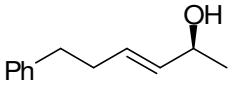
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.994	95021379	2317777	48.402	54.523
2	22.846	101294977	1933241	51.598	45.477
Total		196316355	4251018	100.000	100.000



PeakTable

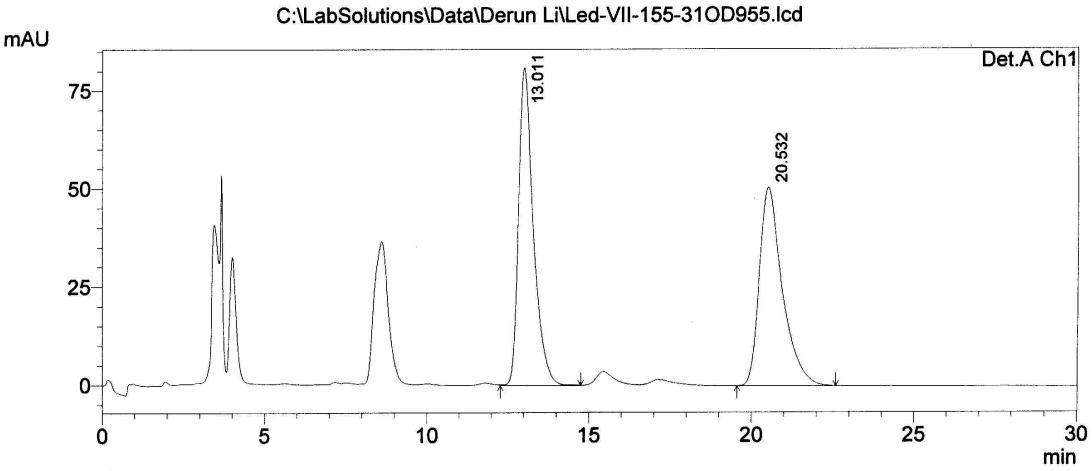
Detector A Ch1 254nm

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



(*S*)-6-Phenylhex-3(*E*)-en-2-ol (30)

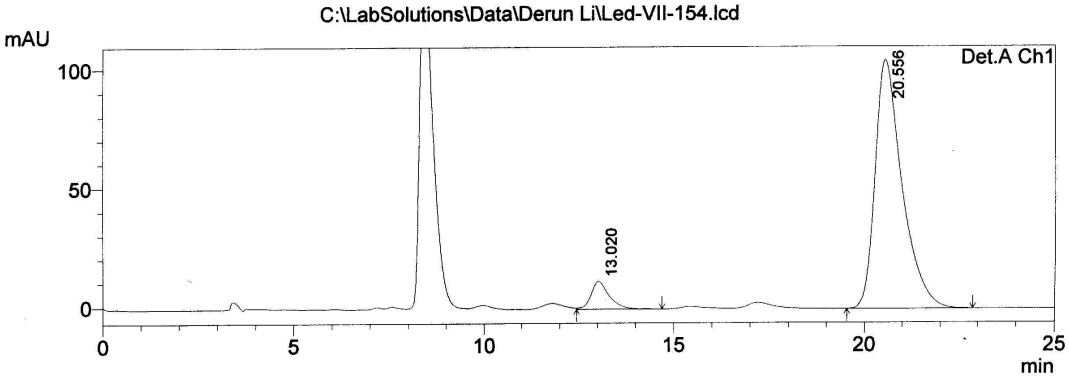
[Chiralcel OD column (250 mm × 4.6 mm), 5% *i*PrOH/hexane, 1.0 mL/min, 254 nm]



PeakTable

Detector A Ch1 254nm

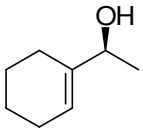
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.011	2559620	80983	50.768	61.555
2	20.532	2482162	50580	49.232	38.445
Total		5041781	131563	100.000	100.000



PeakTable

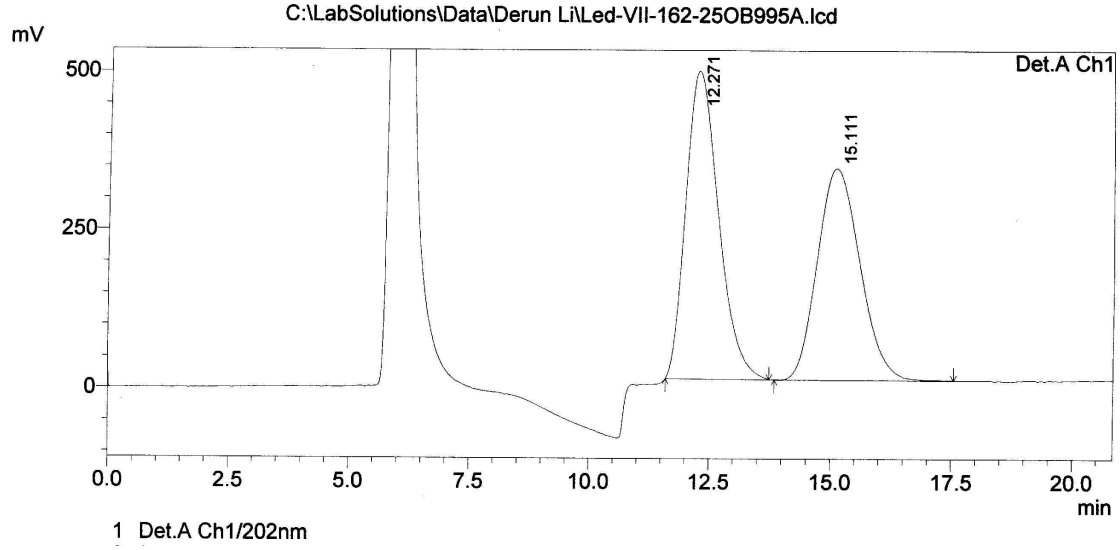
Detector A Ch1 254nm

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

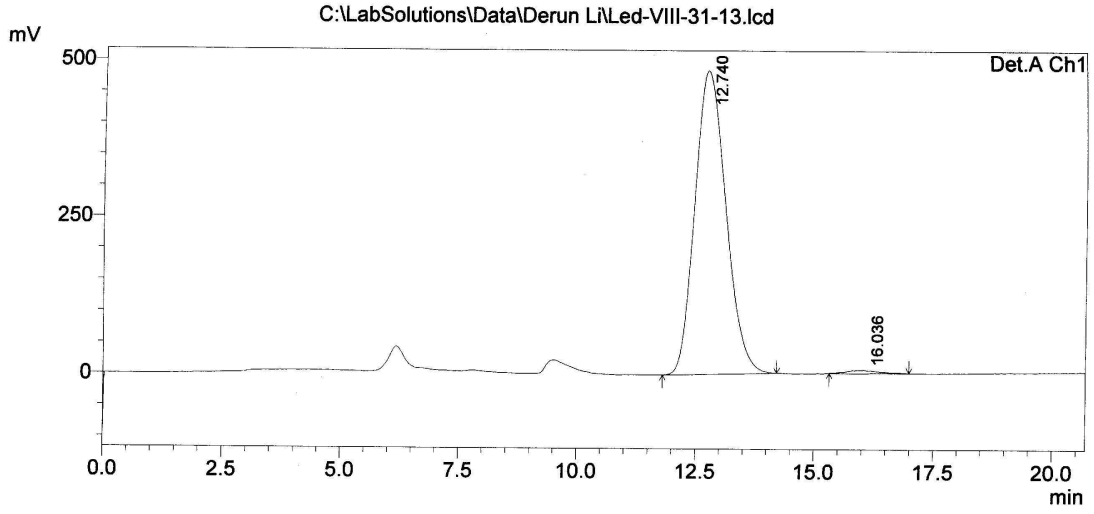


(*S*)-1-Cyclohexenylethanol (32)

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

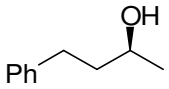


Peak#	Ret. Time	Area	Height	Area %
1	12.271	23025141	486438	52.962
2	15.111	20449762	334456	47.038
Total		43474904	820894	100.000



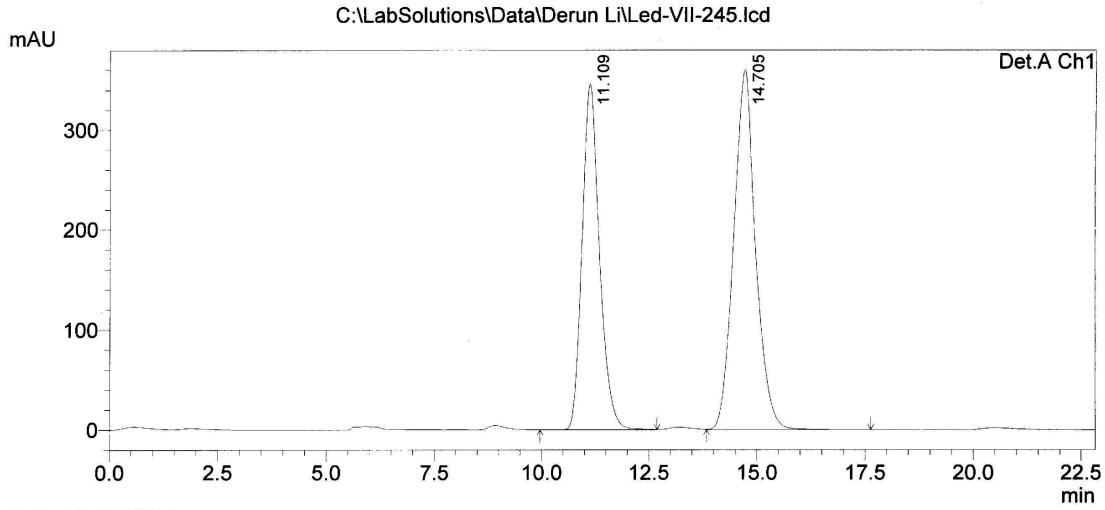
PeakTable

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% *i*PrOH/hexane, 0.5 mL/min, 254 nm]

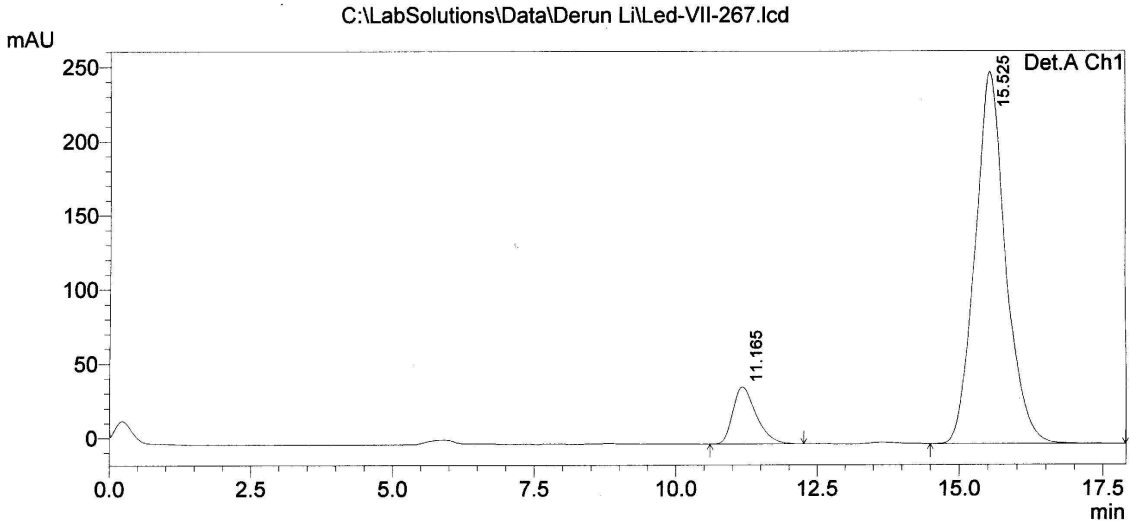


1 Det.A Ch1/254nm

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

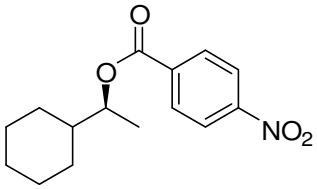


1 Det.A Ch1/254nm

PeakTable

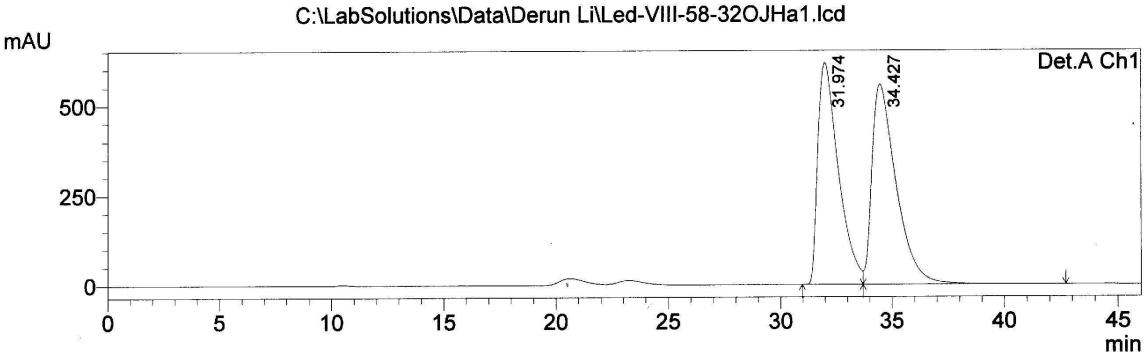
Detector A Ch1 254nm

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]

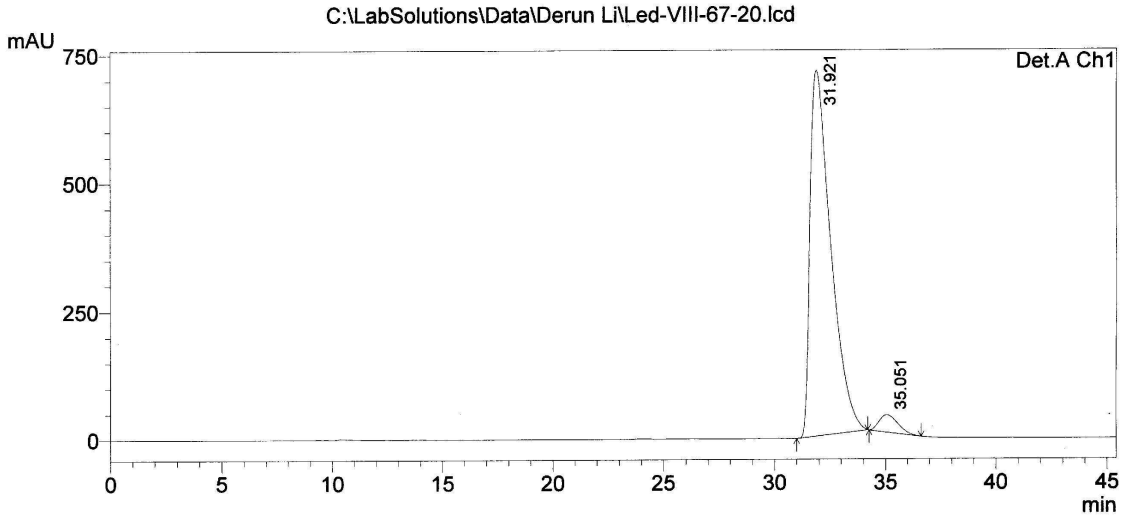


1 Det.A Ch1/254nm

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



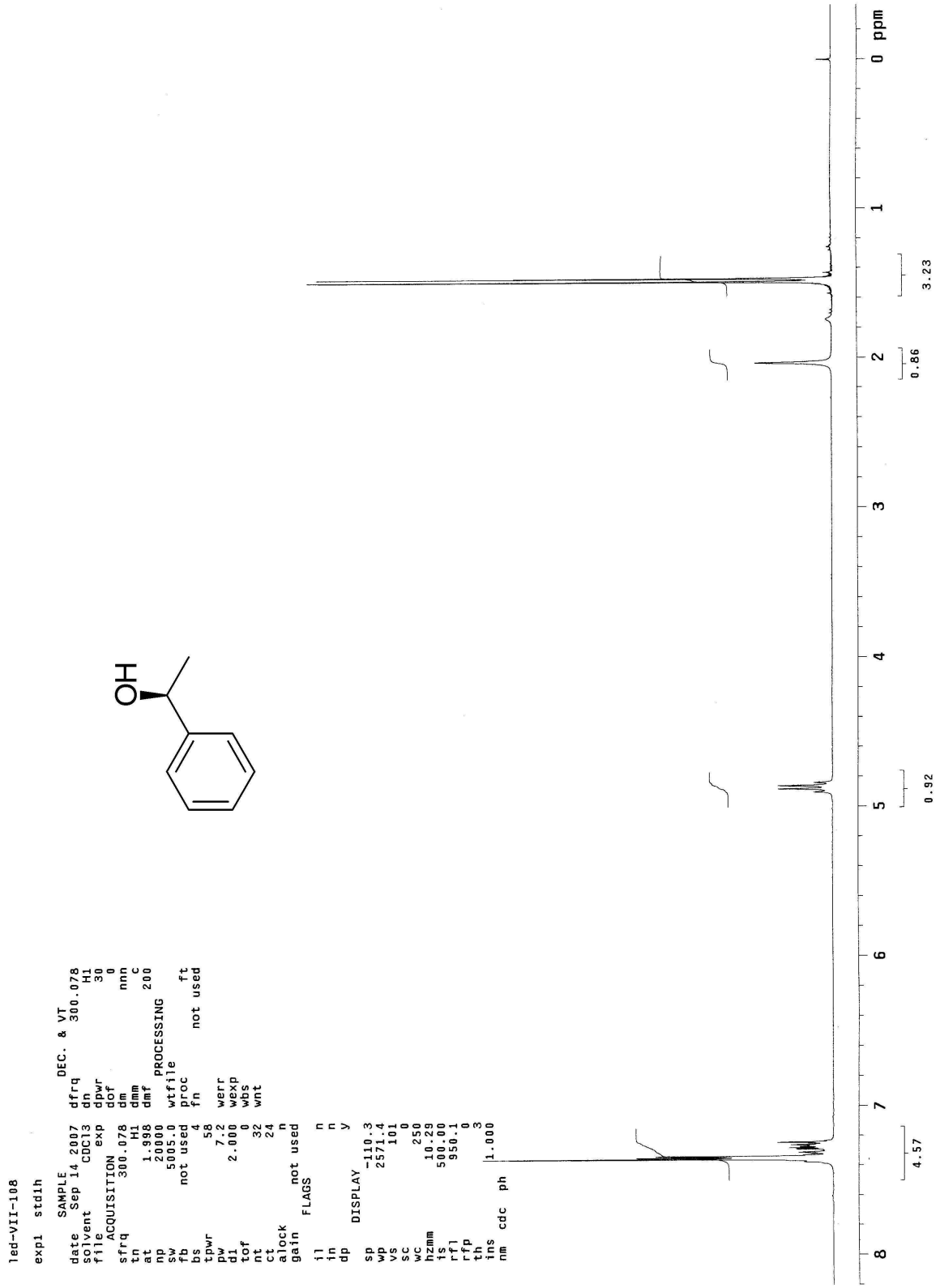
1 Det.A Ch1/254nm

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

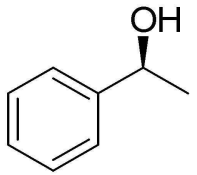
VI. NMR spectra

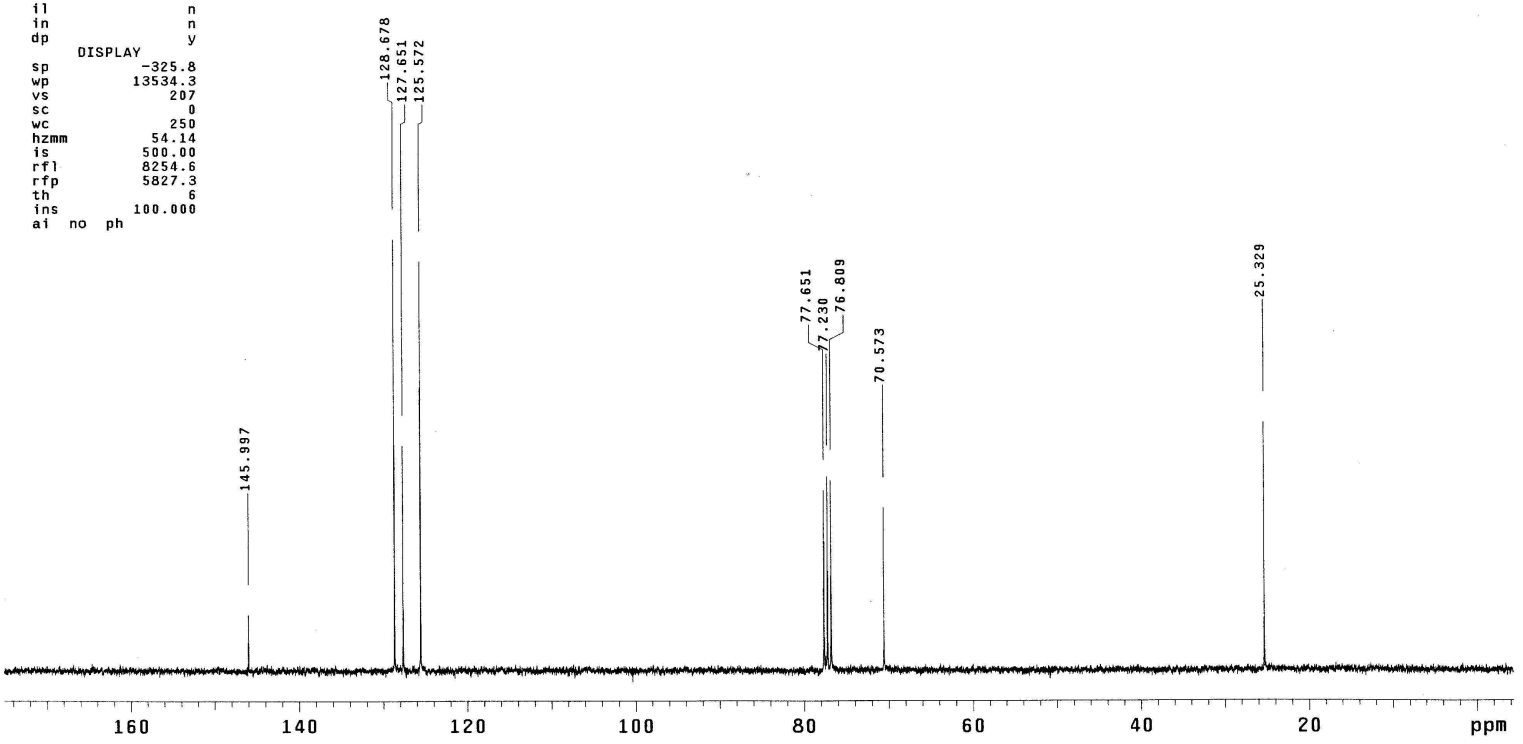


led-VII-108-C13

expl std13c

SAMPLE		DEC. & VT	
date	Sep 14 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	1.000	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	1000	wnt	
ct	192		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-325.8		
wp	13534.3		
vs	207		
sc	0		
wc	250		
hzmm	54.14		
is	500.00		
rfl	8254.6		
rfp	5827.3		
th	6		
ins	100.000		
ai no ph			

