

Supporting Information

Cascade Synthesis of (*E*)-2-Alkylidenecyclobutanols

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General Procedures. All reactions were maintained under an argon atmosphere and conducted in anhydrous solvents freshly distilled from sodium benzophenone ketyl or CaH₂ as appropriate. Extracts were dried over anhydrous Na₂SO₄ and then filtered prior to removal of volatiles under reduced pressure. Unless otherwise noted, commercially available materials were used without further purification. Flash chromatography (FC) was performed using E Merck silica gel 60 (240–400 mesh). Thin layer chromatography was performed using pre-coated plates purchased from E. Merck (silica gel 60 PF254, 0.25 mm). Nuclear magnetic resonance (NMR) spectra were recorded on Varian 300 or 400 spectrometers at operating frequencies of 300/400 MHz (¹H) or 75/100 MHz (¹³C). Chemical shifts (δ) are given in ppm relative to residual solvent (usually chloroform $\delta = 7.27$ for ¹H NMR or $\delta = 77.23$ for proton decoupled ¹³C NMR), and coupling constants (J) in Hz. Melting points are uncorrected. The Michigan State University Mass Spectroscopy Facility or Prof. Kasem Nithipatikom (Medical College of Wisconsin) kindly provided high-resolution mass spectral analyses.

Chemicals. **8** and **24** were prepared as described before (*vide infra*). Ketones **6**, **10**, **12**, and **26** (R = Ph, Me) were obtained from commercial sources.

(E)-(4,4,4-Trichlorobut-1-enyl)benzene* (8). To a stirring, 0 °C solution of cinnamyl bromide (5 g, 25.64 mmol) and anhydrous chloroform (6.0 g, 51.28 mmol) in anhydrous DMF (50 mL) was added solid sodium hydride (65% oil suspension, 1.78 g, 76.92 mmol). After 2 h, the reaction mixture was warmed to room temperature, stirred for another 2 h, then quenched with sat. aq. ammonium chloride (36 mL), and extracted with Et₂O (4 × 50 mL). The combined ethereal extracts were washed with brine (2 × 75 mL), water (2 × 75 mL), dried, and concentrated under reduced pressure. The residue was purified by SiO₂ column chromatography using hexane as eluent to give **8** (5.34 g, 75%) as a colorless oil. TLC: SiO₂, hexanes (100%), R_f ≈ 0.6; ¹H NMR (300 MHz, CDCl₃) δ 7.45-7.21 (m, 5H), 6.67 (d, $J = 15.9$ Hz, 1H), 6.35-6.30 (dt, $J = 7.0, 15.9$ Hz, 1H), 3.54 (d, $J = 7.0$ Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 137.31, 129.09, 128.60, 127.24, 127.04, 121.73, 99.27, 58.46.

*Prep. Ref.: Baati, R.; Barma, D. K.; Krishna, U. M.; Mioskowski, C.; Falck, J. R. *Tetrahedron Lett.* **2002**, *43*, 959–961.

(4,4,4-Trichlorobutyl)benzene* (5). A suspension of **8** (2.1 g, 8.9 mmol) and 5% Pd/C (100 mg) in EtOAc (25 mL) was stirred at room temperature under a hydrogen atmosphere (1 atm). After 12 h, the reaction mixture was filtered through a short pad of silica gel using EtOAc as eluent. The filtrate was dried and concentrated under reduced pressure to give **5** (2.08 g, 98%) as a colorless oil sufficiently pure to be used without purification. TLC: SiO₂, hexane (100%), R_f ≈ 0.65; ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.20 (m, 5H), 2.74-2.67 (m, 4H), 2.18-2.08 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 141.04, 128.81, 128.60, 126.50, 100.15, 54.75, 34.59, 28.15.

*Prep. Ref.: Baati, R.; Barma, D. K.; Krishna, U. M.; Mioskowski, C.; Falck, J. R. *Tetrahedron Lett.* **2002**, *43*, 959–961.