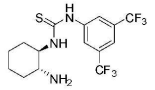


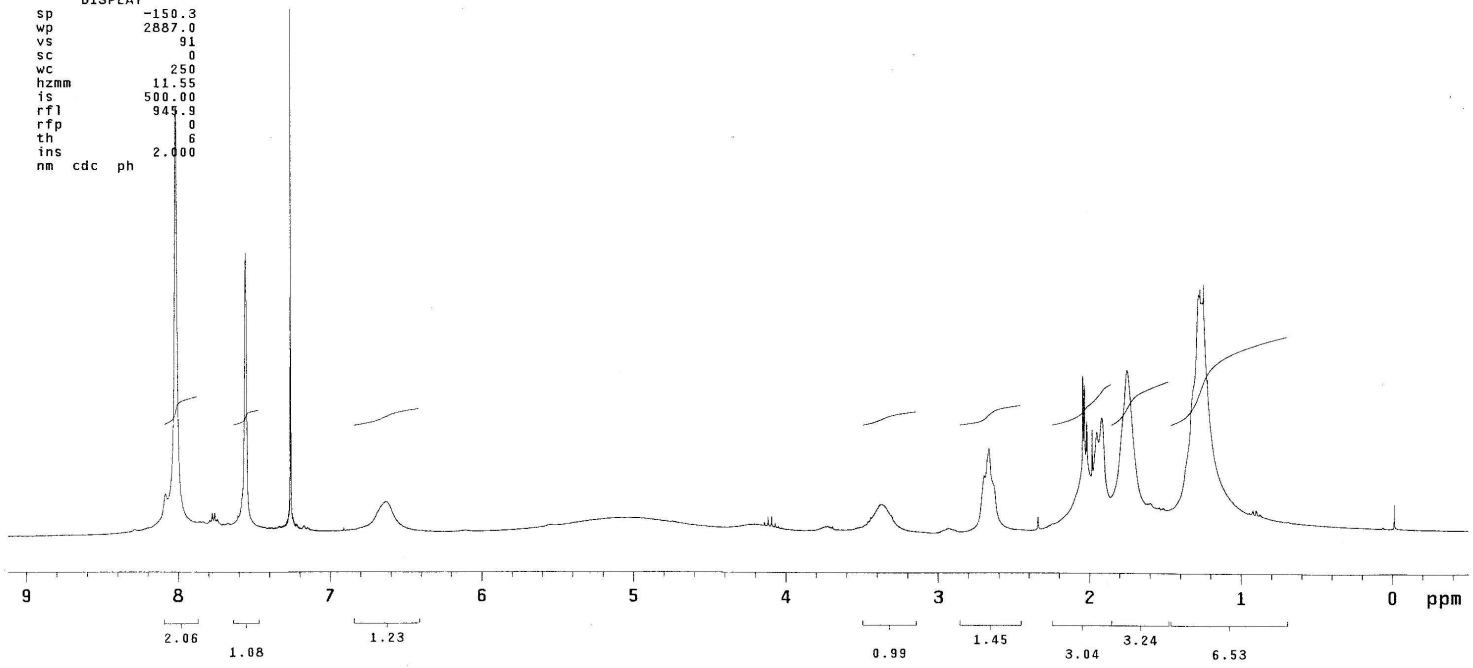


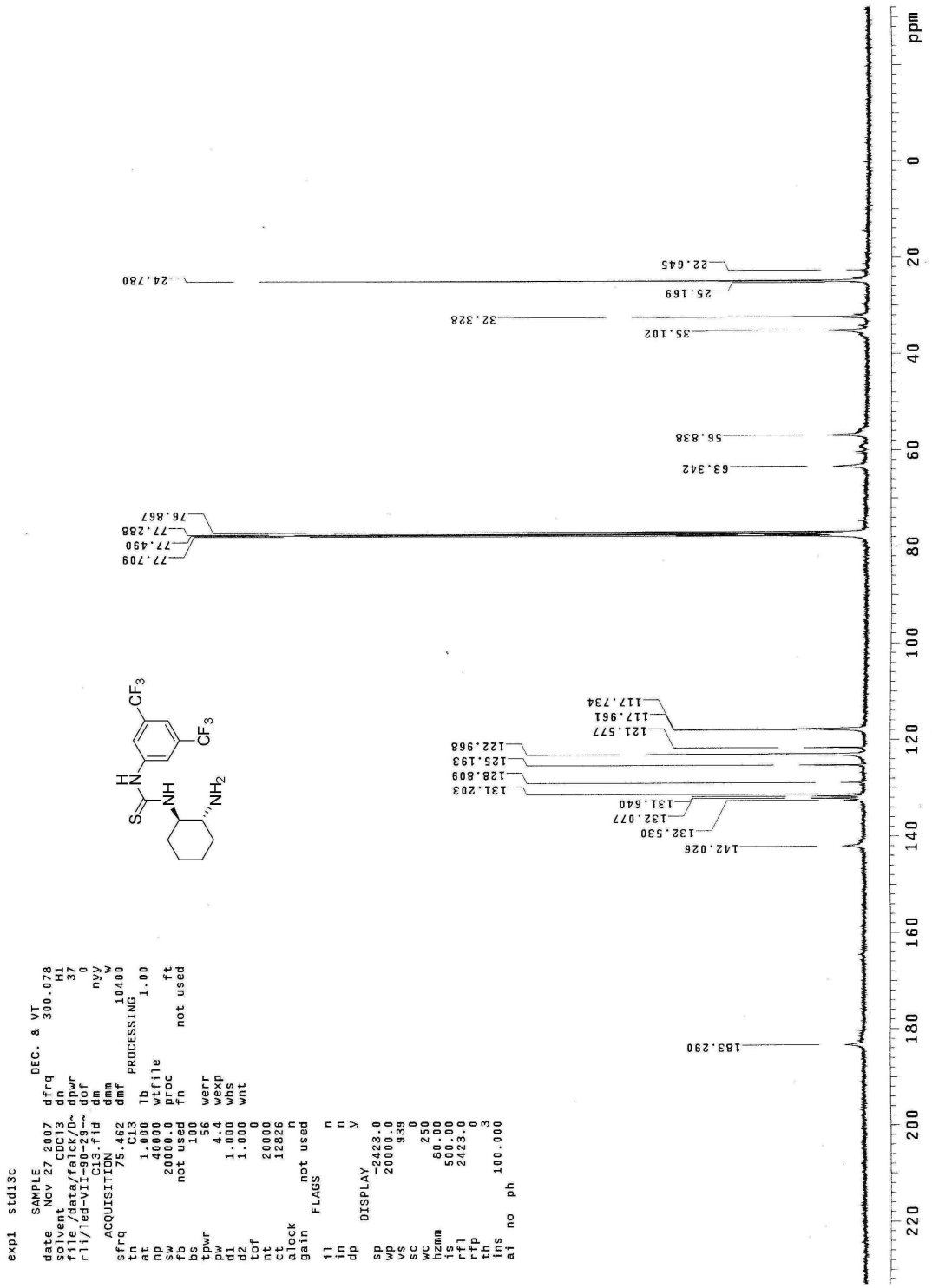
led-VII-90-29

expl std1h

SAMPLE		DEC. & VT	
date	Nov 27 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-150.3		
wp	2887.0		
vs	91		
sc	0		
wc	250		
hzmm	11.55		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	2.000		
nm cdc ph			



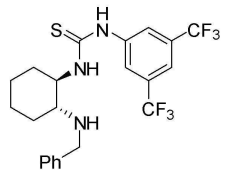


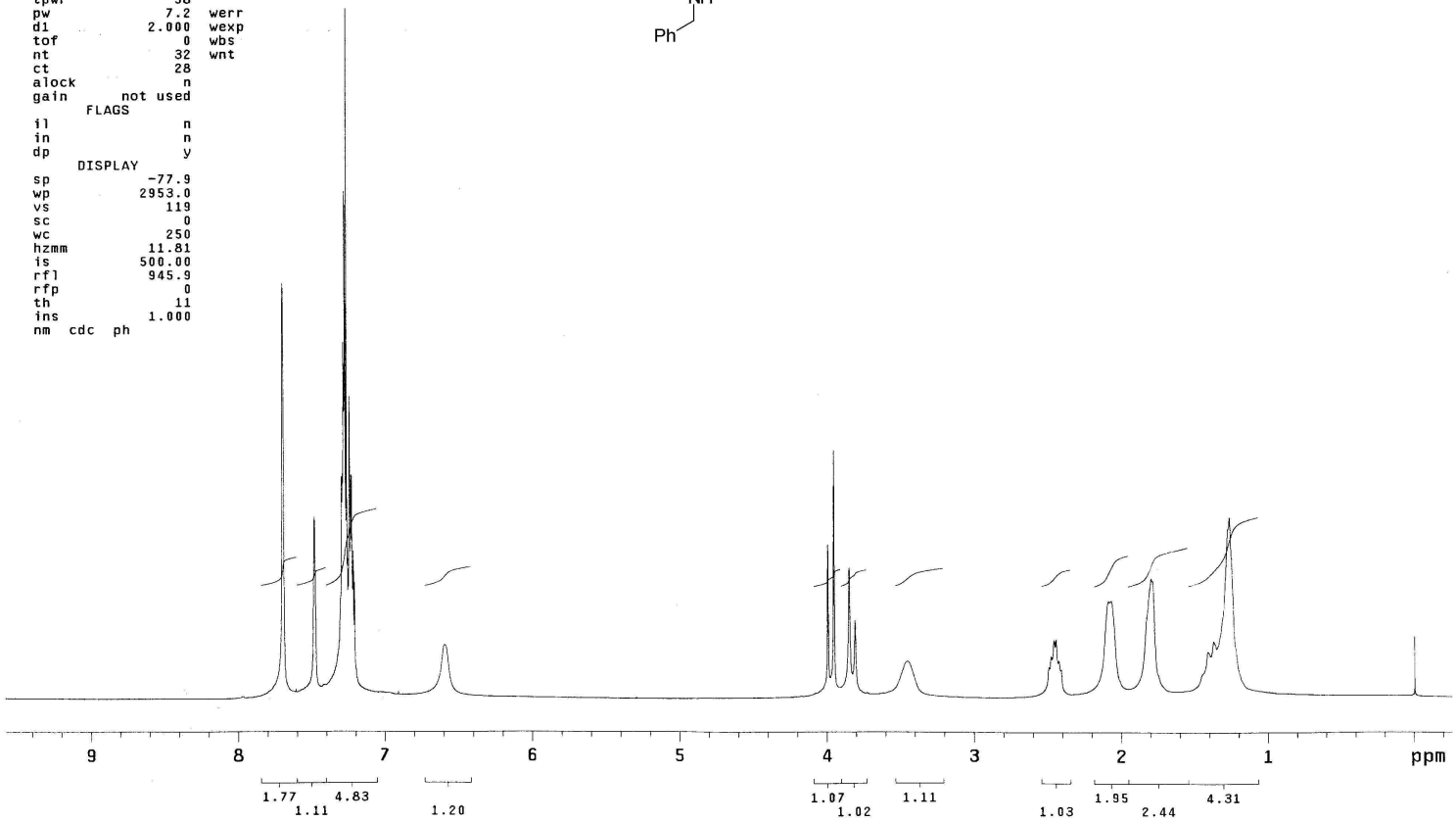

exp1 std13c
SAMPLE
date Nov 27 2007
solvent CDCl3
file /data/falck/D~
rli/led-VII-90-29-~
C13.fid
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 100
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 20000
ct 12826
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -2423.0
wp 20000.0
vs 939
sc 0
wc 250
hzmm 80.00
is 500.00
rfl 2423.0
rfp 0
th 3
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
183.290
142.026
132.530
132.077
131.640
131.203
128.809
125.193
122.968
121.577
117.961
117.734
77.709
77.490
77.288
76.867
63.342
56.838
35.102
32.328
25.169
24.780
22.645
ppm

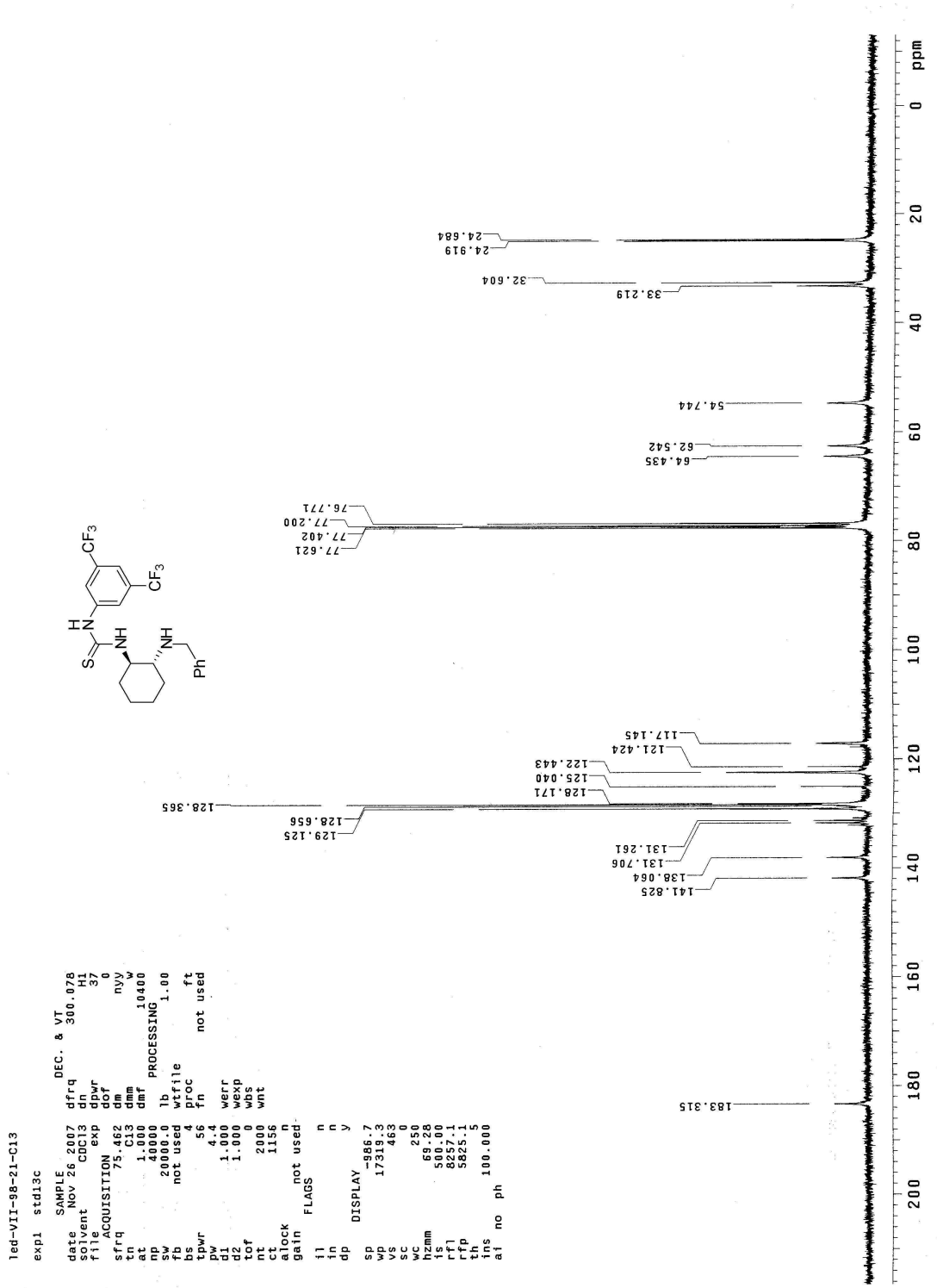
led-VII-98-21

exp1 stdlh

SAMPLE		DEC. & VT	
date	Nov 24 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58	werr	
pw	7.2	wexp	
d1	2.000	wbs	
tof	0	wnt	
nt	32		
ct	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-77.9		
wp	2953.0		
vs	119		
sc	0		
wc	250		
hzmm	11.81		
is	500.00		
rfl	945.9		
rfp	0		
th	11		
ins	1.000		
nm cdc ph			





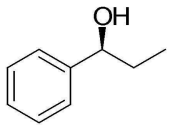

led-VII-98-21-C13
expl std13c
SAMPLE
date Nov 26 2007
solvent CDCl3
file ACQUISITION
sfrq 75.462
tn C13
at 1.000
nt 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 2000
ct 1156
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -986.7
wp 17319.3
vs 463
sc 0
wc 250
hzmm 69.28
is 500.00
rfl 8257.1
rfp 5825.1
th 5
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dnuc H1
dpwr 37
dof 0
dm nyy
dmm y
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc
fn not used
werr
wexp
wbs
wnt
CF3
CF3
H
N
S
NH
NH
Ph
183.315
141.825
138.064
131.706
131.261
129.125
128.656
128.365
128.171
125.040
122.443
121.424
117.145
77.621
77.402
77.200
76.771
64.435
62.542
54.744
33.219
32.604
24.919
24.684
ppm

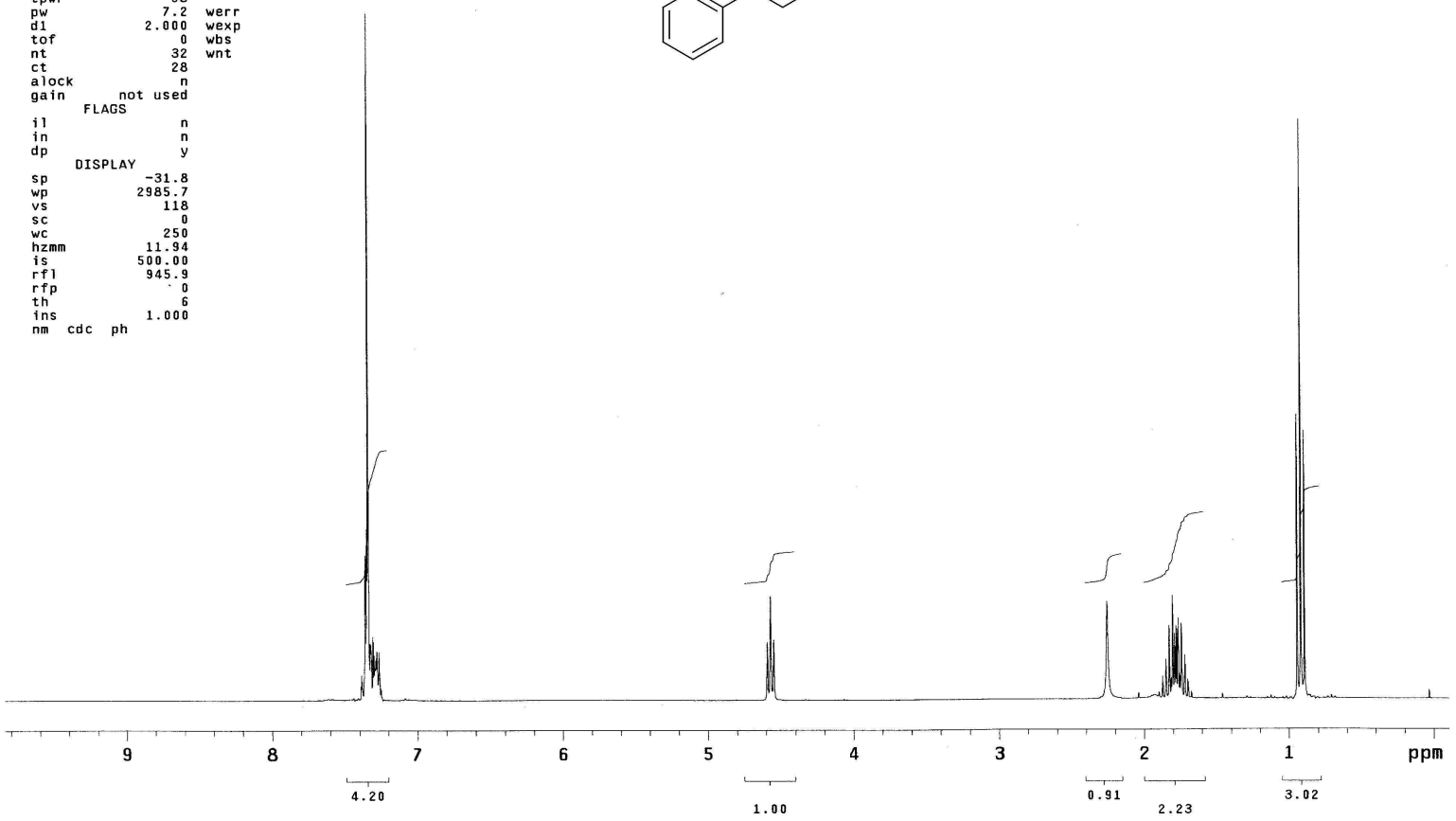
led-VII-126-29

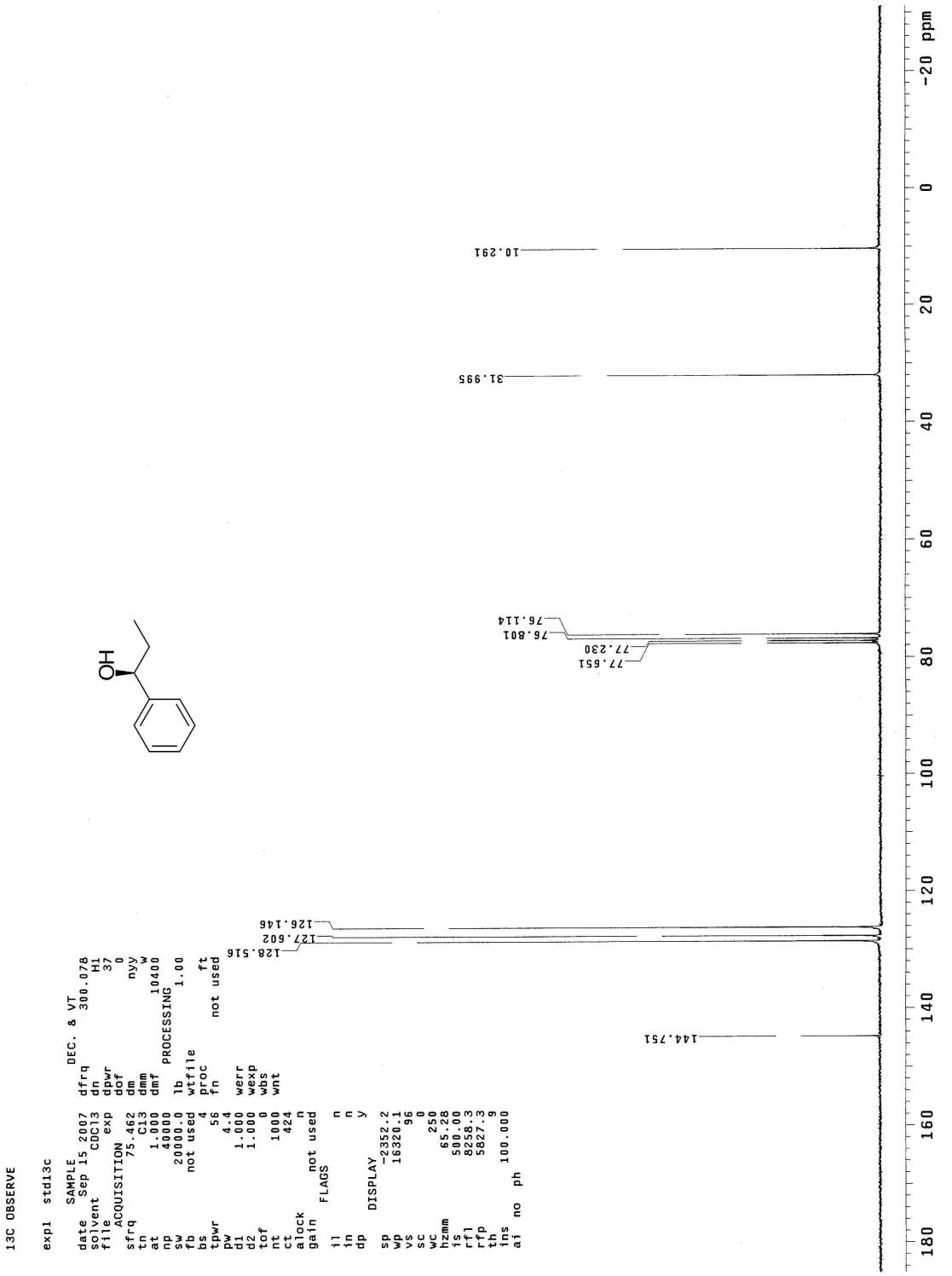
expl std1h

SAMPLE		DEC. & VT	
date	Sep 15 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-31.8		
wp	2985.7		
vs	118		
sc	0		
wc	250		
hzmm	11.94		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	1.000		

nm cdc ph



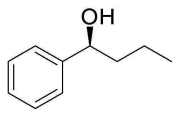


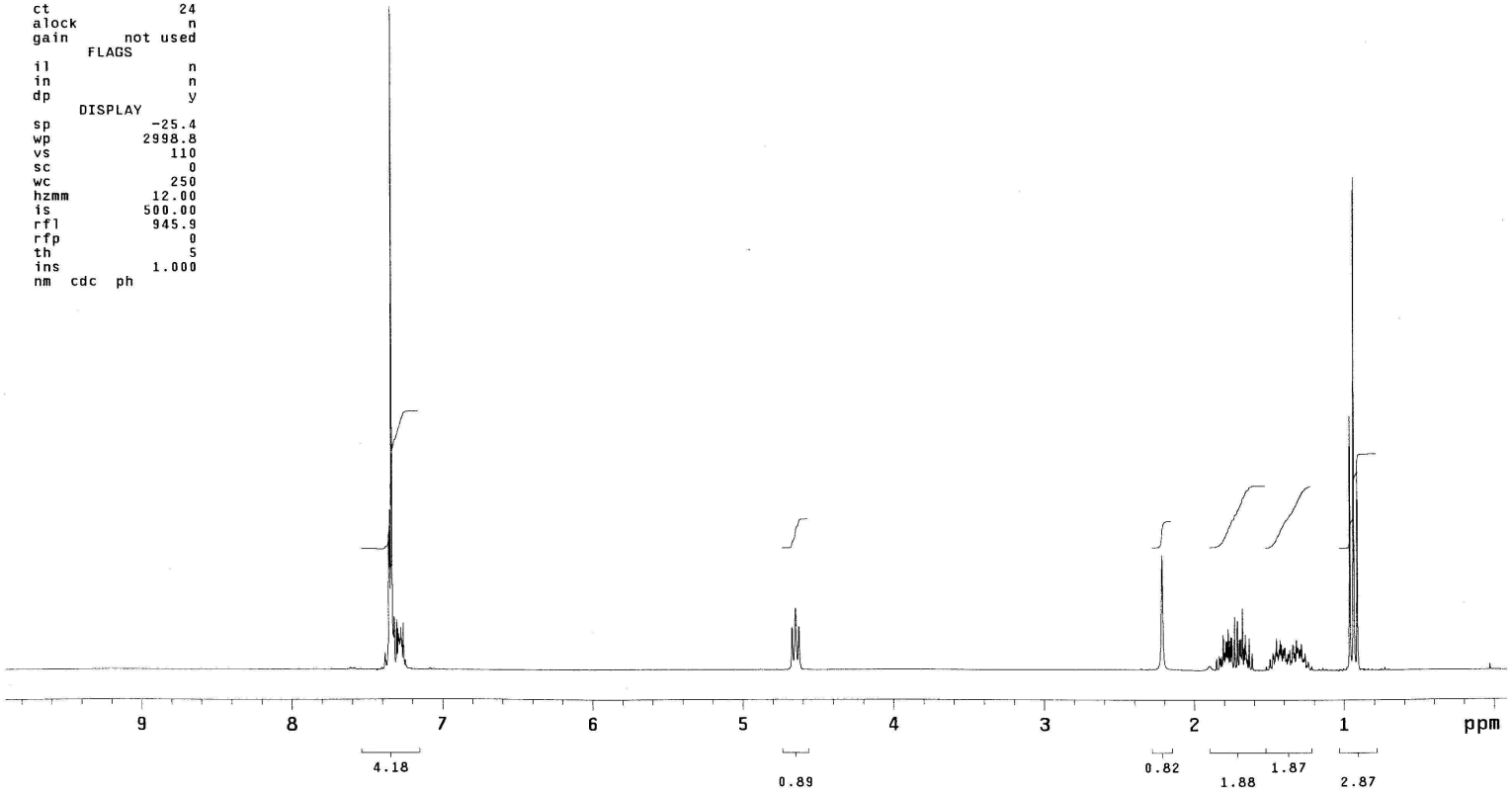

13C OBSERVE
expl std13c
SAMPLE
date Sep 15 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs not used
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 424
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -2352.2
wp 16320.1
vs 96
sc 0
wc 250
hzmm 65.28
rfl 5000.0
rfp 8258.3
th 5827.3
ins 1
ai no ph 100.000
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
126.146
127.602
128.516
144.751
10.291
31.995
76.114
76.801
77.230
77.651
180
160
140
120
100
80
60
40
20
0
-20 ppm

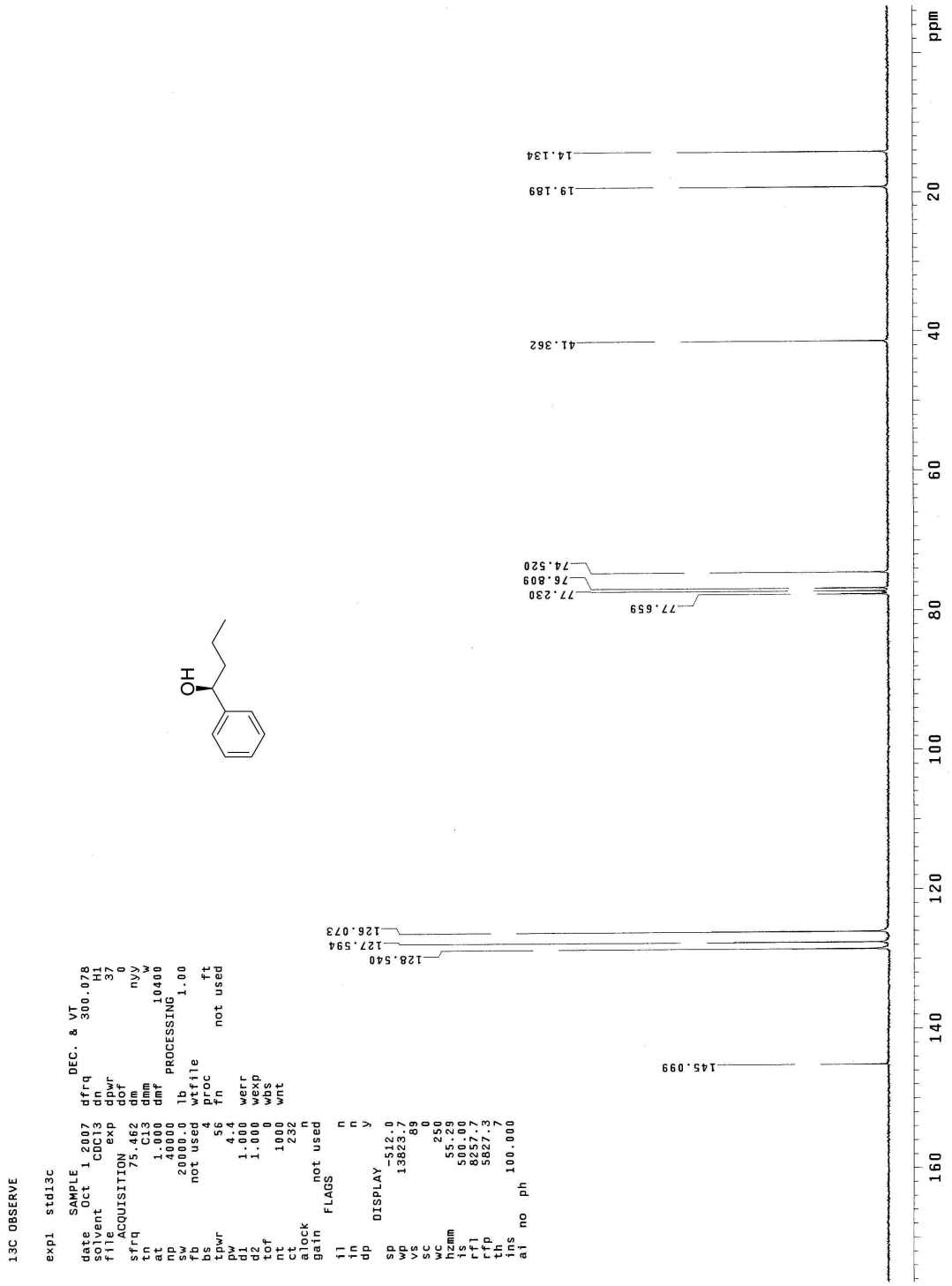
led-VII-141-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 1 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-25.4		
wp	2998.8		
vs	110		
sc	0		
wc	250		
hzmm	12.00		
is	500.00		
rfl	945.9		
rfp	0		
th	5		
ins	1.000		
nm cdc ph			



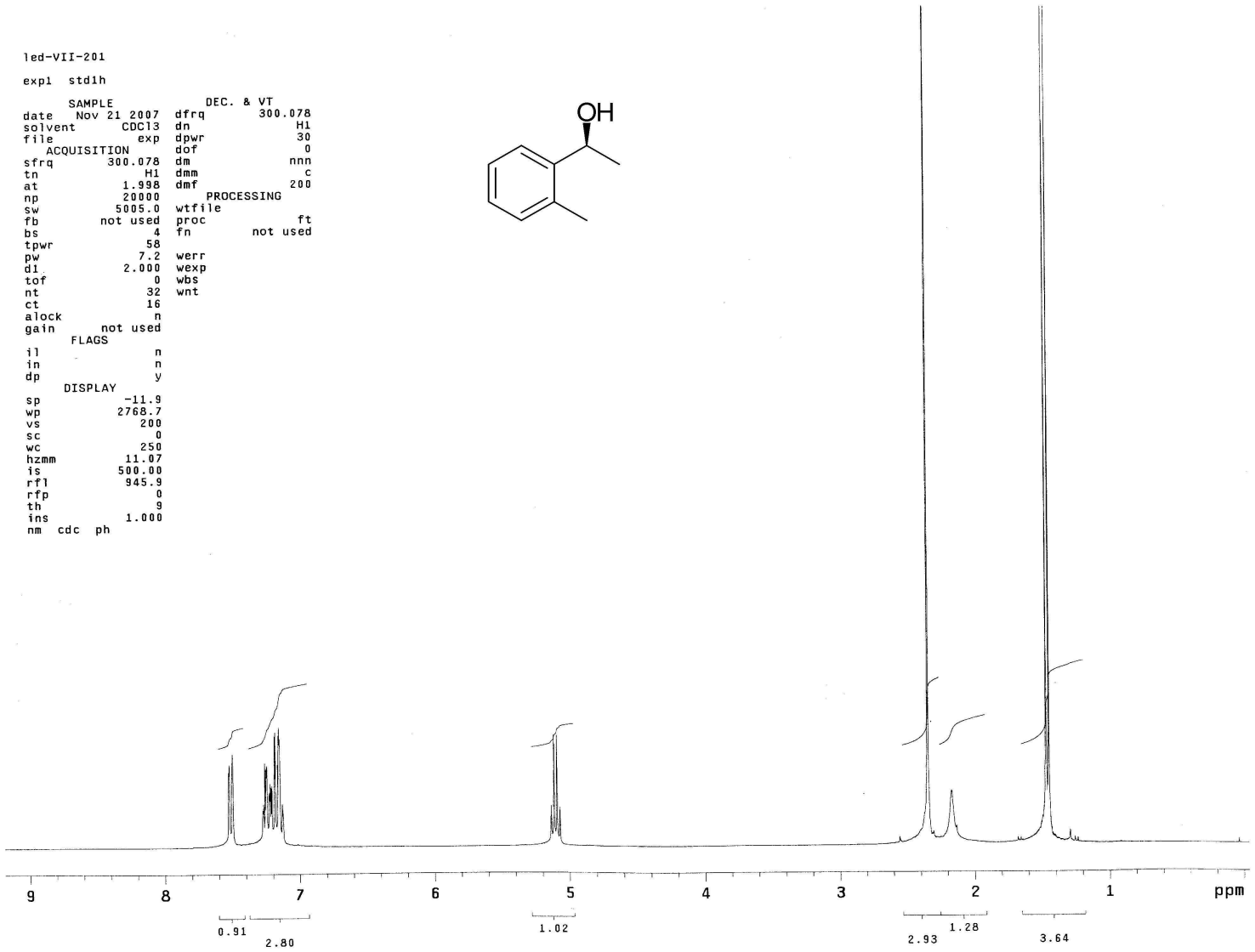



13C OBSERVE
exp1 std13c
SAMPLE
date Oct 1 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 232
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -512.0
wp 13823.7
vs 89
sc 0
wc 250
hzmm 55.29
is 500.00
rfl 8257.7
rfp 5827.3
th 7
ins 100.000
al no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc not used
fn
werr
wexp
wbs
wnt
OH
145.099
128.540
127.594
126.073
77.659
77.230
76.809
74.520
41.362
19.189
14.134
160
140
120
100
80
60
40
20
ppm

led-VII-201

expl std1h

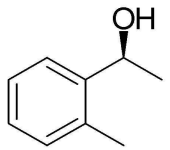
SAMPLE		DEC. & VT	
date	Nov 21 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	7.2	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-11.9		
wp	2768.7		
vs	200		
sc	0		
wc	250		
hzmm	11.07		
is	500.00		
rfl	945.9		
rfp	0		
th	9		
ins	1.000		
nm cdc ph			

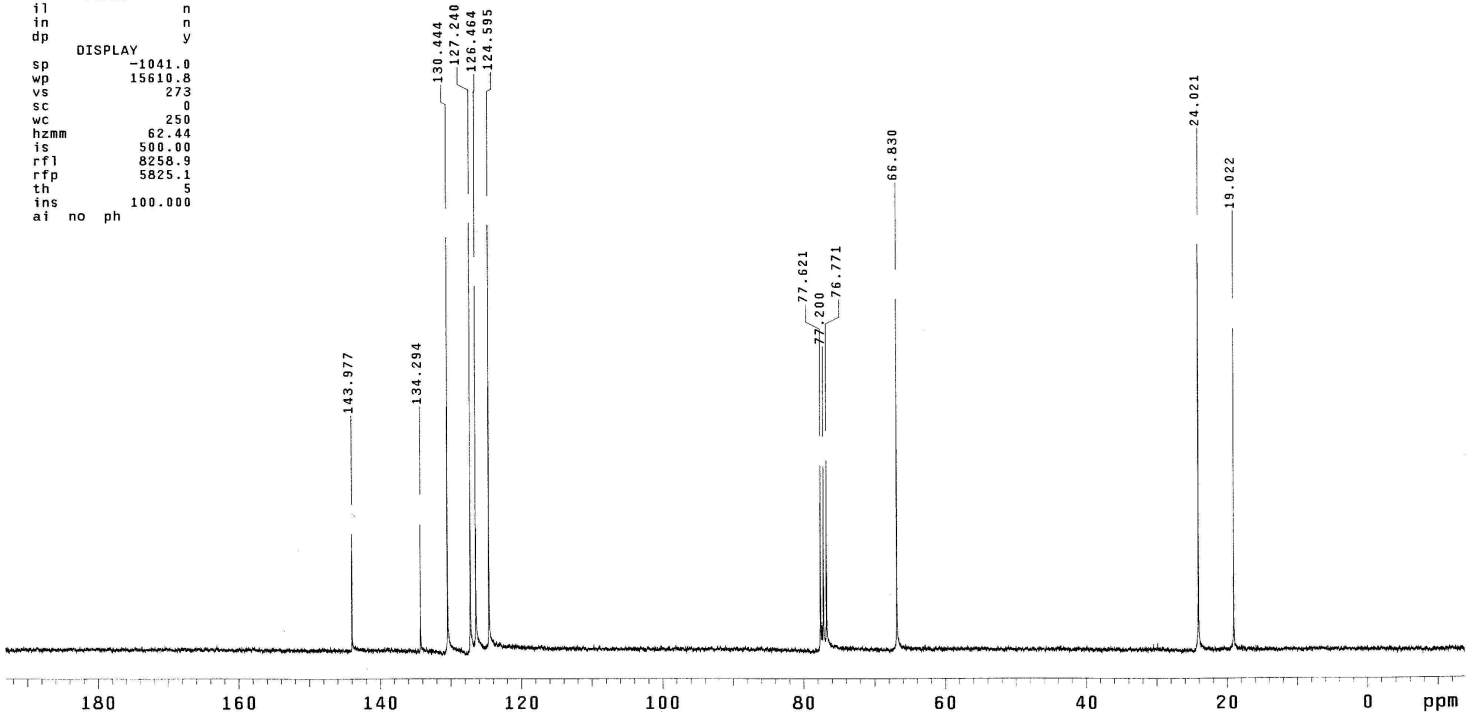


led-VII-201-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Nov 21 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	1.000	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	1000	wnt	
ct	892		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1041.0		
wp	15610.8		
vs	273		
sc	0		
wc	250		
hzmm	62.44		
is	500.00		
rfl	8258.9		
rfp	5825.1		
th	5		
ins	100.000		
ai no ph			

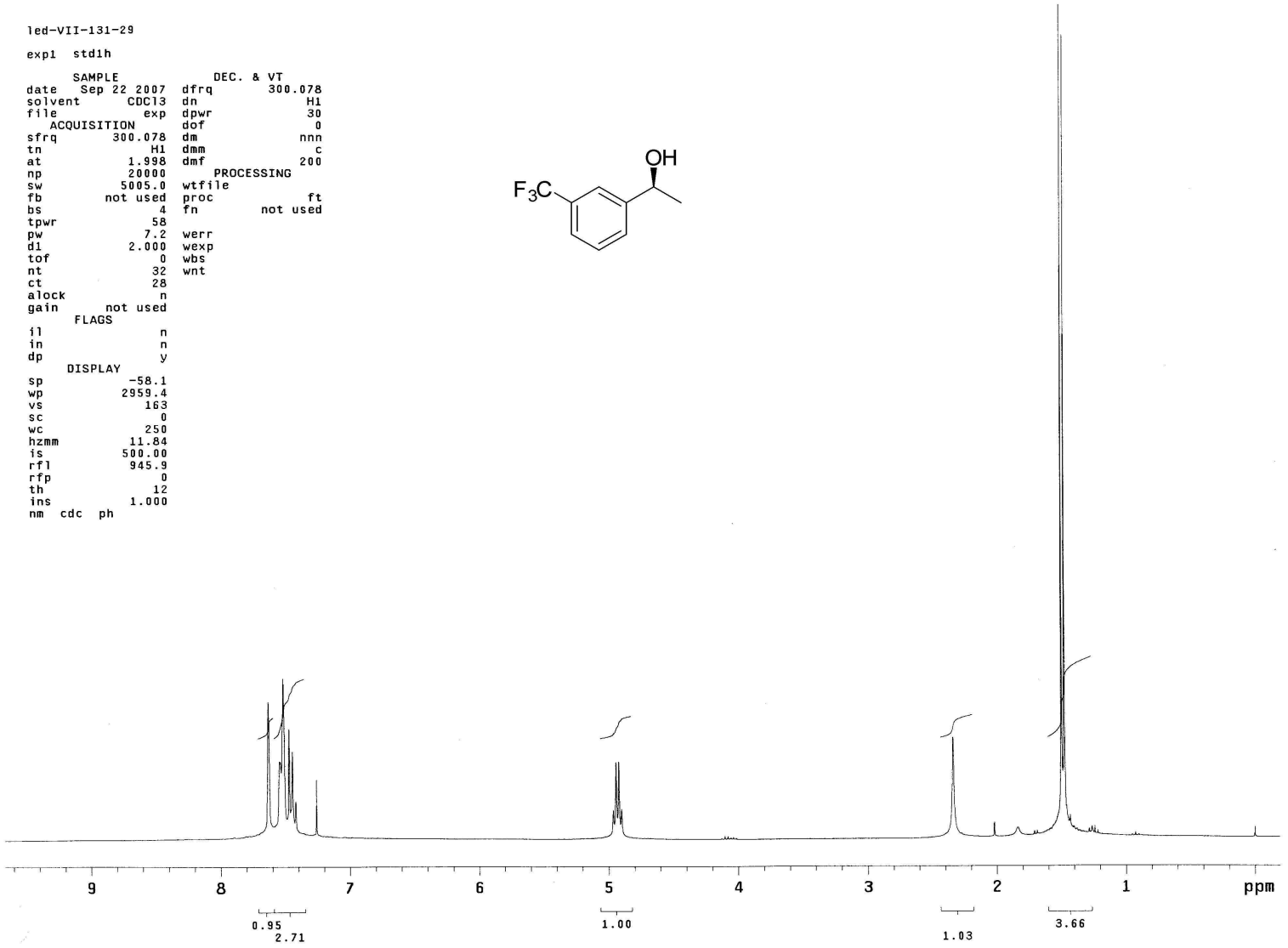


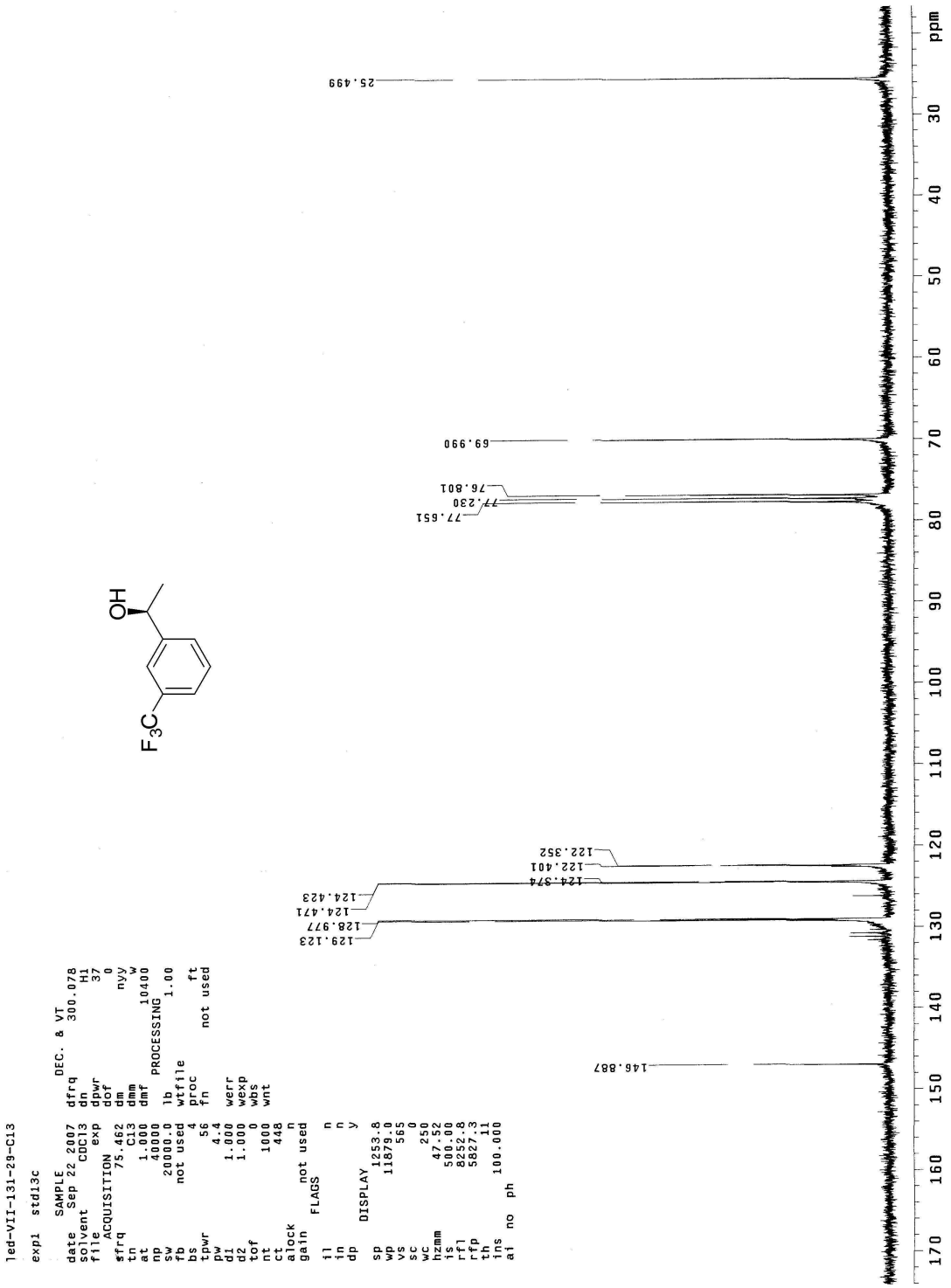


led-VII-131-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Sep 22 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-58.1		
wp	2959.4		
vs	163		
sc	0		
wc	250		
hzmm	11.84		
is	500.00		
rfl	945.9		
rfp	0		
th	12		
ins	1.000		
nm cdc ph			

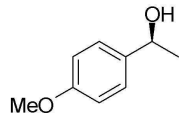


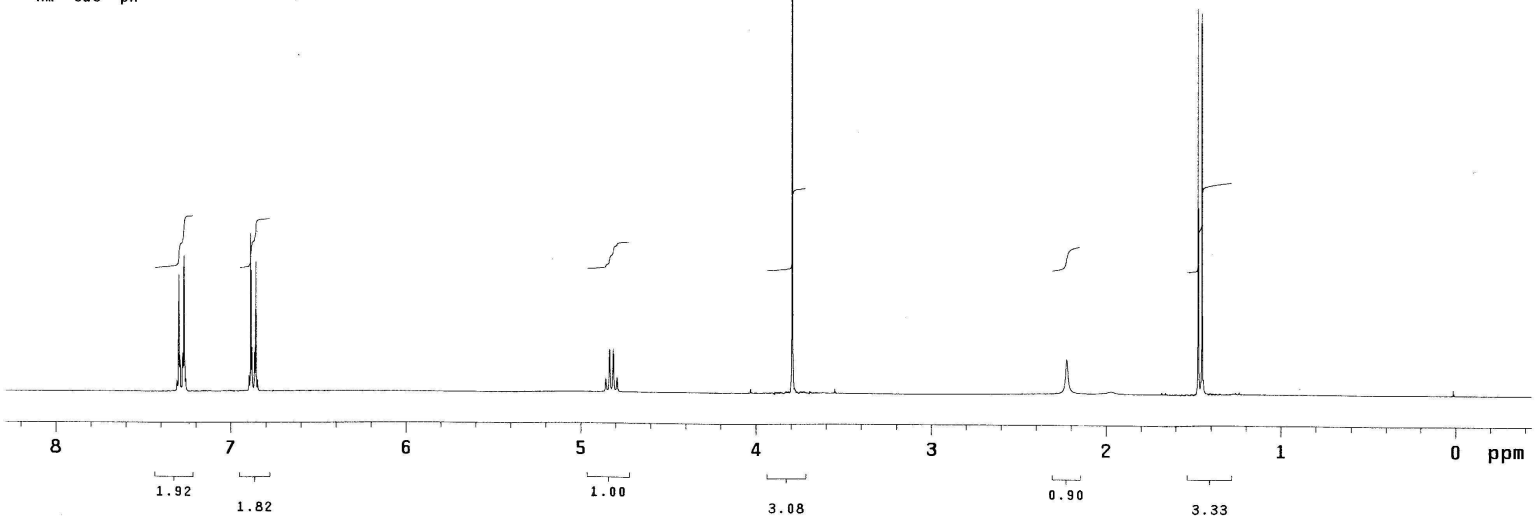

led-VII-131-29-C13
exp1 std13c
SAMPLE
date Sep 22 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 448
alock n
gain not used
FLAGS
fl n
in n
dp y
DISPLAY
sp 1253.8
wp 11879.0
vs 565
sc 0
wc 250
hzmm 47.52
is 500.00
rfl 8252.8
rfp 5827.3
th 11
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
F3C
25.499
69.990
76.801
77.230
77.651
122.352
122.401
124.374
124.423
124.471
128.977
129.123
146.887
ppm

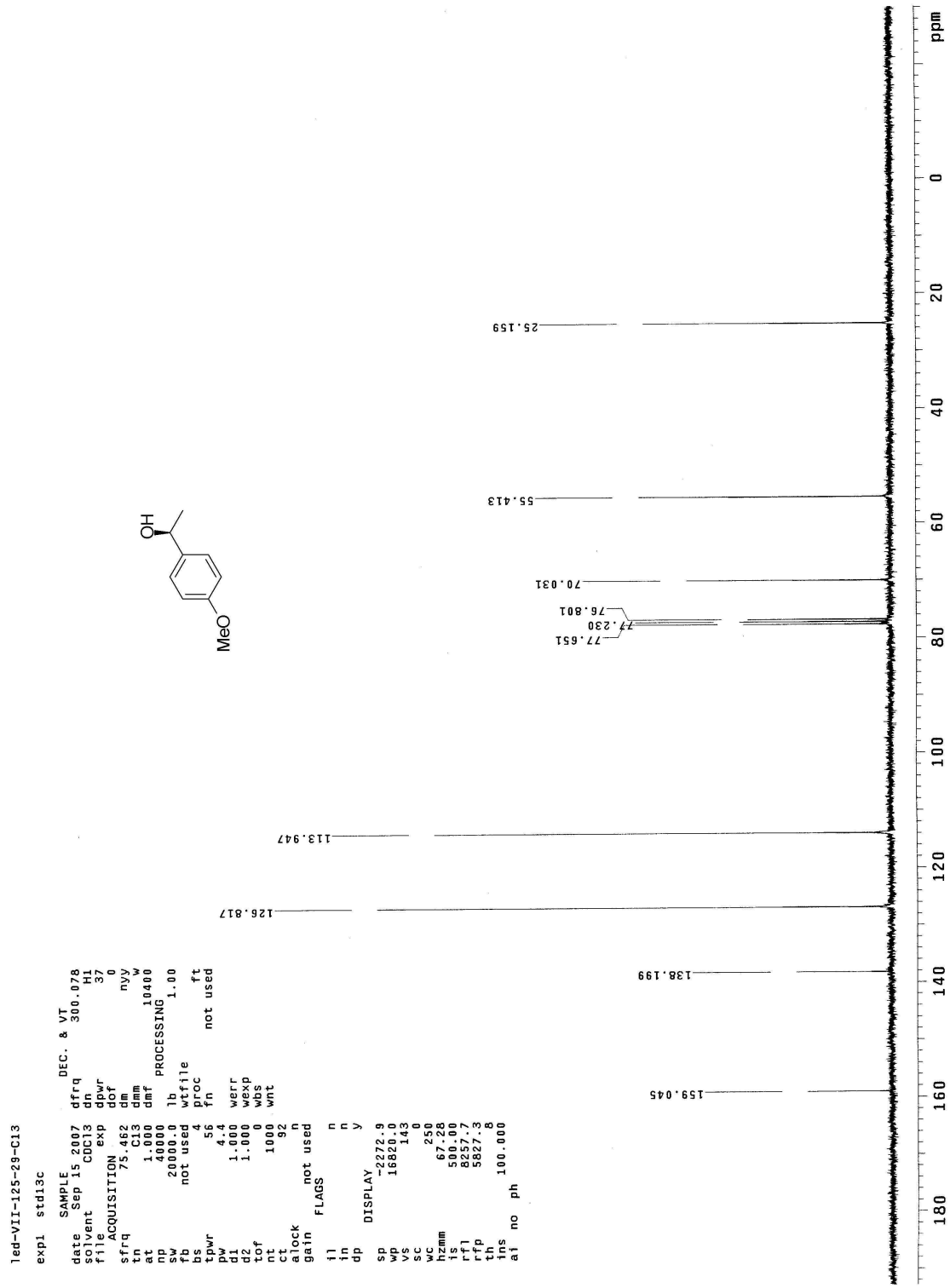
led-VII-125-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Sep 15 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-130.5		
wp	2617.5		
vs	133		
sc	0		
wc	250		
hzmm	10.47		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	1.000		
nm cdc ph			