2-(2,2,2-Trichloroethyl)naphthalene (16). Following the procedure utilized for the preparation of **8**, 2-(bromomethyl)naphthalene (3.2 g, 14.62 mmol) was converted into **16** (2.47 g, 66%), isolated as a colorless oil. TLC: SiO₂, hexane (100%), R_f ≈ 0.7; ¹H NMR (300 MHz, CDCl₃) δ 7.83-7.92 (m, 4H), 7.50-7.59 (m, 3H), 4.07 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 134.72, 133.78, 132.49, 128.20, 128.14, 127.85, 126.99, 126.40, 126.05, 125.95, 99.67, 60.42; HRMS calcd for C₁₂H₉Cl₃ [M]⁺ 257.9770, found 257.9770.

1-Bromo-4-(2,2,2-trichloroethyl)benzene (18). Following the procedure utilized for the preparation of **8**, 4-bromobenzyl bromide (Aldrich Chem. Co.) was converted into **18** (74%), isolated as a colorless oil. TLC: SiO₂, hexane (100%), R_f ≈ 0.65; ¹H NMR (300 MHz, CDCl₃) δ 7.26-7.53 (m, 4H), 6.79 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 131.96, 128.67, 128.46, 120.41, 99.45, 60.05; HRMS calcd for C₈H₆BrCl₃ [M+1]⁺ 286.8797, found 286.8801.

2,2,2-Trichloro-1-(2,4-difluorophenyl)ethanol (20). Adapting literature* procedure, trichloroacetic acid (2.50 g, 15 mmol) and sodium trichloroacetate (2.75 g, 15 mmol) were simultaneously added to a stirring, room temperature solution of 2,4-difluorobenzaldehyde (1.42 g, 10 mmol) in anhydrous DMF (10 mL) at a rate so as not to exceed 25 °C. An initial rapid evolution of CO₂ was observed. After 30 min, the mixture was diluted with ether-hexane (40 mL, 1:1), washed with sat. sodium bicarbonate solution (3 × 20 mL) and brine (20 mL). The organic layer was separated, dried, concentrated, and the residue was passed through a short pad of silica gel using 5% EtOAc/hexane as eluent to afford **20** (2.12 g, 82%) as a colorless oil. TLC: SiO₂,

EtOAc/hexane (15:85), $R_f \approx 0.35$; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.81-7.61 (m, 1H), 6.96-6.82 (m, 2H), 5.58 (d, $J = 4.6$ Hz, 1H), 3.32 (br s, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 164.01 (dd, $J = 12.15$ Hz, $J = 205.65$ Hz), 160.68 (dd, $J = 12.75$ Hz, $J = 206.32$ Hz), 131.23 (dd, $J = 4.27$ Hz, $J = 9.75$ Hz), 119.56 (dd, $J = 3.67$ Hz, $J = 12.15$ Hz), 111.58 (dd, $J = 3.6$ Hz, $J = 21.22$ Hz), 103.70 (dd, $J = 26.02$ Hz, $J = 52.12$ Hz), 102.66, 76.97; HRMS calcd for $\text{C}_8\text{H}_5\text{Cl}_3\text{F}_2\text{O}$ $[\text{M}+1]^+$ 260.9452, found 260.9449.

*Prep. Ref.: Wang, Z.; Campagna, S.; Yang, K.; Xu, G.; Pierce, M. E.; Fortunak, J. M.; Confalone, P. N. *J. Org. Chem.* **2000**, *65*, 1889-1891.