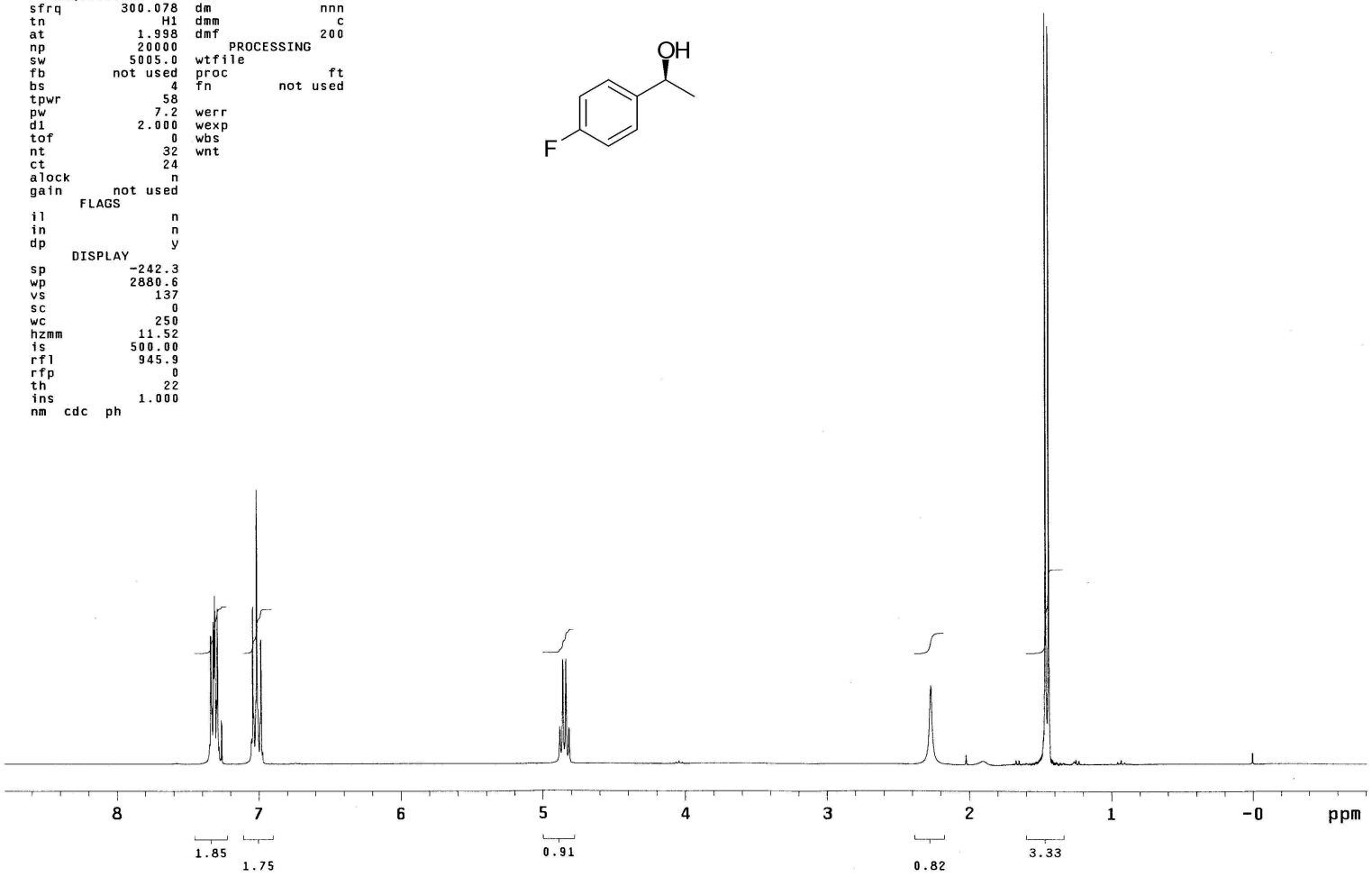


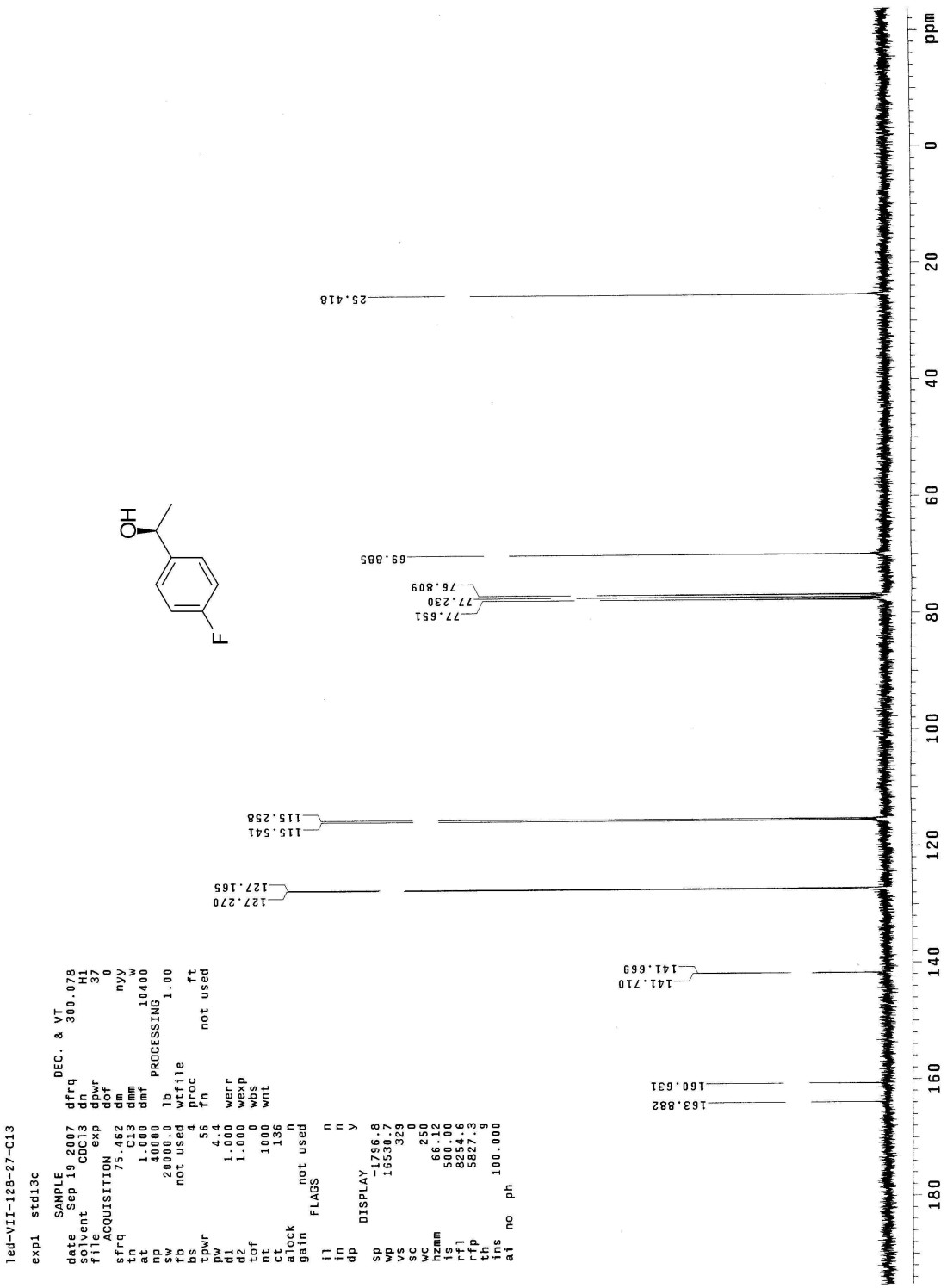

led-VII-125-29-C13
exp1 std13c
SAMPLE
date Sep 15 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb
bs not used
tpwr 4
pw 56
d1 4.4
d2 1.000
tof 0
nt 1000
ct 92
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -2272.9
wp 16820.0
vs 143
sc 0
wc 250
hzmm 67.28
is 500.00
rfl 8257.7
rfp 5827.3
th 8
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc
fn not used
werr
wexp
wbs
wnt
OH
MeO
159.045
138.199
126.817
113.947
77.651
77.230
76.801
70.031
55.413
25.159
ppm

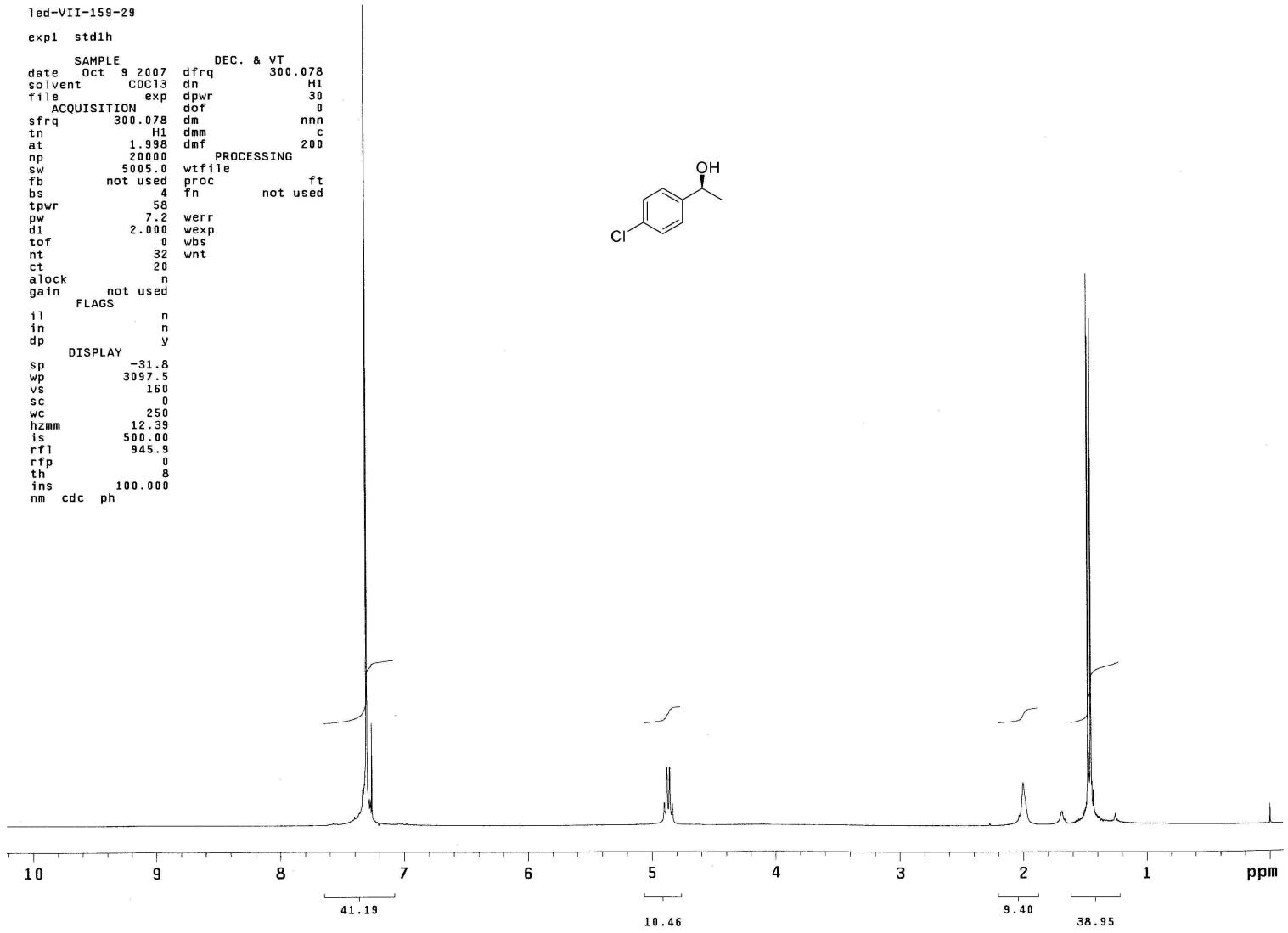
led-VII-128-27

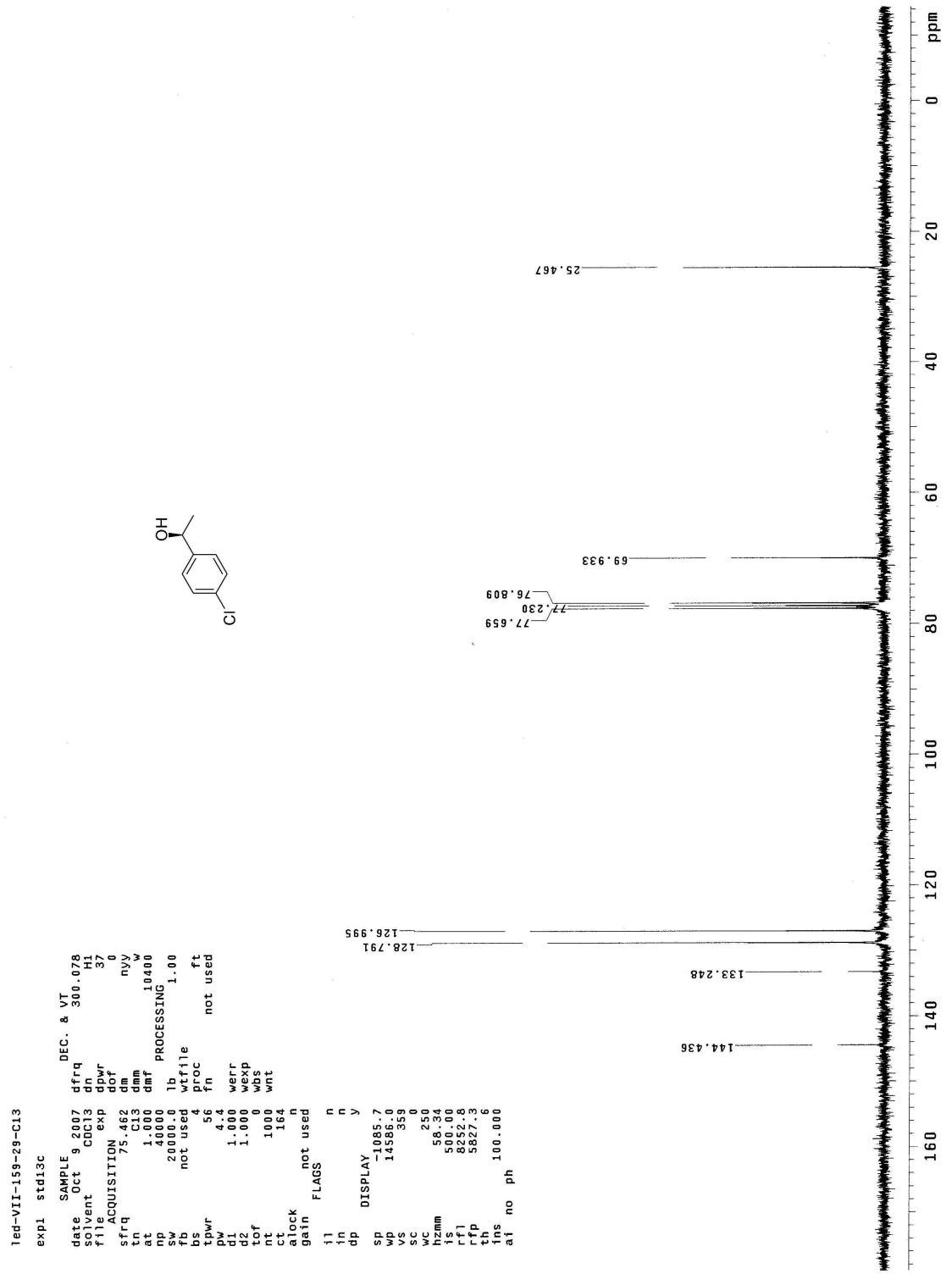
exp1 stdlh

SAMPLE		DEC. & VT	
date	Sep 19 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-242.3		
wp	2880.6		
vs	137		
sc	0		
wc	250		
hzmm	11.52		
is	500.00		
rfl	945.9		
rfp	0		
th	22		
ins	1.000		
nm cdc ph			




led-VII-128-27-C13
exp1 std13c
SAMPLE
DEC. & VT
date Sep 19 2007
dfrq 300.078
solvent CDCl3
dn H1
file exp
dpwr 37
ACQUISITION
dof 0
sfrq 75.462
dm nyy
tn C13
dmm w
at 1.000
dmf 10400
np 40000
PROCESSING
sw 20000.0
lb 1.00
fb not used
wtfile
bs 4
proc ft
tpwr 56
fn not used
pw 4.4
d1 1.000
werr
d2 1.000
wexp
tof 0
wbs
nt 1000
wnt
ct 136
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1796.8
wp 16530.7
vs 329
sc 0
wc 250
hzmm 66.12
is 500.00
rfl 8254.6
rfp 5827.3
th 9
ins 100.000
ai no ph
OH
F
25.418
69.885
76.809
77.230
77.651
115.258
115.541
127.165
127.270
141.669
141.710
160.631
163.882
ppm


led-VII-159-29
exp1 std1h
SAMPLE
date Oct 9 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
d1 2.000
tof 0
nt 32
ct 20
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -31.8
wp 3097.5
vs 160
sc 0
wc 250
hzmm 12.39
is 500.00
rfl 945.9
rfp 0
th 8
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
Cl
10
9
8
7
6
5
4
3
2
1
ppm
41.19
10.46
9.40
38.95

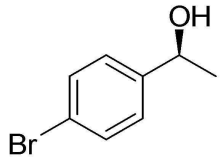

led-VII-159-29-C13
exp1 std13c
SAMPLE
date Oct 9 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 164
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1085.7
wp 14586.0
vs 359
sc 0
wc 250
hzmm 58.34
is 500.00
rfl 8252.8
rfp 5827.3
th 6
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc
fn not used
werr
wexp
wbs
wnt
OH
Cl
144.436
133.248
128.791
126.995
77.659
77.230
76.809
69.933
25.467
ppm

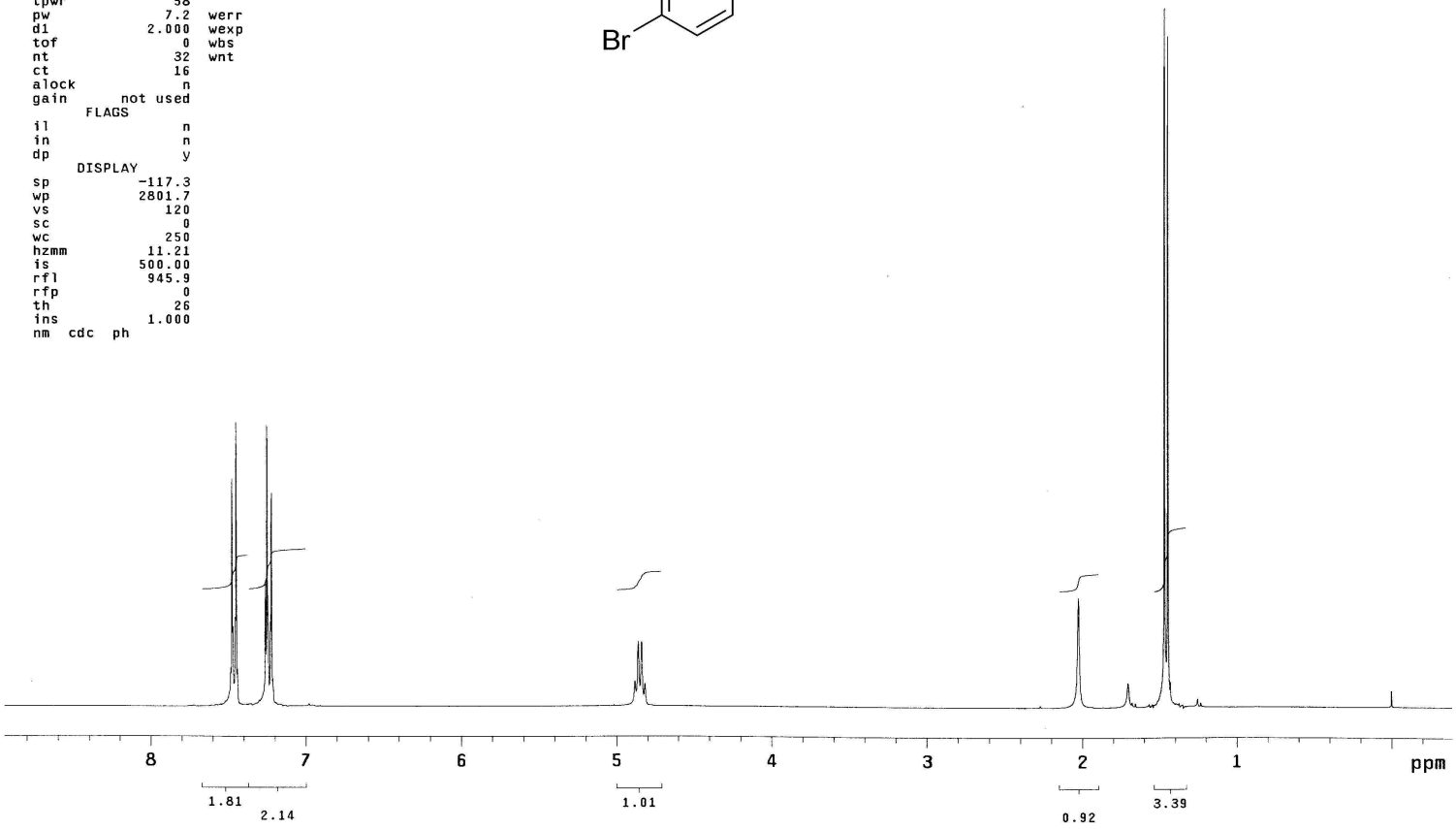
led-VII-161-59

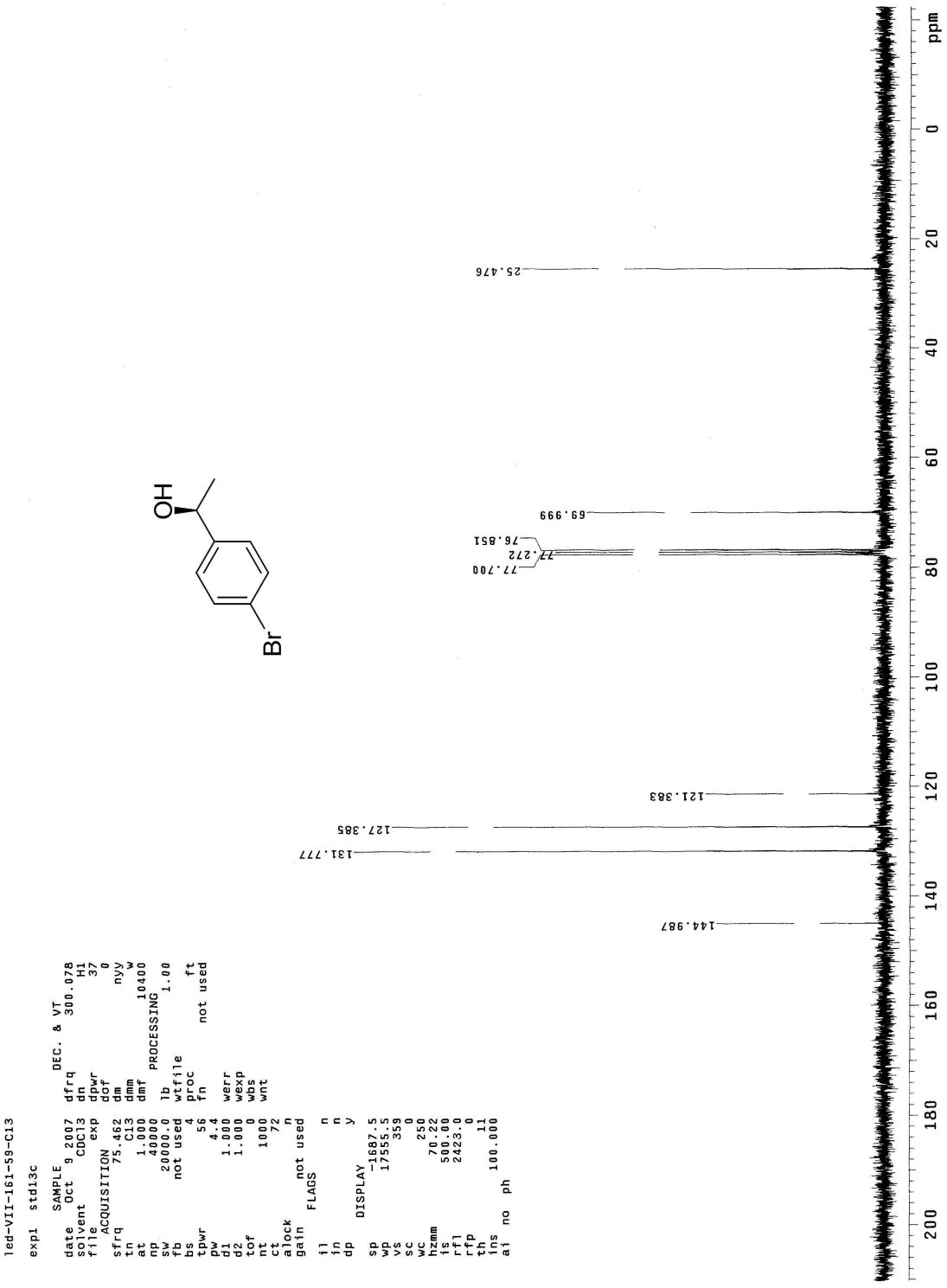
exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 9 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-117.3		
wp	2801.7		
vs	120		
sc	0		
wc	250		
hzmm	11.21		
is	500.00		
rfl	945.9		
rfp	0		
th	26		
ins	1.000		

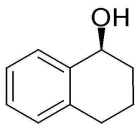
nm cdc ph

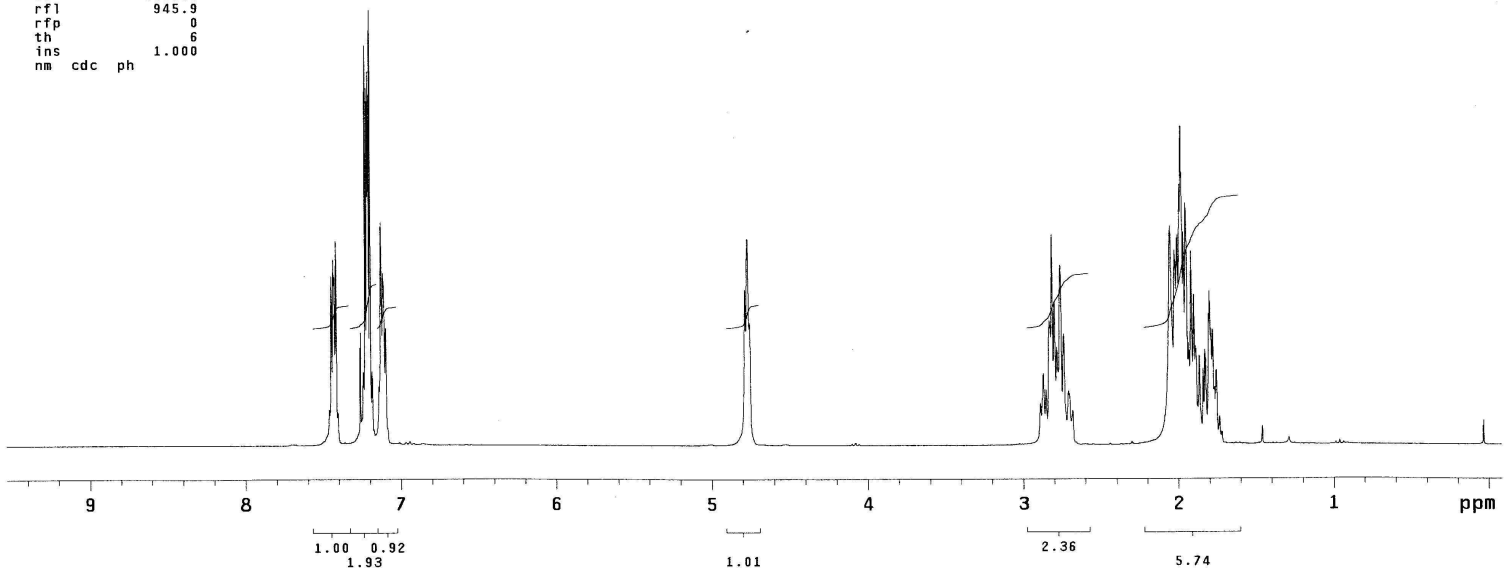


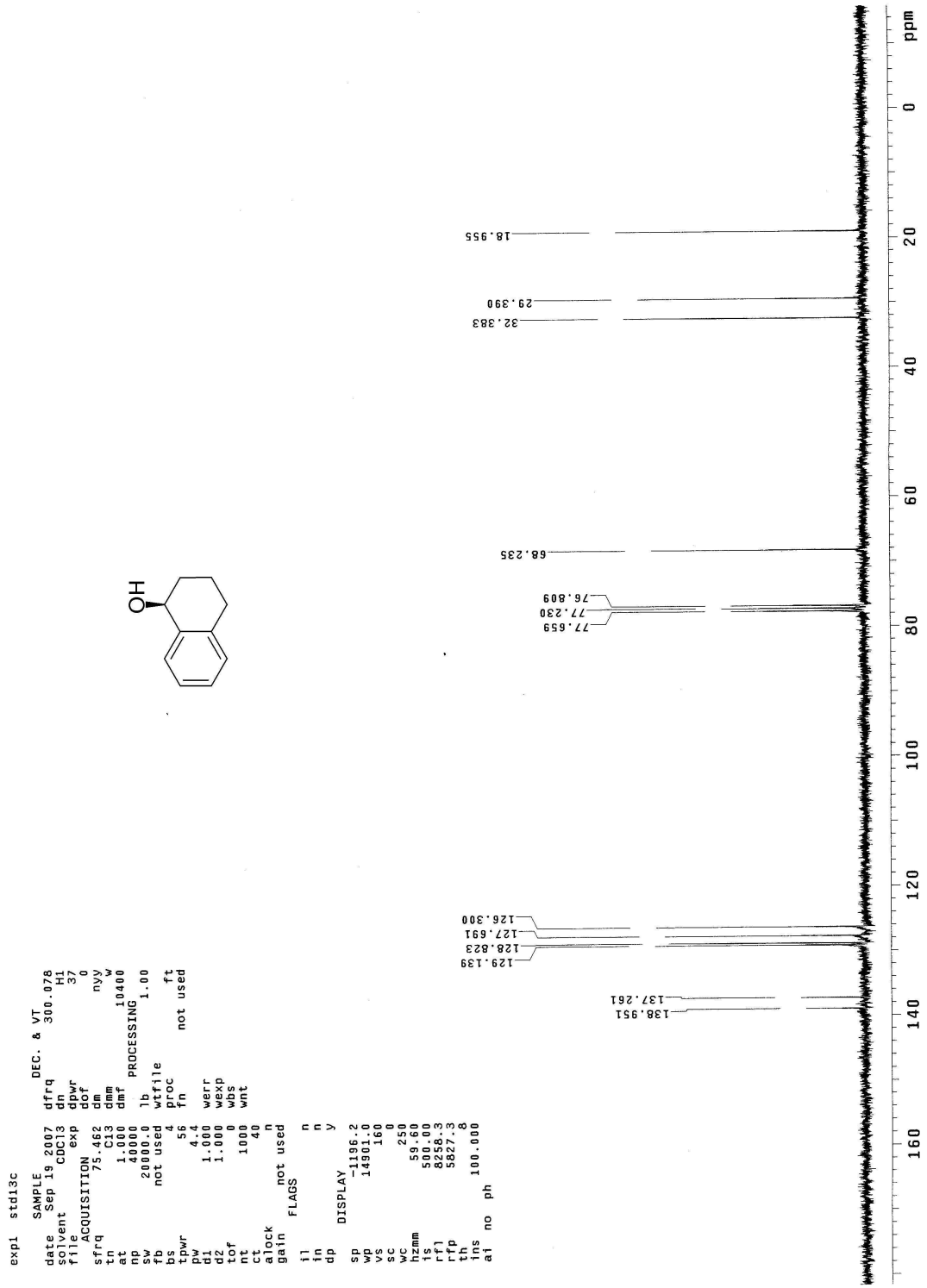



led-VII-161-59-C13
exp1 std13c
SAMPLE
date Oct 9 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 72
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1687.5
wp 17555.5
vs 359
sc 0
wc 250
hzmm 70.22
is 500.00
rfl 2423.0
rfp 0
th 11
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
Br
144.987
131.777
127.385
121.383
77.700
77.272
76.851
69.999
25.476
ppm

```
exp1  std1h

      SAMPLE
date   Sep 19 2007
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        300.078
tn               H1
at              1.998
np              20000
sw             5005.0
fb          not used
bs                 4
tpwr              58
pw               7.2
d1             2.000
tof                0
nt                32
ct                20
alock              n
gain        not used
      FLAGS
il                 n
in                 n
dp                 y
     DISPLAY
sp             -25.4
wp            2887.0
vs                73
sc                 0
wc               250
hzmm           11.55
is            500.00
rfl            945.9
rfp                0
th                 6
ins            1.000
nm  cdc  ph

      DEC. & VT
dfrq        300.078
dn               H1
dpwr             30
dof               0
dm             nnn
dmm                c
dmf              200
    PROCESSING
wtfile
proc              ft
fn          not used

werr
wexp
wbs
wnt
```



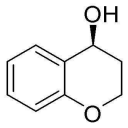


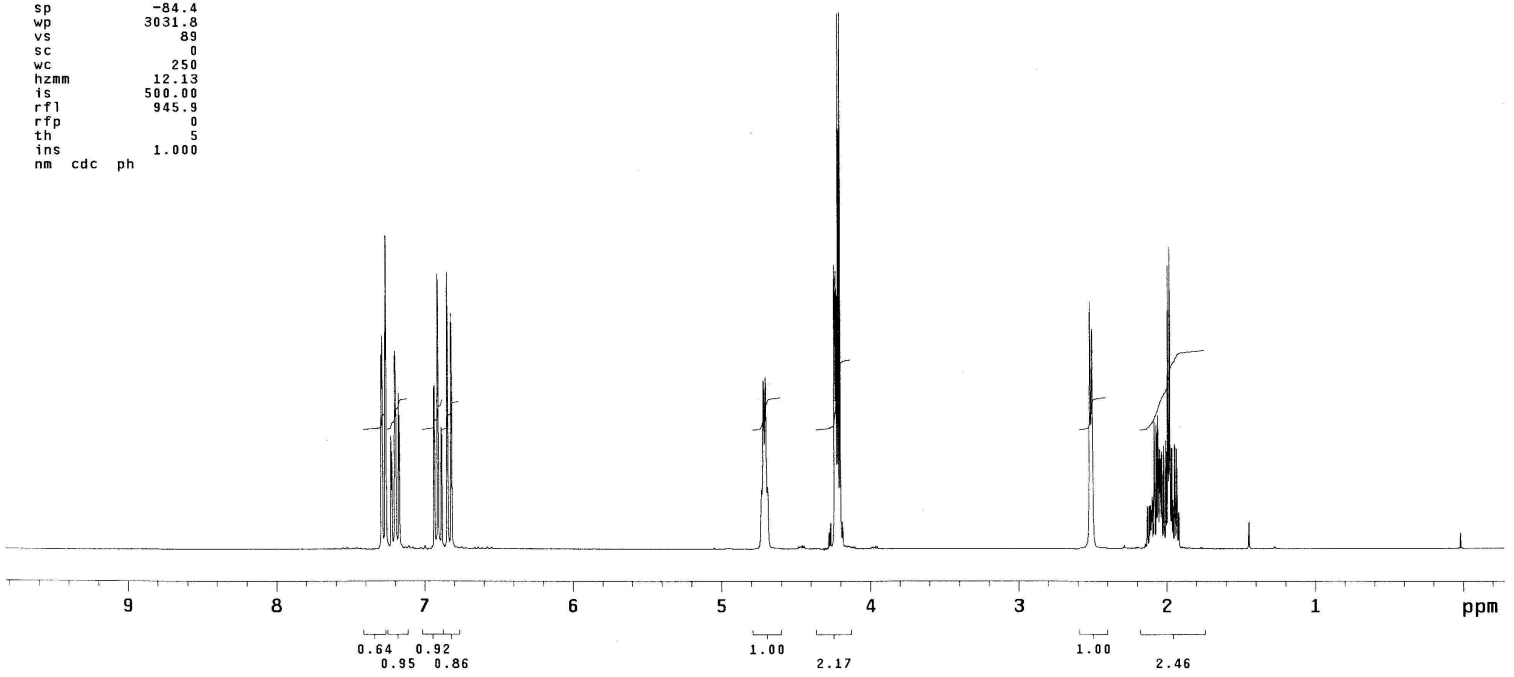

exp1 std13c
SAMPLE
date Sep 19 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs not used
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 40
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1196.2
wp 14901.0
vs 160
sc 0
wc 250
hzmm 59.60
is 500.00
rfl 8258.3
rfp 5827.8
th 8
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
18.955
29.390
32.383
68.235
76.809
77.230
77.659
126.300
127.691
128.823
129.139
137.261
138.951
ppm
0
20
40
60
80
100
120
140
160

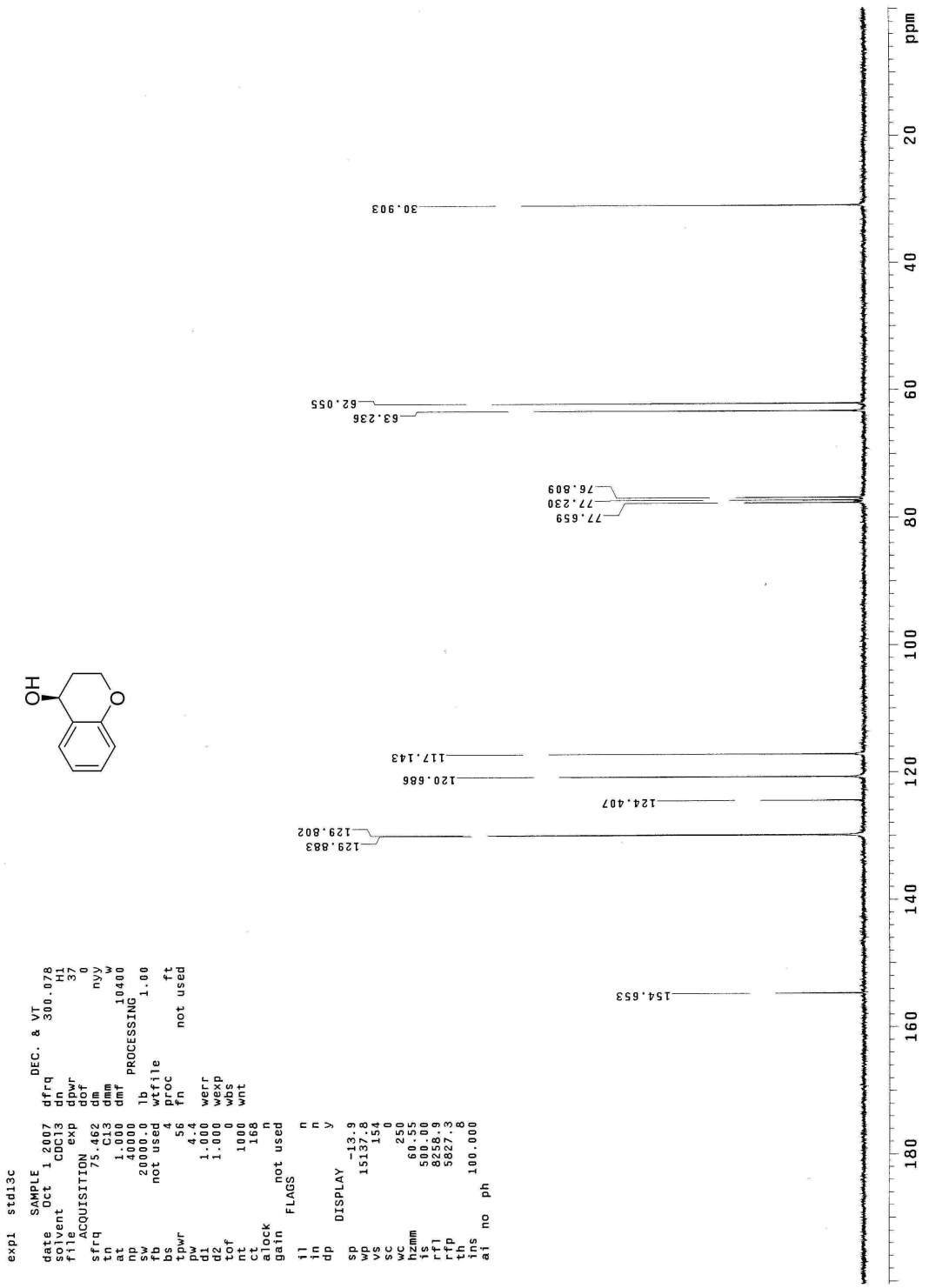
led-VII-140-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 1 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58	werr	
pw	7.2	wexp	
d1	2.000	wbs	
tof	0	wnt	
nt	32		
ct	24		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-84.4		
wp	3031.8		
vs	89		
sc	0		
wc	250		
hzmm	12.13		
is	500.00		
rfl	945.9		
rfp	0		
th	5		
ins	1.000		
nm cdc ph			



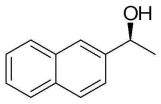


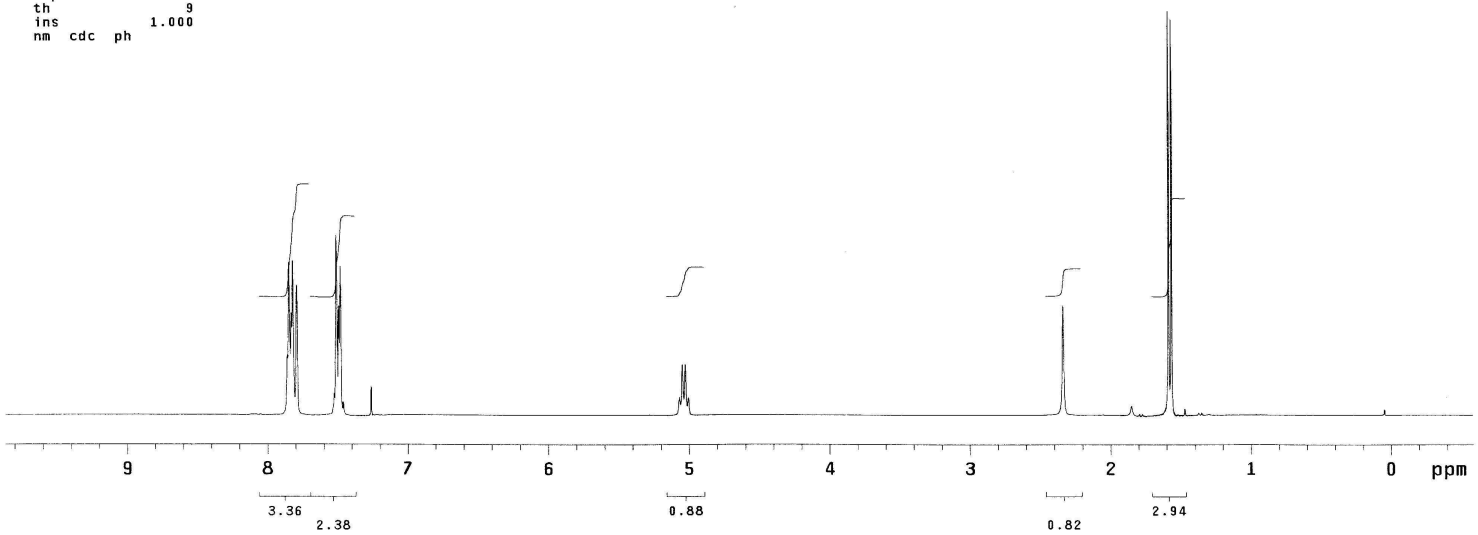

exp1 std13c
SAMPLE
date Oct 1 2007
solvent CDCl3
file ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 168
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -13.9
wp 15137.8
vs 154
sc 0
wc 250
hzmm 60.55
is 500.00
rfl 3259.9
rfp 5827.3
th 8
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc not used
ft
ffn not used
werr
wexp
wbs
wnt
154.653
129.883
129.802
124.407
120.686
117.143
77.659
77.230
76.809
63.236
62.055
30.903
ppm
180
160
140
120
100
80
60
40
20

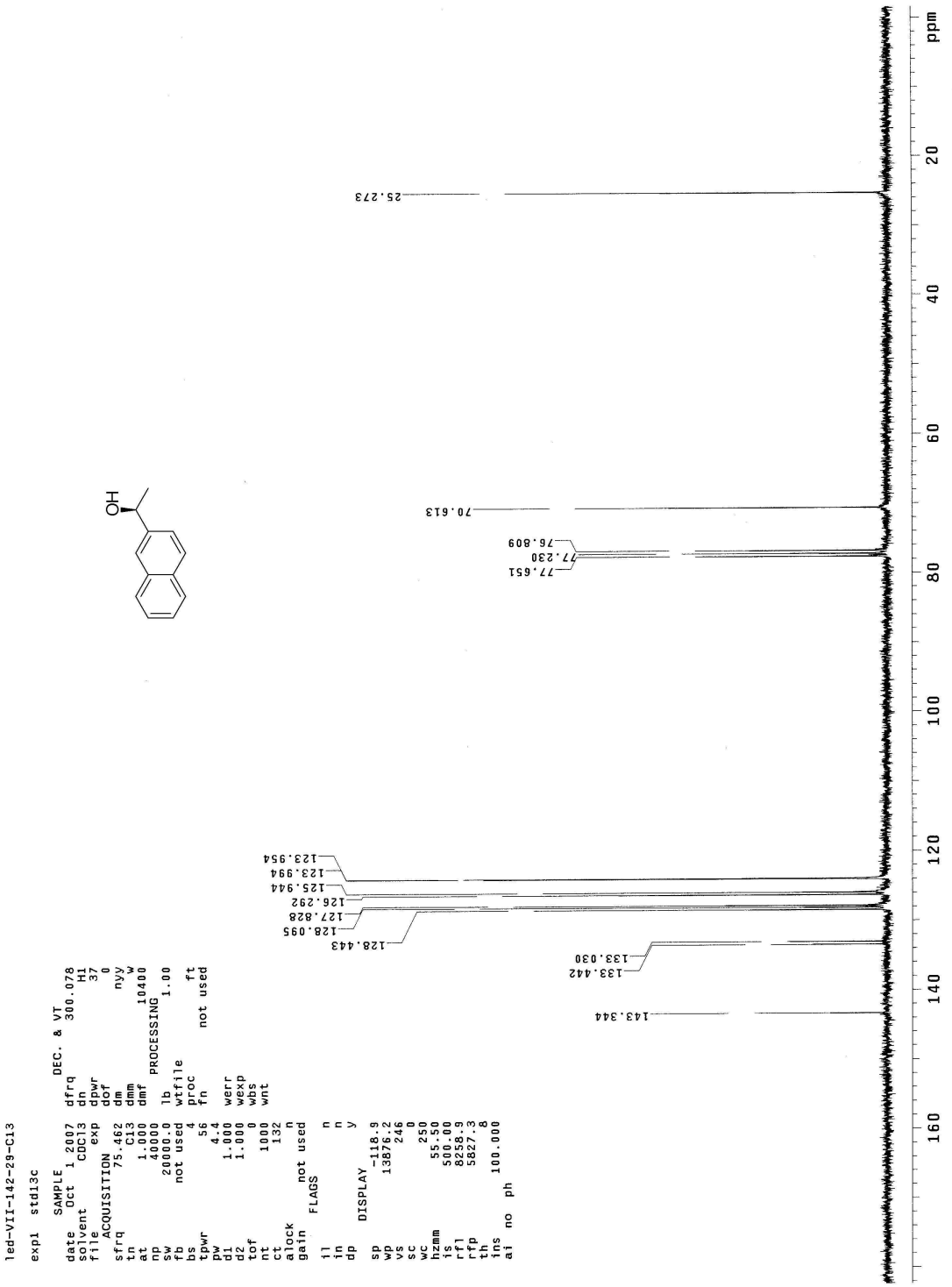
led-VII-142-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 1 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-176.6		
wp	3136.9		
vs	68		
sc	0		
wc	250		
hzmm	12.55		
is	733.67		
rfl	945.9		
rfp	0		
th	9		
ins	1.000		
nm cdc ph			