8-(tert-Butyldiphenylsilyloxy)oct-1-en-3-one (14). Vinyl magnesium bromide (1 M soln in THF, 2.16 g, 16.75 mmol) was added to a stirring, 0 °C solution of 6-(tert-butylidiphenylsilyloxy)hexanal (5.4 g, 15.25 mmol) in dry THF (100 mL). After 30 min, the reaction mixture was quenched with sat. ammonium chloride solution (20 mL) and extracted with ether (4×25 mL). The combined ethereal extracts were washed with brine (2×60 mL), water (2×40 mL), dried, and concentrated under reduced pressure. The residue was purified by SiO_2 column chromatography using EtOAc/hexane (1:9) to afford 8-(tert-butylidiphenylsilyloxy)oct-1-en-3-ol (5.47 g, 94%) as a colorless oil. TLC: SiO_2 , EtOAc/hexane (15:85), $R_f \approx 0.4$; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.69-7.64 (m, 4 H), 7.45-7.34 (m, 6H), 5.92-5.80 (m, 1H), 5.25-5.06 (m, 2H), 4.13-4.03 (m, 1H), 3.66 (t, $J = 6.9$ Hz, 2H) 1.62-1.26 (m, 8H), 1.05 (s, 9H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 141.53, 135.18, 129.85, 127.97, 127.94, 114.92, 73.52, 64.18, 37.20, 32.82, 27.22, 25.40, 19.55; HRMS calcd for $\text{C}_{24}\text{H}_{34}\text{O}_2\text{Si}$ $[\text{M}+1]^+$ 383.2406, found 383.2409.

To a stirring, -78 °C solution of oxalyl chloride (3.40 g, 27.22 mmol) in dry CH_2Cl_2 (50 mL) was added anhydrous DMSO (3.08 g, 39.48 mmol). After 30 min, a solution of 8-(tert-butylidiphenylsilyloxy)oct-1-en-3-ol (5.2 g, 13.16 mmol) in dry CH_2Cl_2 (20 mL) was added. After another 1 h, Et_3N (6.65 g, 65.80 mmol) was added and the reaction mixture was slowly warmed to room temperature over 1 h. After 30 min at room temperature, water (20 mL) was added and the reaction mixture was extracted with CH_2Cl_2 (4×50 mL). The combined organic extracts were washed with water (2×75 mL), brine (75 mL), dried, all volatiles were removed under reduced pressure. The residue was purified via SiO_2 column chromatography using

EtOAc/hexane (10:90) as eluent to give **14** (4.60 g, 92%) as a colorless oil. TLC: SiO₂, EtOAc/hexane (1:9), R_f ≈ 0.4; ¹H NMR (300 MHz, CDCl₃) δ 7.78-7.63 (m, 4H), 7.45-7.34 (m, 6H), 6.32 (dd, *J* = 10.2, 17.7 Hz, 1H), 6.20 (dd, *J* = 10.2, 17.7 Hz, 1H), 5.81 (dd, *J* = 1.5, 10.2 Hz, 1H), 3.66 (t, *J* = 6.6 Hz, 2H), 2.56 (t, *J* = 7.8 Hz, 2H), 1.66-1.53 (m, 4H), 1.44-1.35 (m, 2H), 1.04 (s, 9H); ¹³C NMR (75 MHz, CDCl₃) δ 200.41, 136.85, 135.83, 134.28, 129.85, 128.13, 127.91, 63.94, 39.85, 32.63, 27.17, 25.71, 19.49; HRMS calcd for C₂₄H₃₂O₂Si [M+1]⁺ 381.2250, found 381.2250.

4-Methylene-6-phenylhexan-3-one (22). A solution of tri-*n*-butyltin hydride (10 mmol, 2.7 mL) in dry THF (15 mL) was added dropwise followed by Pd(PPh₃)₂Cl₂ (0.2 mmol, 140 mg) to a room temperature solution of 4-phenyl-1-butyne (10 mmol, 1.30 g) in dry THF (50 mL). Upon complete addition, the reaction mixture was stirred 3 h and then the solvent was removed under reduced pressure. The residue was purified by SiO₂ column chromatography using hexanes as a eluent to afford tri-*n*-butyl(4-phenylbut-1-en-2-yl)stannane (3.20 g, 76%) as a light yellow oil. TLC: SiO₂, hexane (100%), R_f ≈ 0.58; ¹H NMR (300 MHz, CDCl₃) δ 7.31-7.27 (m, 3H), 7.25-7.18 (m, 2H), 5.93-5.81 (m, 1H), 5.16-4.95 (m, 1H), 2.71-2.66 (m, 2H), 2.56-2.50 (m, 2H), 1.56-1.46 (m, 12H), 1.38-1.26 (m, 6H), 0.95-0.87 (m, 9H).

Prep. Ref.: Gevorgyan, V.; Liu, J.-X.; Yamamoto, Y. *J. Org. Chem.* **1997**, *62*, 2963-2967.

Following the procedure of Risch*, a mixture of tri-*n*-butyl(4-phenylbut-1-en-2-yl)stannane (420 mg, 1 mmol), propionyl chloride (137 mg, 1.5 mmol), and Pd(Bn)Cl(PPh₃)₂ (1 mol%) in dry HMPA (5 mL) was stirred at 65 °C. After 3 h, Et₂O (45 mL) and aq. KF solution (10 mL, 1.5 M) were added. After 30 min, the organic phase was separated and washed successively with aq. NaHCO₃ solution (30 mL, 5%), brine, dried, and concentration in vacuo. The residue was subjected to silica gel chromatography using EtOAc/hexane (5:95) as eluent to afford ketone **22** (180 mg, 68%) as a light yellow oil. TLC: SiO₂, EtOAc/hexane (1:9), R_f ≈ 0.55; ¹H NMR (300 MHz, CDCl₃) δ 7.28-7.26 (m, 2H), 7.19-7.16 (m, 3H), 5.98 (s, 1H), 5.68 (s, 1H), 2.74-2.67 (m, 4H), 2.62-2.56 (m, 2H), 1.11 (t, *J* = 6.3 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 202.57, 148.00, 141.87, 128.75, 128.56, 126.14, 124.51, 35.13, 33.29, 31.15, 8.70. HRMS calcd for C₁₃H₁₆O [M+1]⁺ 189.1279, found 189.1279.

*Procedure Ref.: Risch, N. *Chem. Ber.* **1985**, *118*, 4073-4085.

2-Methylenecyclohexanone (24). According to literature procedure*, acetyl chloride (38 mg, 2 mol%) was added by syringe to a stirring, room temperature mixture of Zn dust (activated; 2.86 g, 44.10 mmol), copper(I) chloride (427 mg, 4.41 mmol, 10 mol%), and diiodomethane (3.92 g, 14.70 mmol) in ether (50 mL). Next, a solution of 1-(trimethylsilyloxy)cyclohexene (2.50 g, 14.70 mmol; TCI) in ether (20 mL) was added and the reaction mixture was then warmed to 45-50 °C. After 45 min, a solution of diiodomethane (3.92 g, 14.70 mmol) in ether (50 mL) was added to the reaction mixture over a period of 20 min at such a rate as to maintain a manageable reflux. After 2 h, the contents of the reaction flask are transferred to a large Erlenmeyer flask, cooled in an ice bath, and treated with sat. ammonium chloride (50 mL). The resulting precipitate was removed by vacuum filtration and the filter cake was washed with hexane (100 mL). The aq. layer was extracted with CH₂Cl₂ (2 × 60 mL). The combined organic fractions were dried, and concentrated under reduced pressure. The residue was vacuum distilled and the distillate was purified by SiO₂ column chromatography using 2% EtOAc/hexane as eluent to give (bicyclo[4.1.0]heptan-1-yloxy)trimethylsilane (1.89 g, 70%).

A solution of (bicyclo[4.1.0]heptan-1-yloxy)trimethylsilane (1.84 g, 10 mmol) in dry CH₂Cl₂ (2 mL) was slowly added to a 0 °C solution of tin(IV) chloride (10 mmol) in dry CH₂Cl₂ (10 mL). After 30 min, the solvent and TMS-Cl were evaporated under reduced pressure. The crude product was recrystallized from pentane to afford 2-[(trichlorostannyl) methylcyclohexanone (3.29 g, 99%) as a light brown solid, mp 106-107 °C.