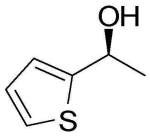


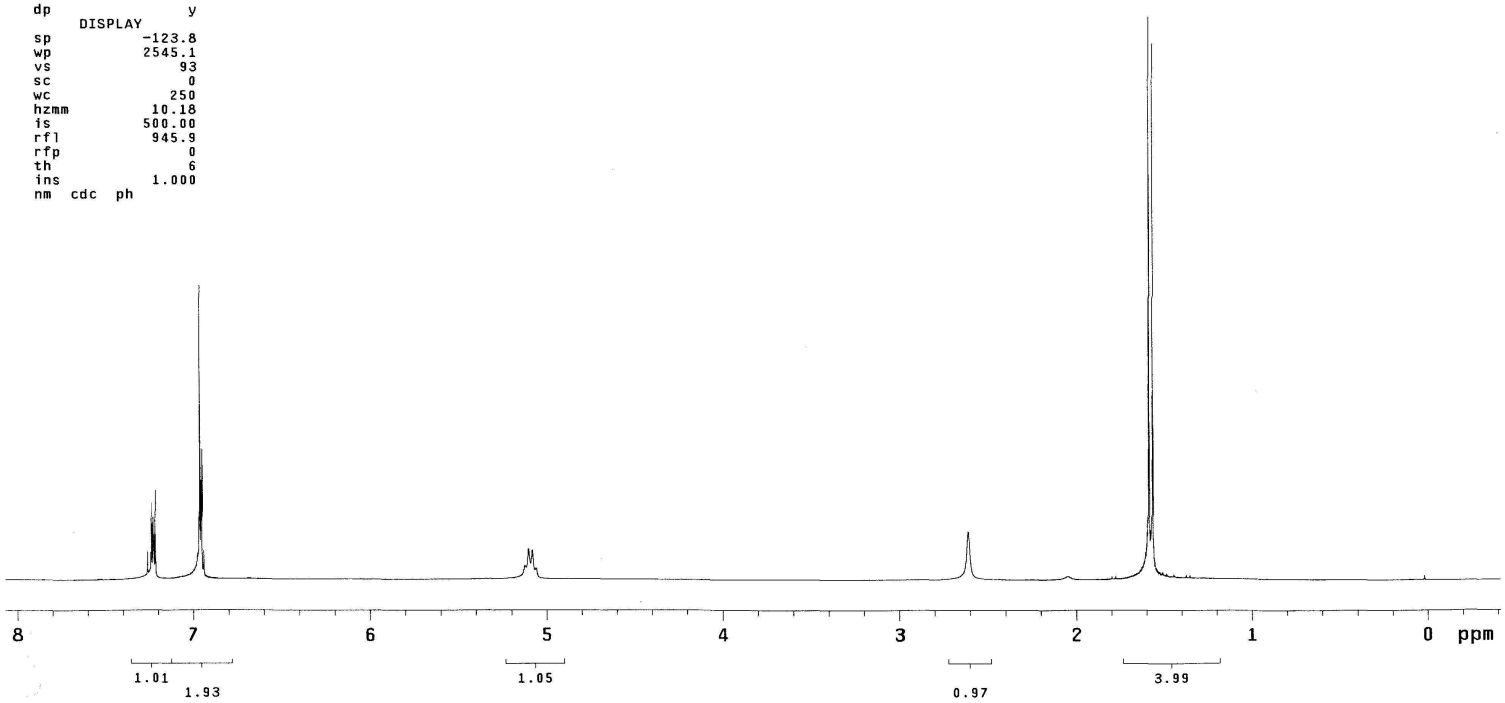

led-VII-142-29-C13
exp1 std13c
SAMPLE
date Oct 1 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 1000
ct 132
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -118.9
wp 13876.2
vs 246
sc 0
wc 250
hzmm 55.50
is 500.00
rfl 8258.9
rfp 5827.3
th 8
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
25.273
70.613
76.809
77.230
77.651
123.954
123.994
125.944
126.292
127.828
128.095
128.443
133.030
133.442
143.344
ppm

led-VII-133-29

exp1 std1h

SAMPLE		DEC. & VT	
date	Sep 22 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	20		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-123.8		
wp	2545.1		
vs	93		
sc	0		
wc	250		
hzmm	10.18		
is	500.00		
rfl	945.9		
rfp	0		
th	6		
ins	1.000		
nm cdc ph			

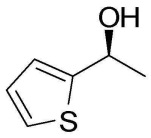


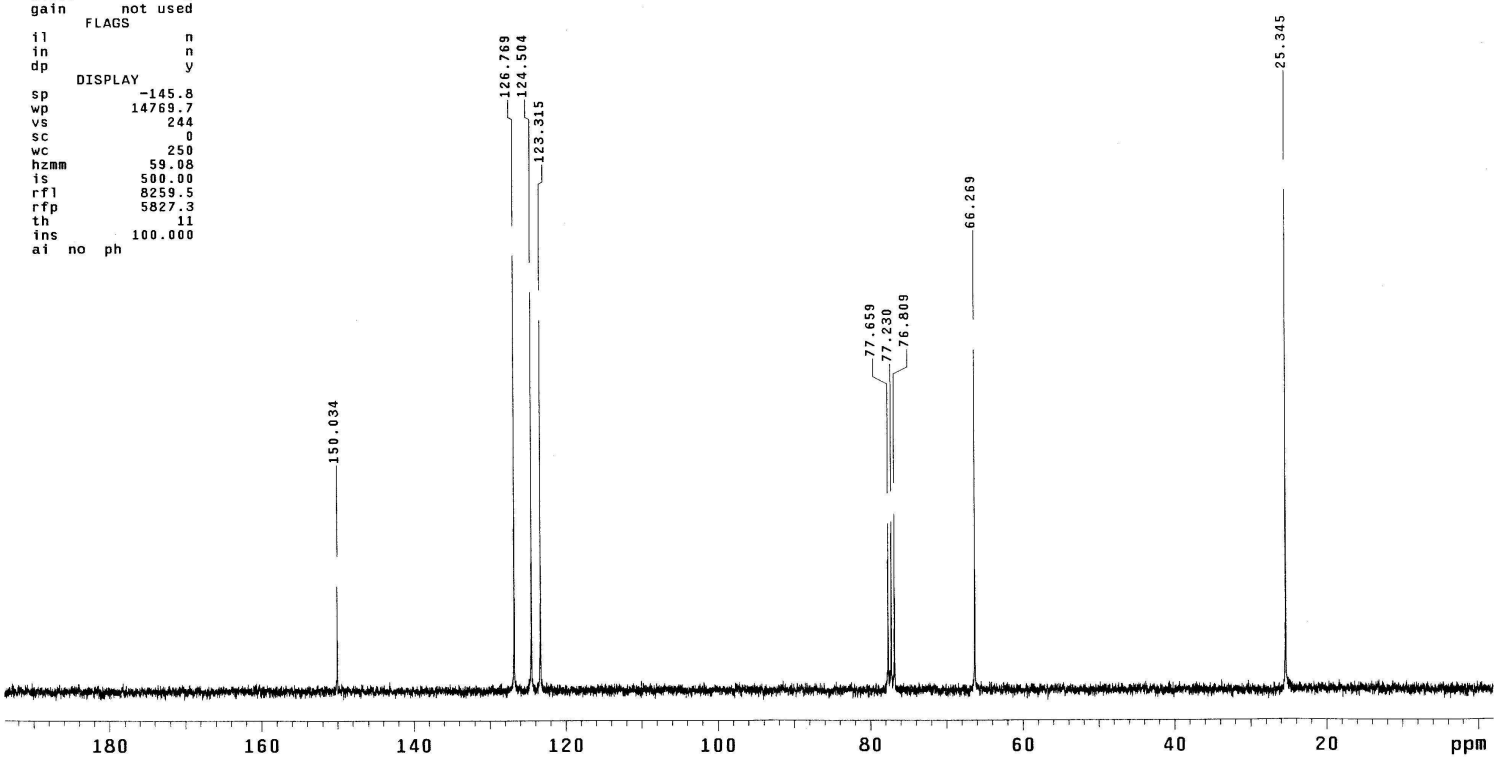


led-VII-133-29-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Sep 22 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	1.000	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	1000	wnt	
ct	136		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-145.8		
wp	14769.7		
vs	244		
sc	0		
wc	250		
hzmm	59.08		
is	500.00		
rfl	8259.5		
rfp	5827.3		
th	11		
ins	100.000		
ai no ph			

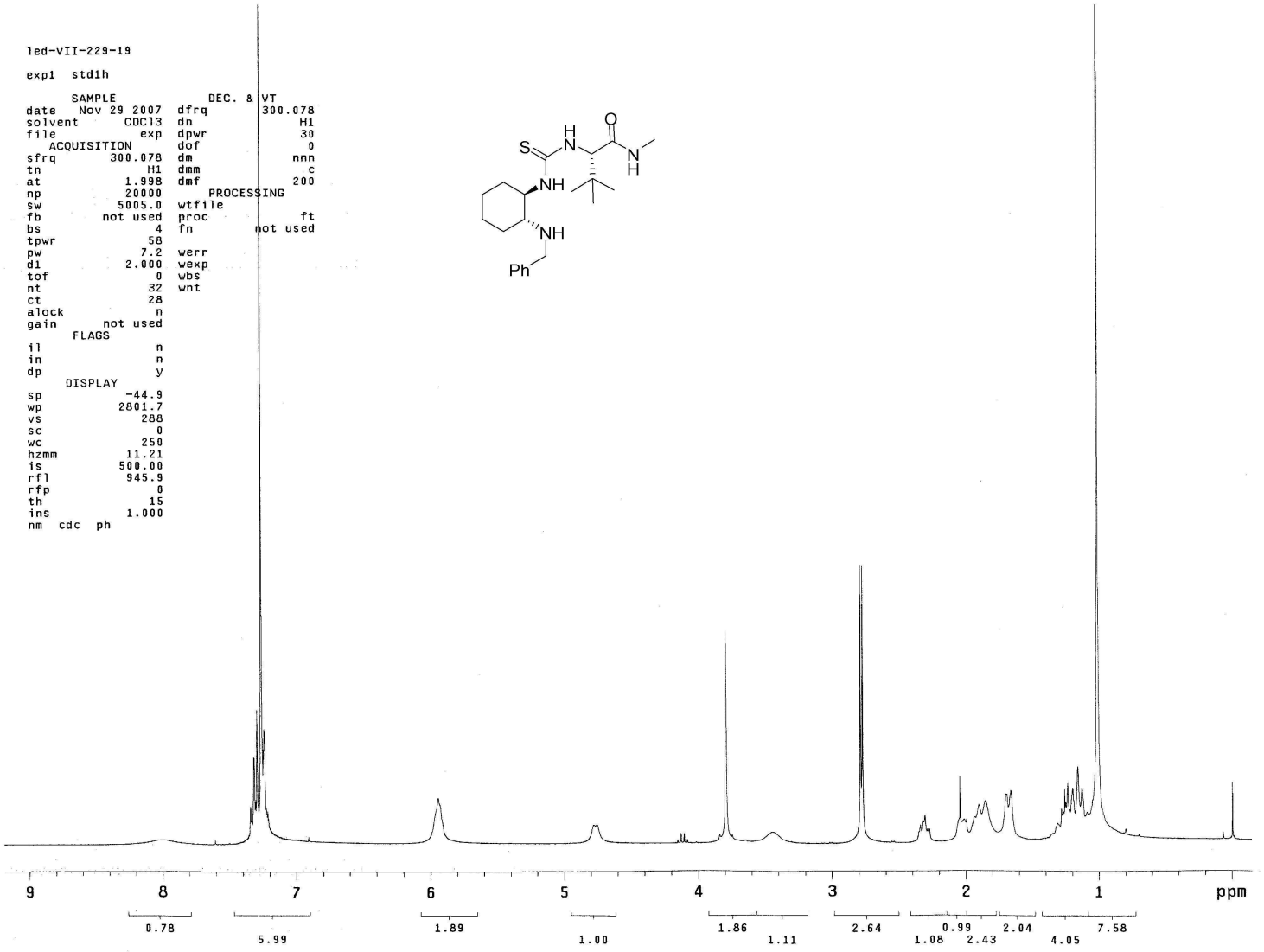


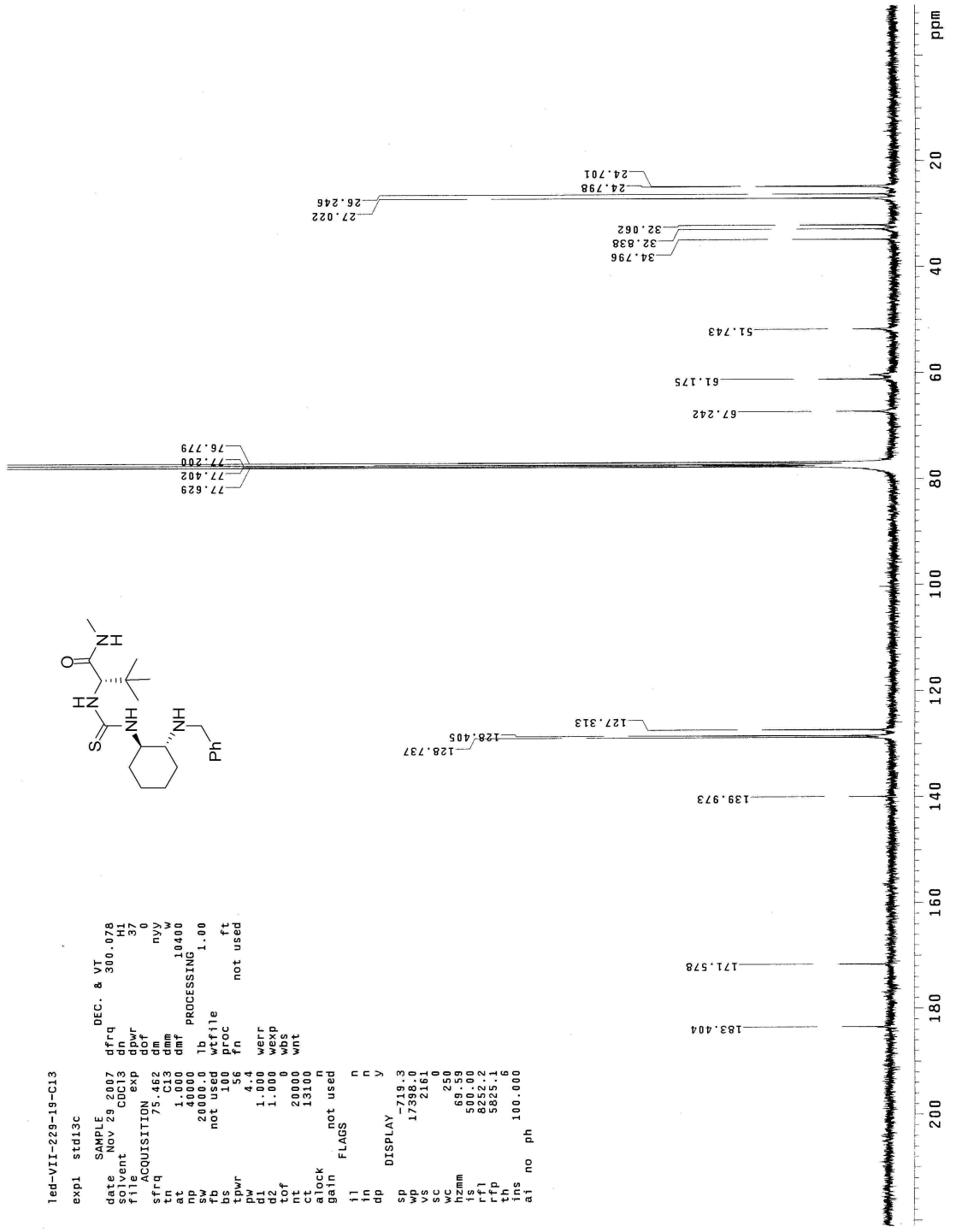


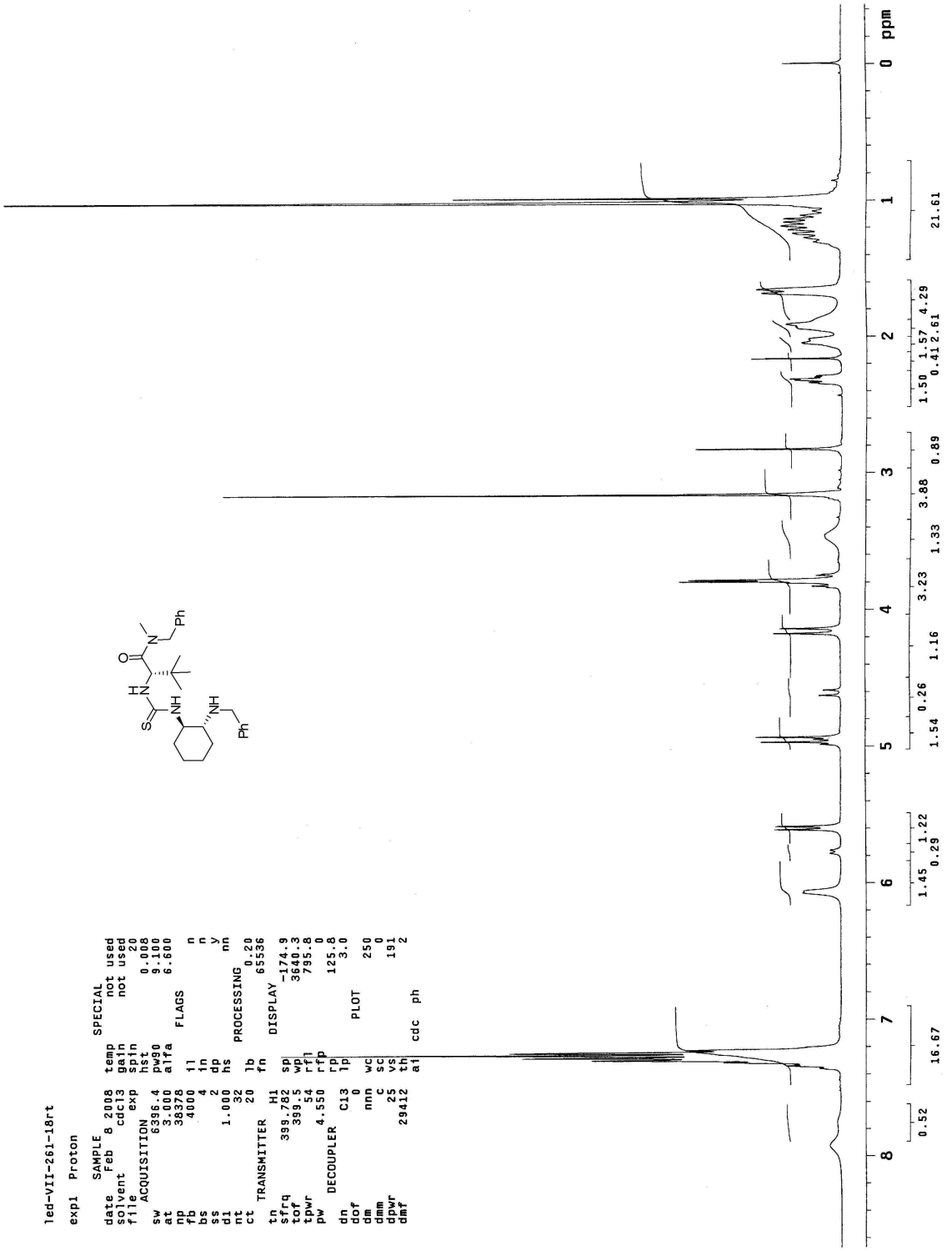
led-VII-229-19

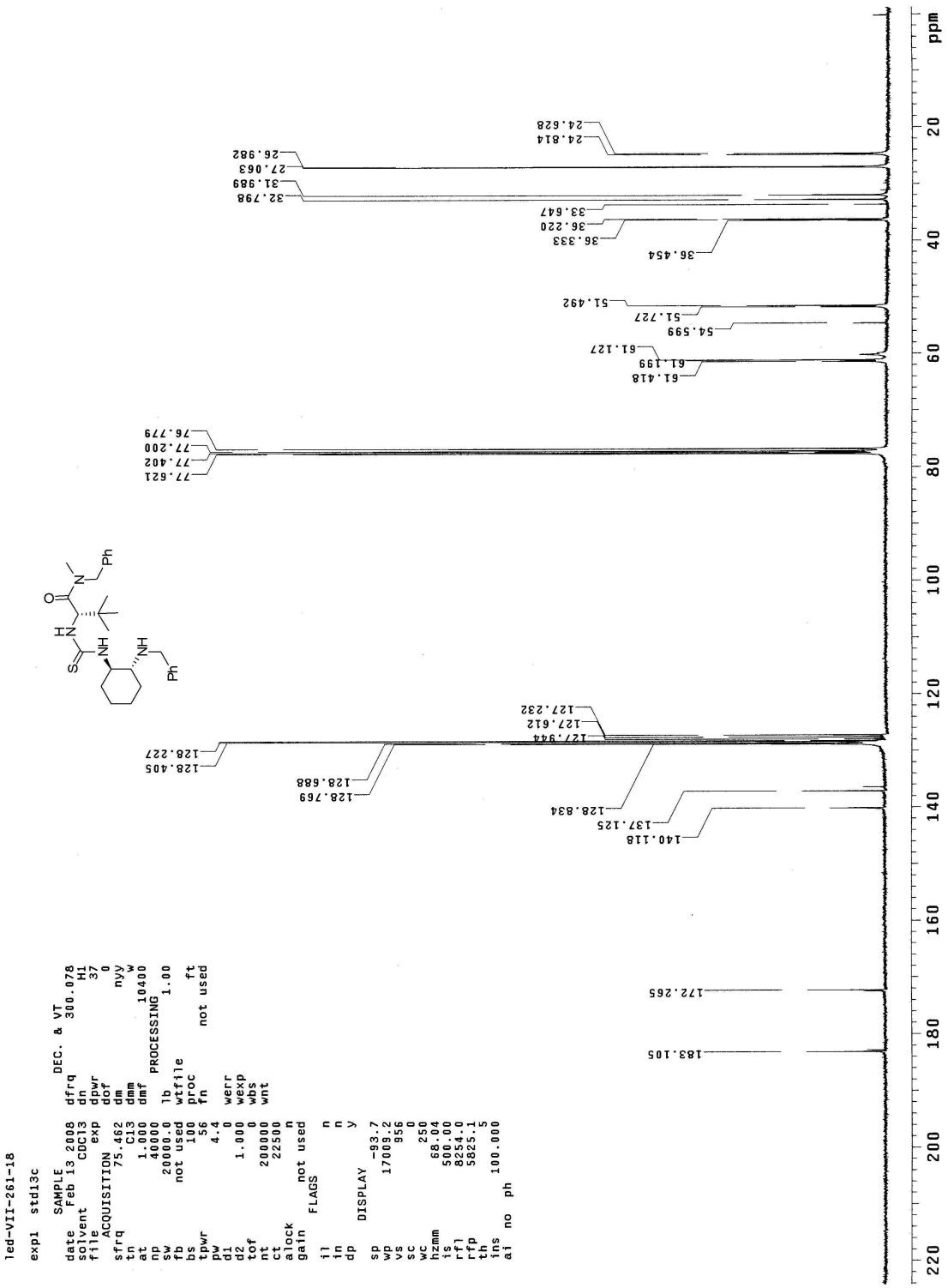
exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 29 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-44.9		
wp	2801.7		
vs	288		
sc	0		
wc	250		
hzmm	11.21		
is	500.00		
rfl	945.9		
rfp	0		
th	15		
ins	1.000		
nm cdc ph			




led-VII-229-19-C13
expl std13c
SAMPLE
date Nov 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 100
tpwr 56
pw 4.4
d1 1.000
d2 1.000
tof 0
nt 20000
ct 13100
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -719.3
wp 17398.0
vs 2161
sc 0
wc 250
hzmm 69.59
is 500.00
rfl 8252.2
rfp 5825.1
th 6
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile ft
proc
fn not used
werr
wexp
wbs
wnt
77.629
77.402
77.200
76.779
183.404
171.578
139.973
128.737
128.405
127.313
67.242
61.175
51.743
34.796
32.838
32.062
27.022
26.246
24.798
24.701
ppm
20
40
60
80
100
120
140
160
180
200

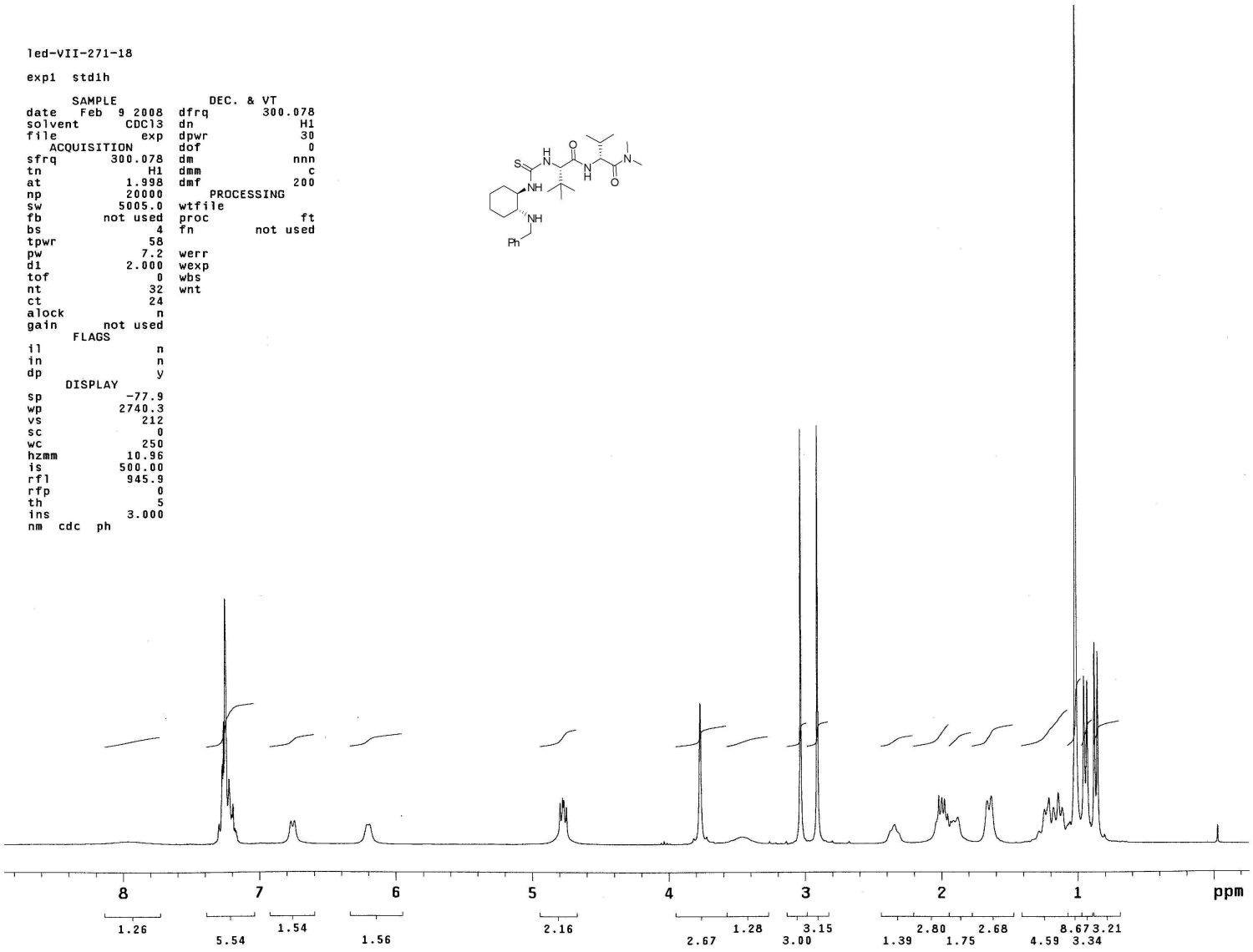

led-VII-261-18rt
exp1 Proton
SAMPLE
date Feb 8 2008
solvent cdcl3
file exp
ACQUISITION
sw 6396.4
at 3.000
np 38378
fb 4000
bs 4
ss 2
d1 1.000
nt 32
ct 20
TRANSMITTER H1
tn
sfrq 399.782
tof 399.5
tpwr 54
pw 4.550
DECOUPLER C13
dn
dof 0
dm nnn
dmm c
dpwr 25
dmf 29412
SPECIAL not used not used
temp
gain
spin 20
hst 0.008
pw90 9.100
alfa 6.600
FLAGS n n y nn
il
in
dp
hs
PROCESSING 0.20
lb
fn 65536
DISPLAY -174.9 3640.3 795.8 0 125.8 3.0
sp
wp
rfl
rfp
rp
lp
PLOT 250 0 191 2
wc
sc
vs
th
cdc ph
af
Ph
Ph
0.52
16.67
1.45 1.22
0.29
1.54 0.26 1.16 3.23 1.33 3.88 0.89
1.50 1.57 4.29
0.41 2.61
21.61
8 7 6 5 4 3 2 1 0 ppm