TMEDA (470 mg, 4.06 mmol) was added to a stirring, 20 °C solution of 2-[(trichlorostannyl)methylcyclohexanone (1.35 g, 4.06 mmol) in CH₂Cl₂ (5 mL). After 15 min, the resulting colorless precipitate was removed by filtration through diatomaceous earth using pentane. Brine (60 mL) was added to the filtrate and the mixture was then extracted with pentane (3 × 20 mL). The combined organic extracts were dried with MgSO₄, filtered, and evaporated under reduced pressure. The residue was purified by SiO₂ column chromatography using Et₂O/hexane (1:3) as eluent to give ketone **24** (322 mg, 72%) as a light yellow oil. TLC: SiO₂, EtOAc/hexane (1:9), R_f ≈ 0.46; ¹H NMR (300 MHz, CDCl₃) δ 5.83-5.82 (m, 1 H), 5.14-5.12 (m, 1H), 2.59-2.55 (m, 2H), 2.47-2.42 (t, *J* = 6.7 Hz, 2H), 1.91-1.85 (m, 2H), 1.79-1.75 (m, 2H).

*Prep. Ref.: Block, E.; Aslam, M.; Eswarakrishnan, V.; Gebreyes, K.; Hutchinson, J.; Iyer, R.; Laffitte, J. Al.; Wall, A. *J. Amer. Chem. Soc.* **1986**, *108*, 4568-4580.

(E)-1-Pentyl-2-(3-phenylpropylidene)cyclobutanol (7). Viscous oil. TLC: SiO₂, hexane/EtOAc (5:1), R_f ≈ 0.24; ¹H NMR (300 MHz, CDCl₃) δ 7.29-7.23 (m, 2H), 7.19-7.14 (m, 3H), 5.44-5.37 (m, 1H), 2.68-2.63 (t, *J* = 7.8 Hz, 2H), 2.34-2.19 (m, 3H) 2.10-1.98 (m, 2H), 1.87-1.76 (m, 2H), 1.58-1.51 (m, 2H), 1.42-1.24 (m, 7H), 0.88 (t, *J* = 6.5 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 148.61, 142.17, 128.81, 128.44, 126.02, 119.46, 79.59, 39.17, 38.86, 36.03, 34.40, 32.46, 29.86, 23.58, 22.93, 21.77, 14.34. HRMS calcd for C₁₈H₂₆O [M+1]⁺ 259.2062, found 259.2066.

(E)-1-Pentyl-2-((E)-3-phenylallylidene)cyclobutanol (9). Viscous oil. TLC: SiO₂, hexane/EtOAc (5:1), R_f ≈ 0.20; ¹H NMR (300 MHz, CDCl₃) δ 7.31-7.29 (m, 2H), 7.23-7.10 (m, 3H), 6.23 (dd, *J* = 11.4, 8.1 Hz, 1H), 6.41 (d, *J* = 11.4 Hz, 1H), 6.13 (td, *J* = 8.1, 1.8 Hz, 1H), 2.65-2.56 (m, 1H), 2.39-2.30 (m, 1H), 2.13-2.07 (m, 1H), 1.97-1.88 (m, 1H), 1.82 (br s, OH), 1.61-1.54 (m, 2H), 1.39-1.32 (m, 2H), 1.27-1.16 (m, 4H), 0.80 (t, *J* = 5.1 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 152.66, 138.02, 131.83, 129.06, 127.81, 126.72, 125.08, 120.84, 80.19, 39.17, 34.76, 32.68, 23.91, 23.15, 22.92, 14.57; HRMS calcd for C₁₈H₂₄O [M+1]⁺ 257.1905, found 259.1910.

(E)-1-Ethyl-2-((E)-3-phenylallylidene)cyclobutanol (11). Viscous oil. TLC: SiO₂, hexane/EtOAc (10:3), R_f ≈ 0.35; ¹H NMR (300 MHz, CDCl₃) δ 7.41-7.18 (m, 5 H), 6.73 (dd, *J* = 11.0, 10.0 Hz, 1H), 6.55 (d, *J* = 15.5 Hz, 1H), 6.23 (td, 1H, *J* = 11.0, 2.4 Hz, 1H), 2.78-2.64 (m, 1H), 2.49-2.36 (m, 1H), 2.23-2.15 (m, 1H), 2.01 (br sm, OH, 1H), 1.76-1.68 (m, 2H), 1.00 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 150.40, 137.22, 132.21, 128.69, 128.04, 126.87, 125.43, 120.55, 80.49, 35.47, 31.86, 25.38, 8.25. HRMS calcd for C₁₅H₁₈O [M+1]⁺ 215.1436, found 215.1440.

(E)-2-Benzylidene-1-pentylcyclobutanol (13). Viscous oil. TLC: SiO₂, hexane/EtOAc (5:1), R_f ≈ 0.21; ¹H NMR (300 MHz, CDCl₃) δ 7.28-7.10 (m, 5 H), 6.32 (t, *J* = 2.4 Hz, 1H), 2.80-2.55 (m, 1H), 2.26-2.16 (m, 1H), 2.07-1.97 (m, 1H), 1.91 (br s, OH), 1.69-1.61 (m, 2H), 1.46-1.35 (m, 2H), 1.31-1.15 (m, 4H), 0.82 (t, *J* = 6.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 150.67, 137.16,

128.68, 128.01, 126.87, 120.40, 80.30, 39.15, 35.98, 32.46, 25.40, 23.60, 22.93, 14.34. HRMS calcd for C₁₆H₂₂O [M+1]⁺ 231.1749, found 231.1753.

(E)-2-Benzylidene-1-(5-(tert-butylidiphenylsilyloxy)pentyl)cyclobutanol (15). Viscous oil. TLC: SiO₂, hexane/EtOAc (10:1), R_f ≈ 0.31; ¹H NMR (300 MHz, CDCl₃) δ 7.67-7.65 (m, 4H), 7.41-7.20 (m, 11H), 6.38 (t, *J* = 2.4 Hz, 1H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.86-2.62 (m, 2H), 2.32-2.22 (m, 1H), 2.17-2.03 (m, 1H), 1.72-1.40 (m, 8H), 1.04 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.52, 137.10, 135.77, 129.84, 128.64, 127.99, 127.92, 127.77, 126.86, 120.41, 80.19, 64.12, 39.13, 35.95, 32.80, 27.07, 26.39, 25.35, 23.68, 19.41; HRMS calcd for C₃₂H₄₀O₂Si [M+1]⁺ 485.2876, found 485.2881.

(E)-1-(5-(tert-Butylidiphenylsilyloxy)pentyl)-2-(naphthalen-2-ylmethylene)cyclobutanol (17). Viscous oil. TLC: SiO₂, hexane/EtOAc (10:1), R_f ≈ 0.35; ¹H NMR (300 MHz, CDCl₃) δ 7.81-7.77 (m, 3H), 7.69-7.66 (m, 4H), 7.47-7.35 (m, 10H), 6.47 (t, *J* = 2.2 Hz, 1H), 3.68 (t, *J* = 6.4 Hz, 2H), 2.92-2.72 (m, 2H), 2.74-2.52 (m, 1H), 2.39-2.19 (m, 1H), 1.80-1.73 (m, 2H), 1.74-1.45 (m, 8H), 1.05 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 151.08, 135.82, 134.76, 134.37, 133.80, 132.50, 129.72, 128.19, 128.15, 127.85, 127.81, 127.00, 126.38, 126.08, 125.93, 120.62, 80.33, 64.17, 39.24, 35.97, 32.85, 27.11, 26.45, 25.52, 23.74, 19.46; HRMS calcd for C₃₆H₄₂O₂Si [M+1]⁺ 535.3032, found 535.3037.