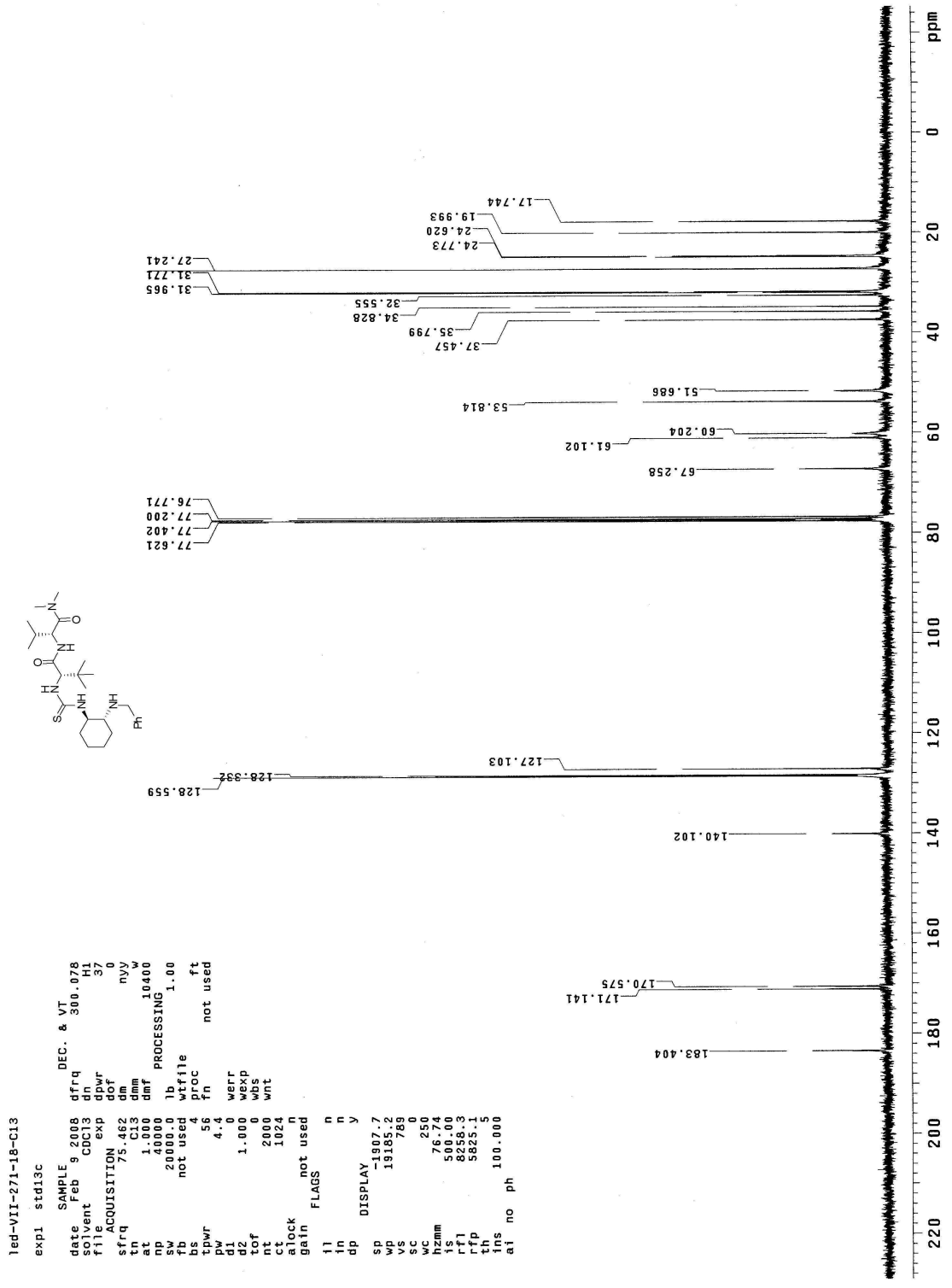

led-VII-261-18
exp1 std13c
SAMPLE
date Feb 13 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 100
tpwr 56
pw 4.4
d1 1.000
d2 0
tof 200000
nt 22500
ct
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -83.7
wp 17009.2
vs 956
sc 0
wc 250
hzmm 68.04
is 500.00
rfl 8254.0
rfp 5825.1
th 5
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
ppm
24.628
24.814
26.982
27.063
31.989
32.798
33.647
36.220
36.333
36.454
51.492
51.727
54.599
61.127
61.199
61.418
76.779
77.200
77.402
77.621
127.232
127.612
127.944
128.227
128.405
128.688
128.769
128.834
137.125
140.118
172.265
183.105

led-VII-271-18

exp1 std1h

SAMPLE		DEC. & VT	
date	Feb 9 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-77.9		
wp	2740.3		
vs	212		
sc	0		
wc	250		
hzmm	10.96		
is	500.00		
rfl	945.9		
rfp	0		
th	5		
ins	3.000		
nm cdc ph			

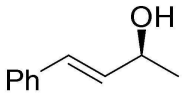


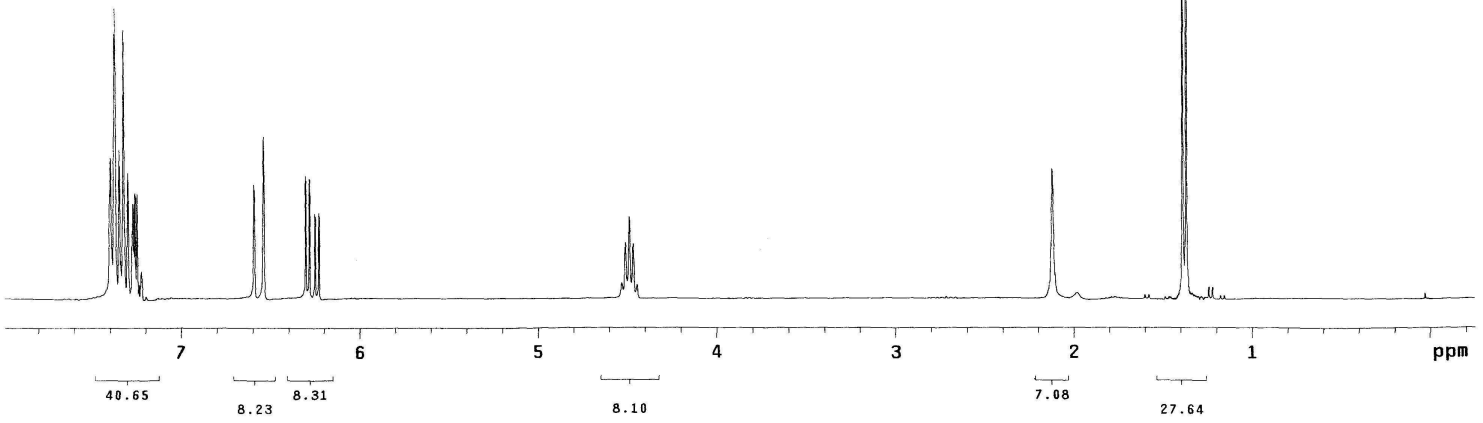

led-VII-271-18-C13
exp1 std13c
SAMPLE
date Feb 9 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 1.000
d2 0
tof 2000
nt 1024
ct
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1907.7
wp 19185.2
vs 789
sc 0
wc 250
hzmm 76.74
is 500.00
rf1 8258.3
rfp 5825.1
th 5
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
ffn not used
werr
wexp
wbs
wnt
183.404
171.141
170.575
140.102
128.559
128.332
127.103
77.621
77.402
77.200
76.771
67.258
61.102
60.204
53.814
51.686
37.457
35.799
34.828
32.555
31.965
31.771
27.241
24.773
24.620
19.993
17.744
ppm

led-VII-109-r

expl std1h

SAMPLE		DEC. & VT	
date	Sep 14 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-75.5		
wp	2472.7		
vs	120		
sc	0		
wc	250		
hzmm	9.89		
is	500.00		
rfl	945.9		
rfp	0		
th	3		
ins	100.000		
nm cdc ph			

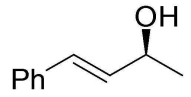


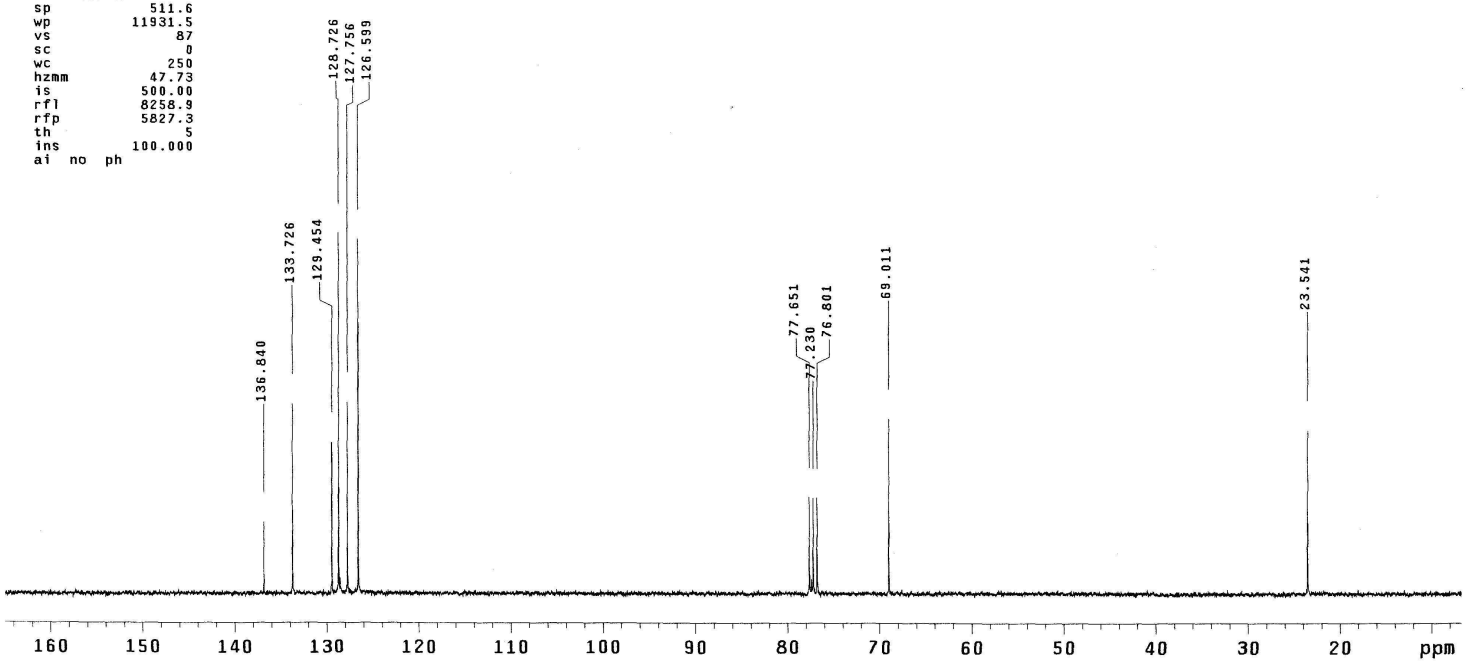


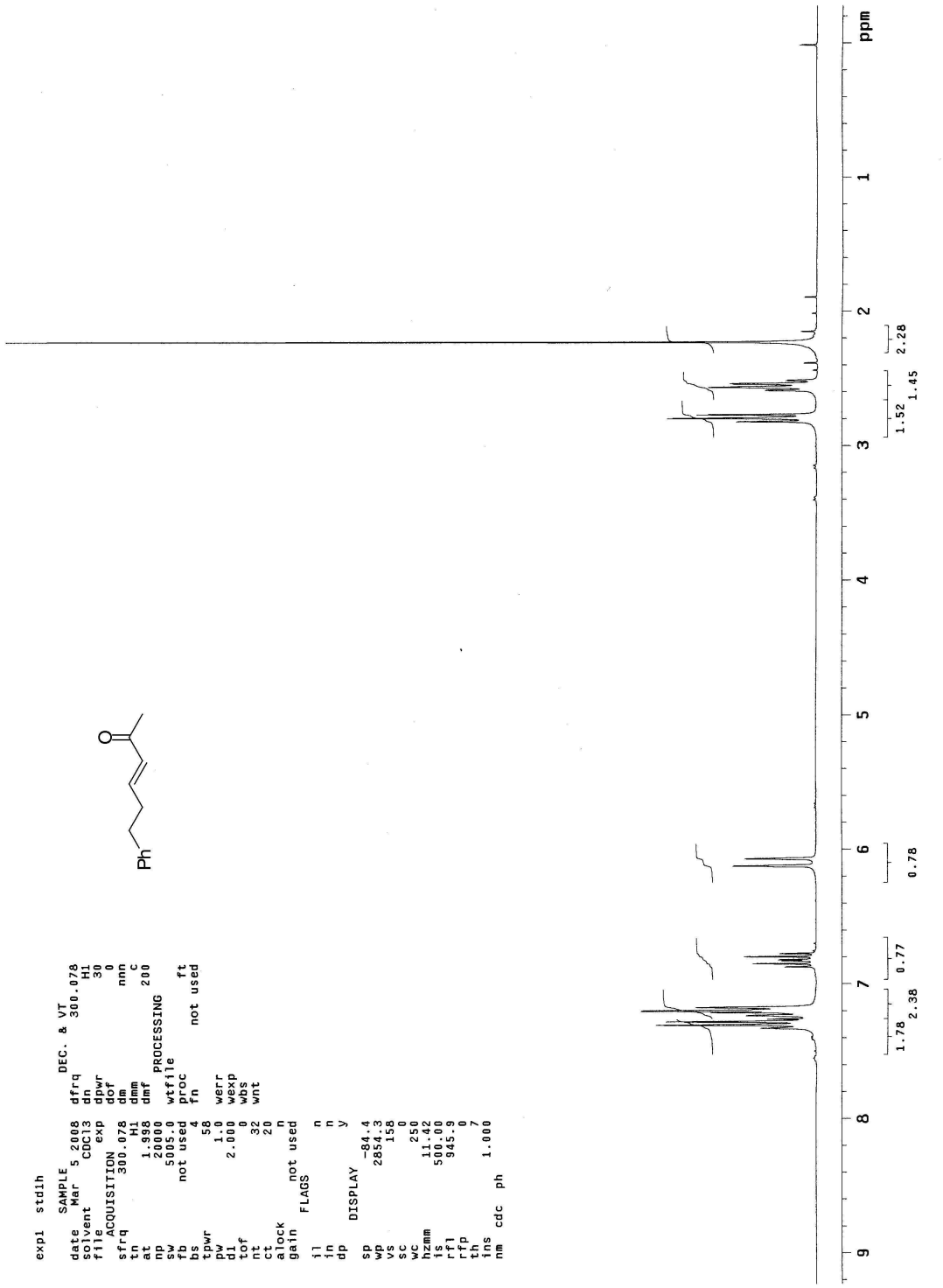
led-VII-109-r-C13

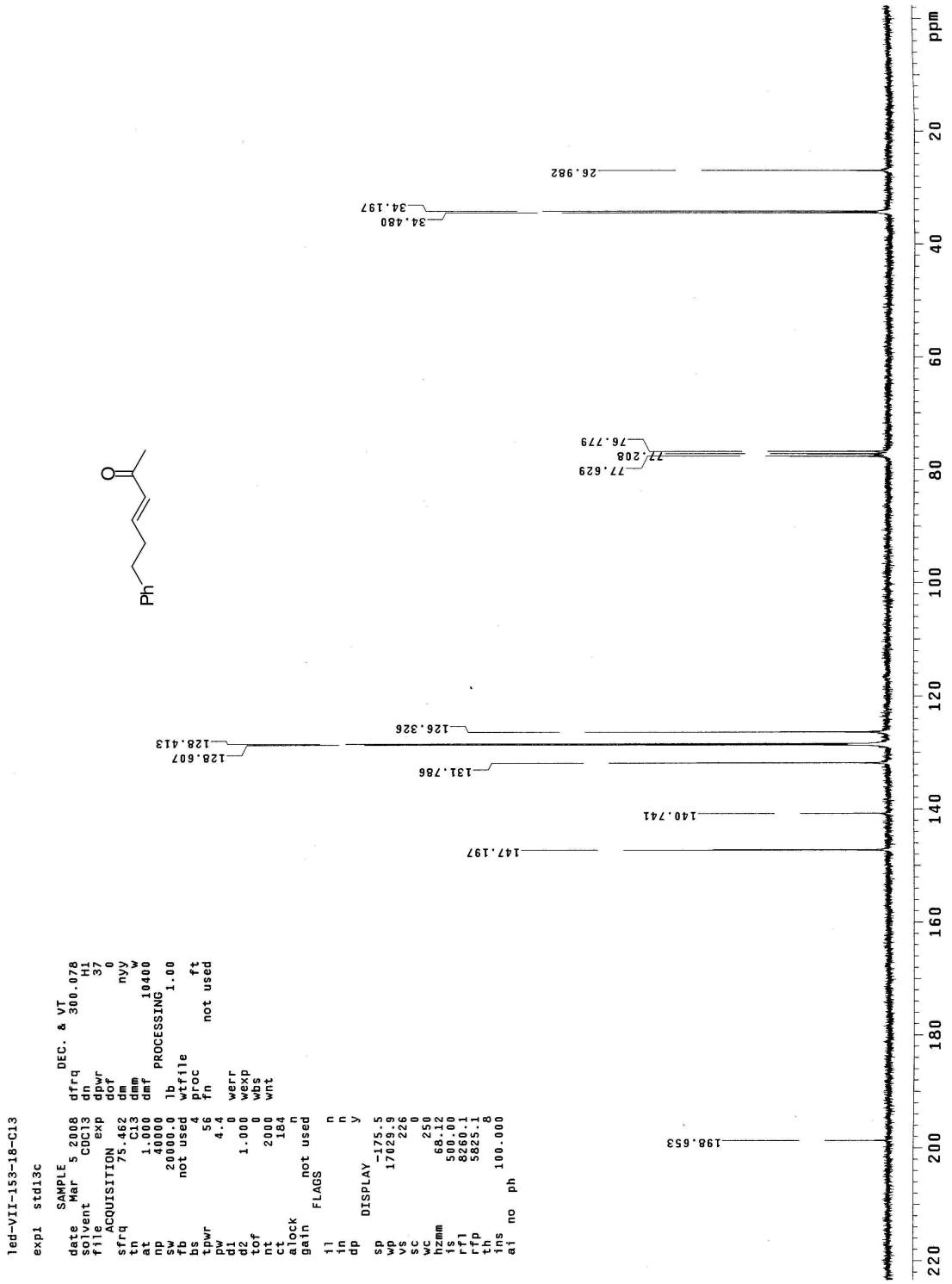
expl std13c

SAMPLE		DEC. & VT	
date	Sep 14 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	1.000	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	1000	wnt	
ct	112		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	511.6		
wp	11931.5		
vs	87		
sc	0		
wc	250		
hzmm	47.73		
is	500.00		
rfl	8258.9		
rfp	5827.3		
th	5		
ins	100.000		
ai no ph			





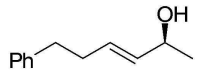

expl stdih
SAMPLE
date Mar 5 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 1.0
pl 2.000
tof 0
nt 32
ct 20
alock n
gain n
FLAGS
il n
in n
dp y
DISPLAY
sp -84.4
wp 2854.3
vs 158
sc 0
wc 250
hzmm 11.42
is 500.00
rfl 945.9
rfp 0
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Ph
O
9
8
7
6
5
4
3
2
1
ppm
1.78
2.38
0.77
0.78
1.52
1.45
2.28

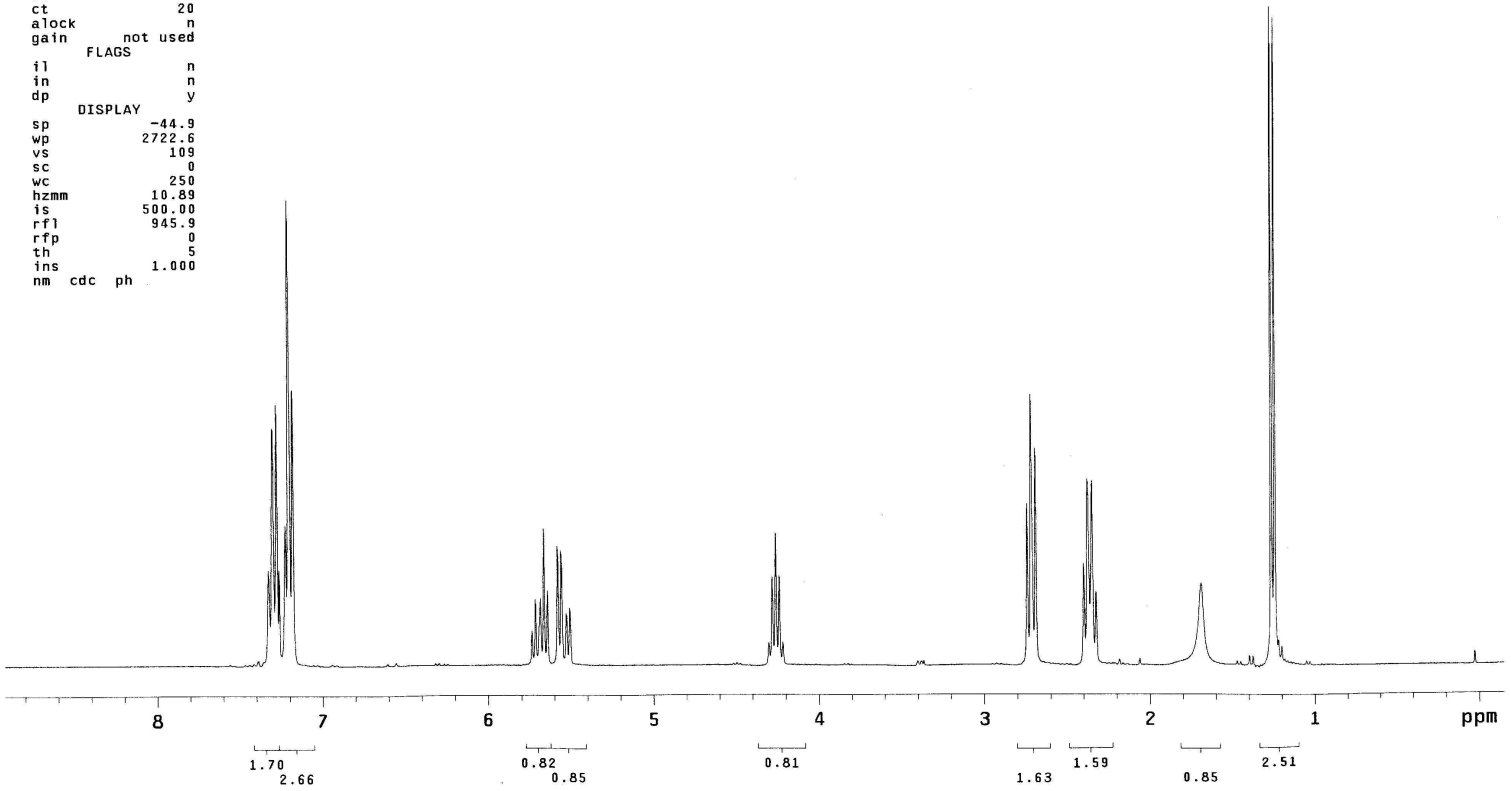

led-VII-153-18-C13
expl std13c
SAMPLE
date Mar 5 2008
solvent CDCl3
file
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 0
d2 1.000
tof 0
nt 2000
ct 184
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -175.5
wp 17029.9
vs 226
sc 250
wc 68.12
hzmm 500.00
is 8260.1
rfl 5825.1
rfp 8
th 100.000
ins
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Ph
198.653
147.197
140.741
131.786
128.607
128.413
126.326
77.629
77.208
76.779
34.480
34.197
26.982
220
200
180
160
140
120
100
80
60
40
20
ppm

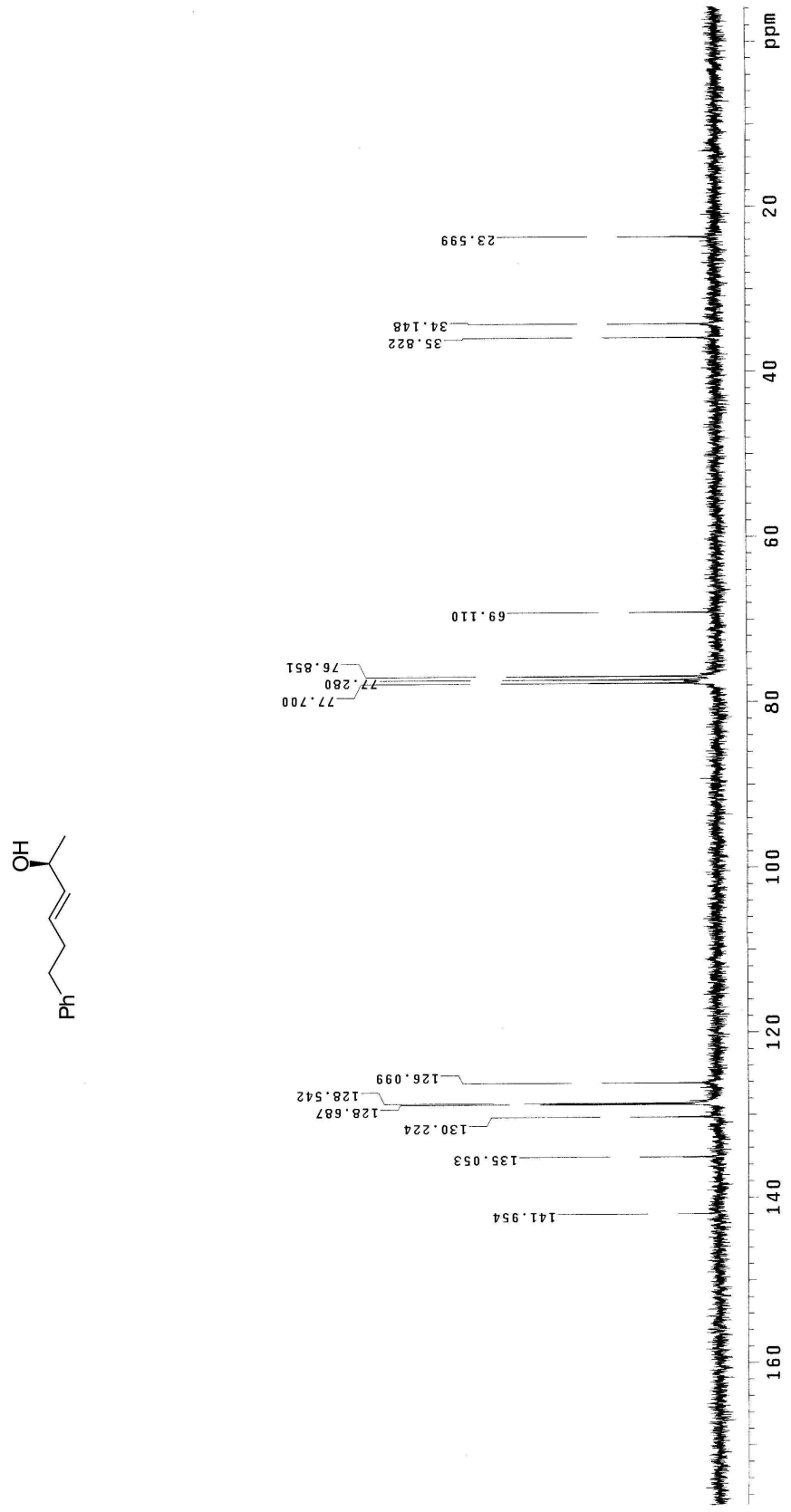
led-VII-154

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 8 2007	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	dmf	200
np	20000	PROCESSING	
sw	5005.0	wtfile	
fb	not used	proc	ft
bs	4	fn	not used
tpwr	58		
pw	1.0	werr	
d1	2.000	wexp	
tof	0	wbs	
nt	32	wnt	
ct	20		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-44.9		
wp	2722.6		
vs	109		
sc	0		
wc	250		
hzmm	10.89		
is	500.00		
rfl	945.9		
rfp	0		
th	5		
ins	1.000		
nm cdc ph			



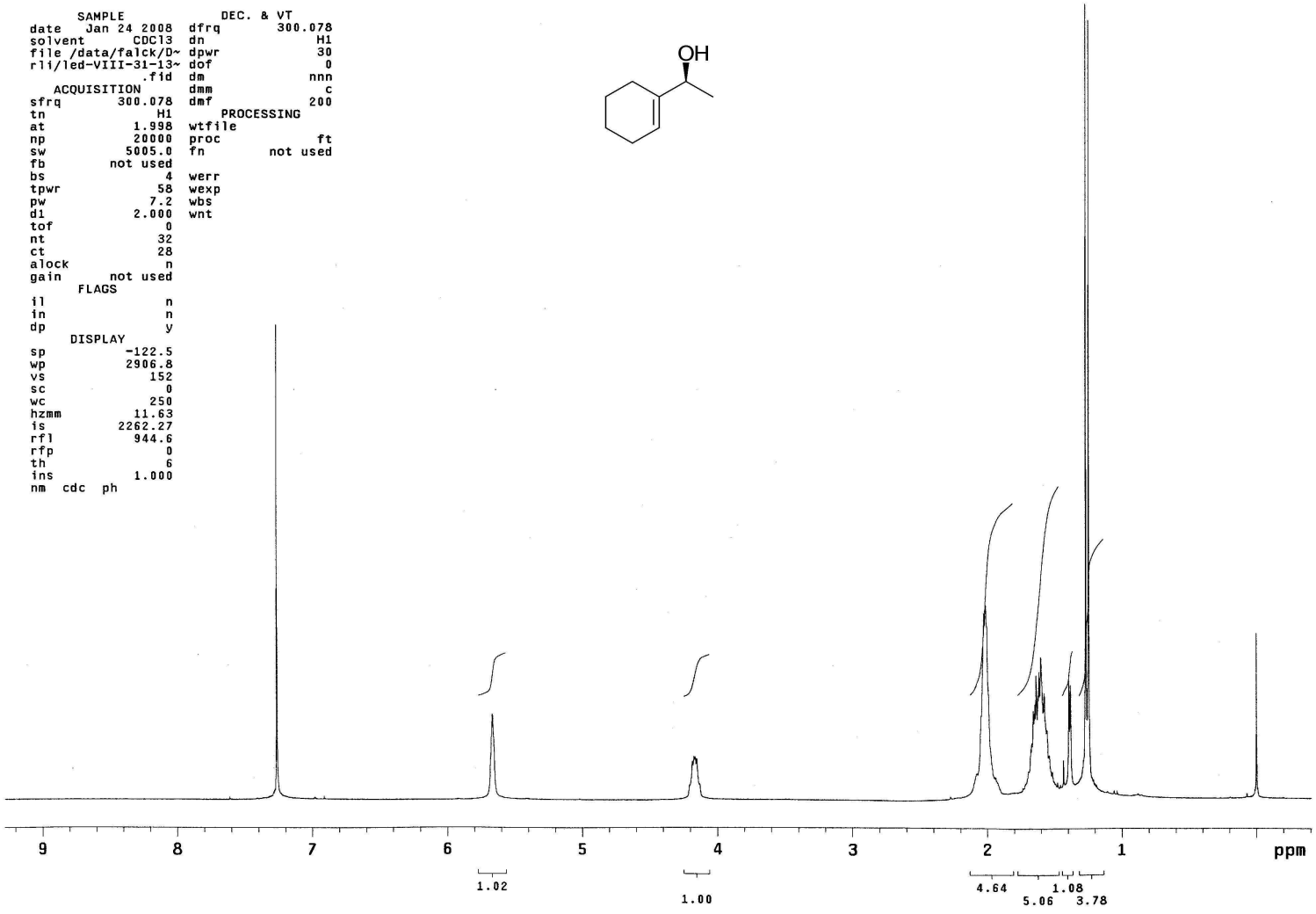


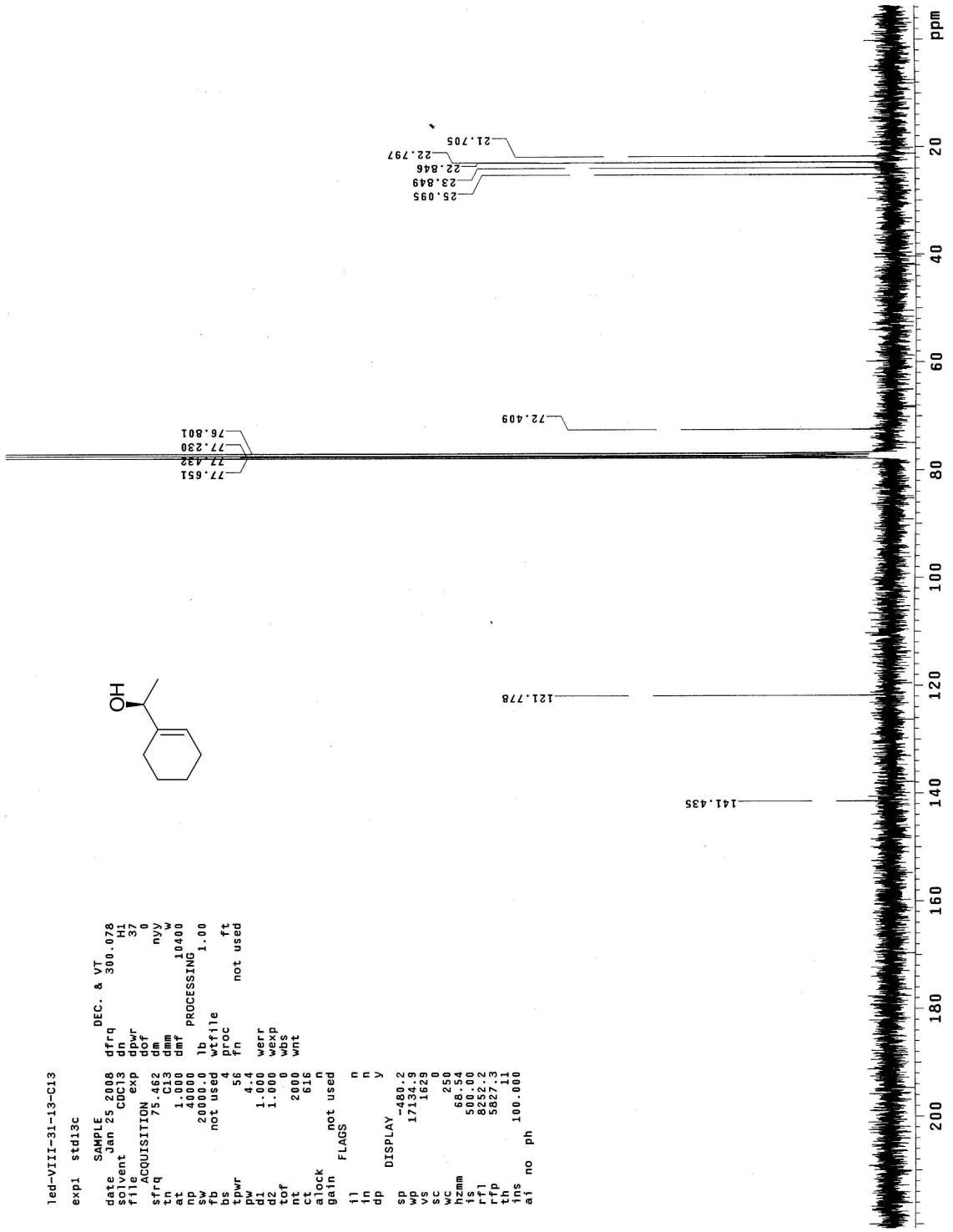

OH
Ph
141.954
135.053
130.224
128.687
128.542
126.099
77.700
77.280
76.851
69.110
35.822
34.148
23.599
ppm
20
40
60
80
100
120
140
160

led-VIII-31-13

exp1 std1h

SAMPLE		DEC. & VT	
date	Jan 24 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	/data/falck/D~	dpwr	30
rli/led-VIII-31-13~	.fid	dof	0
ACQUISITION		dm	nnn
sfrq	300.078	dmm	c
tn	H1	dmf	200
at	1.998	PROCESSING	
np	20000	wtfile	
sw	5005.0	proc	ft
fb	not used	fn	not used
bs	4	werr	
tpwr	58	wexp	
pw	7.2	wbs	
d1	2.000	wnt	
tof	0		
nt	32		
ct	28		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-122.5		
wp	2906.8		
vs	152		
sc	0		
wc	250		
hzmm	11.63		
is	2262.27		
rfl	944.6		
rfp	0		
th	6		
ins	1.000		
nm cdc ph			





led-VIII-31-13-C13

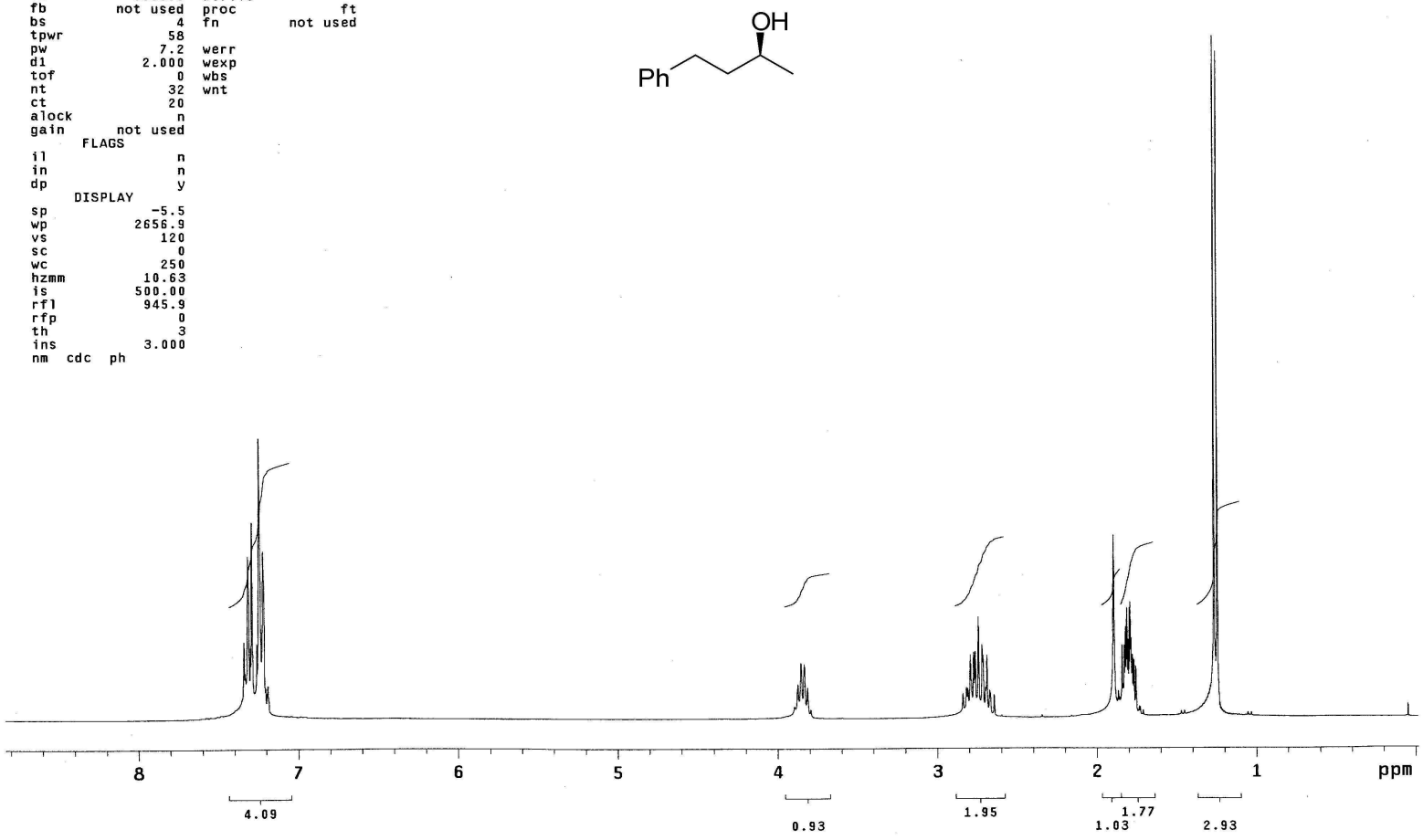
expl std13c

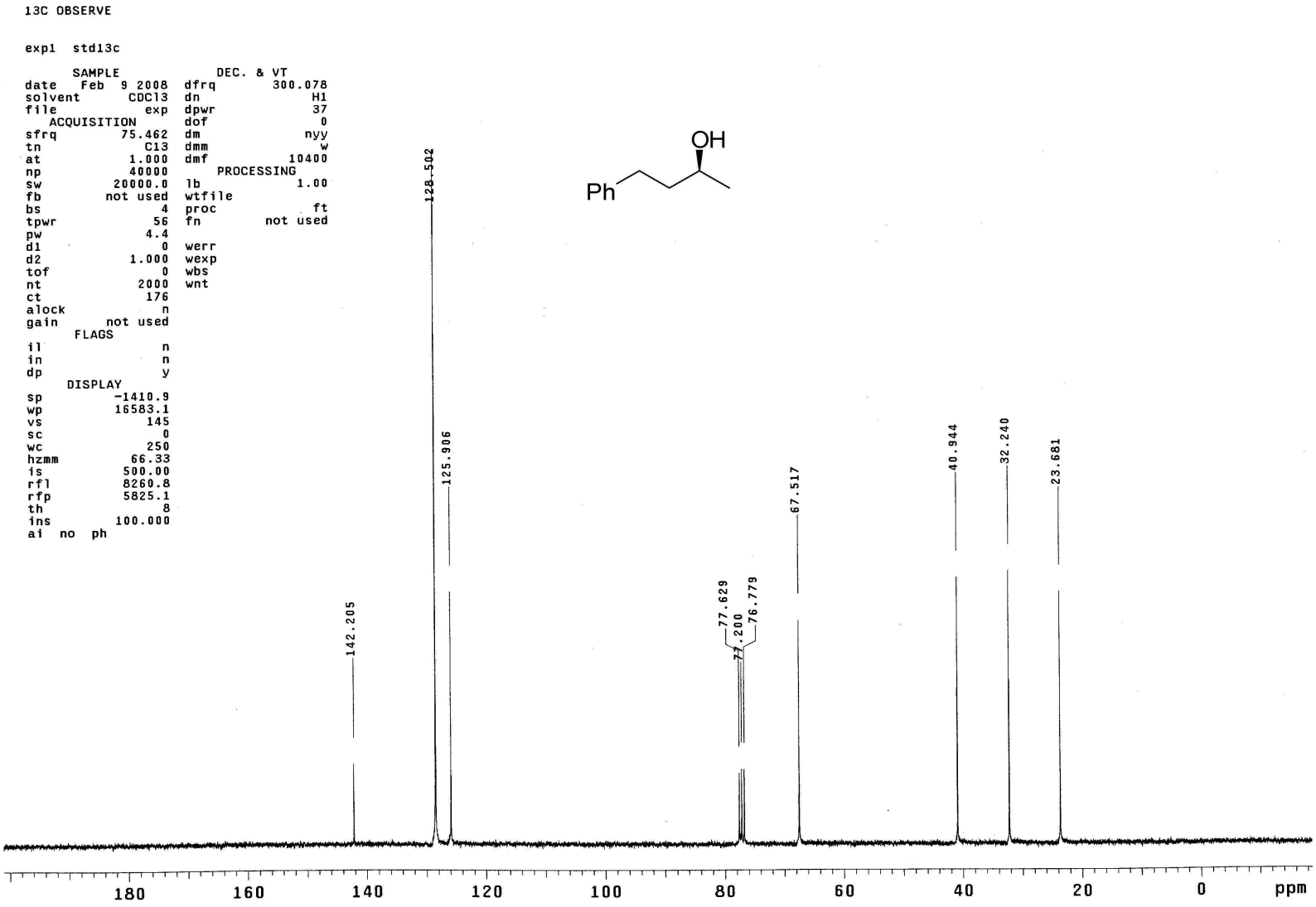
	SAMPLE	DEC. & VT	
date	Jan 25 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
	ACQUISITION	dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	1.000	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	2000	wnt	
ct	616		
alock	n		
gain	not used		
	FLAGS		
il	n		
in	n		
dp	y		
	DISPLAY		
sp	-480.2		
wp	17134.9		
vs	1629		
sc	0		
wc	250		
hzmm	68.54		
is	500.00		
rfl	8252.2		
rfp	5827.3		
th	11		
ins	100.000		
ai	no ph		

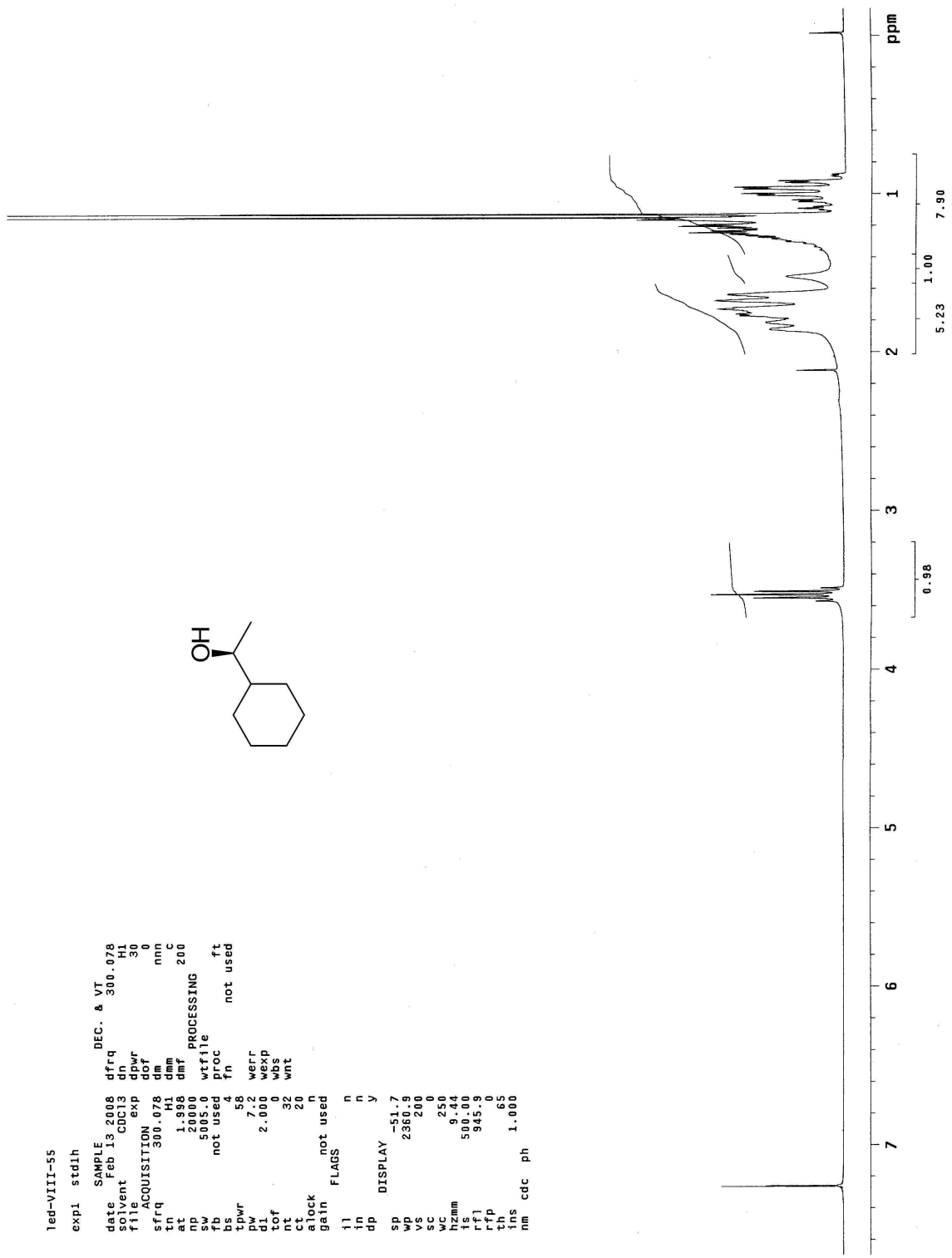
led-VII-97-18

exp1 std1h

SAMPLE		DEC. & VT	
date	Feb 9 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	300.078	dm	nnn
tn	H1	dmm	c
at	1.998	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	20		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-5.5		
wp	2656.9		
vs	120		
sc	0		
wc	250		
hzmm	10.63		
is	500.00		
rfl	945.9		
rfp	0		
th	3		
ins	3.000		
nm cdc ph			




13C OBSERVE
exp1 std13c
SAMPLE
date Feb 9 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 75.462
tn C13
at 1.000
np 40000
sw 20000.0
fb not used
bs 4
tpwr 56
pw 4.4
d1 0
d2 1.000
tof 0
nt 2000
ct 176
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1410.9
wp 16583.1
vs 145
sc 0
wc 250
hzmm 66.33
is 500.00
rfl 8260.8
rfp 5825.1
th 8
ins 100.000
ai no ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 37
dof 0
dm nyy
dmm w
dmf 10400
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
Ph
142.205
128.502
125.906
77.629
77.200
76.779
67.517
40.944
32.240
23.681
180
160
140
120
100
80
60
40
20
0
ppm


led-VIII-55
expl stdih
SAMPLE
date Feb 13 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 300.078
tn H1
at 1.998
np 20000
sw 5005.0
fb not used
bs 4
tpwr 58
pw 7.2
dl 2.000
tof 0
nt 32
ct 20
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -51.7
wp 2360.9
vs 200
sc 0
wc 250
hzmm 9.44
fs 500.00
rfl 945.9
rfp 0
th 65
ins 1.000
nm cdc ph
DEC. & VT
dfrq 300.078
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
OH
0.98
1.00
5.23
7.90
ppm

led-VIII-55-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 13 2008	dfrq	300.078
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	75.462	dm	nyy
tn	C13	dmm	w
at	1.000	dmf	10400
np	40000	PROCESSING	
sw	20000.0	lb	1.00
fb	not used	wtfile	
bs	4	proc	ft
tpwr	56	fn	not used
pw	4.4		
d1	0	werr	
d2	1.000	wexp	
tof	0	wbs	
nt	1000	wnt	
ct	40		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-141.9		
wp	13087.6		
vs	234		
sc	0		
wc	250		
hzmm	52.35		
is	500.00		
rfl	8253.4		
rfp	5825.1		
th	14		
ins	100.000		
ai no ph