(E)-2-(4-Bromobenzylidene)-1-(5-(tert-butylidiphenylsilyloxy)pentyl)cyclobutanol (19). Viscous oil. TLC: SiO₂, hexane/EtOAc (10:1), R_f ≈ 0.31; ¹H NMR (300 MHz, CDCl₃) δ 7.68-7.65 (m, 4H), 7.45-7.36 (m, 8H), 7.13-7.10 (m, 2H), 6.32 (t, *J* = 2.4 Hz, 1H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.82-2.59 (m, 2H), 2.32-2.23 (m, 1H), 2.16-2.05 (m, 1H), 1.75-1.41 (m, 8 H), 1.04 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 151.52, 136.04, 135.80, 134.35, 131.76, 129.73, 129.61, 127.81, 120.60, 119.42, 80.20, 64.13, 39.13, 35.89, 32.80, 27.10, 26.40, 25.35, 23.67, 19.46; HRMS calcd for C₃₂H₃₉BrO₂Si [M+1]⁺ 563.1981, found 563.1984.

(E)-1-(2-(tert-Butylidiphenylsilyloxy)ethyl)-2-(2,4-difluorobenzylidene)cyclobutanol (21). Viscous oil. TLC: SiO₂, hexane/EtOAc (5:1), R_f ≈ 0.35; ¹H NMR (300 MHz, CDCl₃) δ 7.67-7.65 (m, 4H), 7.42-7.34 (m, 6H), 7.29-7.23 (m, 1H), 6.85-6.77 (m, 2H), 6.51 (t, *J* = 2.2 Hz, 1H), 3.66

(t, $J = 6.4$ Hz, 2H), 2.72-2.59 (m, 2H), 2.28-2.22 (m, 1H), 2.12-2.04 (m, 1H), 1.96 (br s, OH), 1.74-1.65(m, 2H), 1.63-1.55 (m, 2H), 1.48-1.39 (m, 4H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.60 (dd, $J_{\text{c,f}} = 8.85$ Hz, 143.7 Hz), 159.28 (dd, $J_{\text{c,f}} = 12.0$, 134.9 Hz), 152.15, 135.79, 134.34, 129.71, 129.42 (dd, $J_{\text{c,f}} = 4.3$, 8.45 Hz), 127.79, 120.12 (dd, $J_{\text{c,f}} = 2.3$, 4.6 Hz), 111.24 (dd, $J_{\text{c,f}} = 3.6$, 4.7 Hz), 104.21 (dd, $J_{\text{c,f}} = 3.8$, 5.1 Hz), 80.16, 64.11, 39.05, 35.68, 32.77, 27.08, 26.37, 25.17, 23.64, 19.44. HRMS calcd for $\text{C}_{32}\text{H}_{38}\text{F}_2\text{O}_2\text{Si}$ $[\text{M}+1]^+$ 521.2687, found 521.2691.

(E)-2-Benzylidene-1-ethyl-4-phenethylcyclobutanol (23: less polar). Viscous oil. TLC: SiO_2 , hexane/EtOAc (10:3), $R_f \approx 0.36$; ^1H NMR (300 MHz, CDCl_3) δ 7.35-7.16 (m, 5H), 6.44 (t, $J = 2.4$ Hz, 1H), 2.94-2.86 (m, 2H), 2.66 (t, $J = 7.2$ Hz, 2H), 2.46-2.32 (m, 2H), 2.01-1.89 (m, 2H), 1.91 (br s, 1H), 1.80-1.72 (m, 2H), 1.68-1.58 (m, 1H), 1.01 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.55, 142.50, 137.12, 128.69, 128.66, 128.59, 128.01, 126.92, 126.01, 121.75, 80.81, 43.73, 34.12, 32.82, 32.49, 32.06, 8.16; HRMS calcd for $\text{C}_{21}\text{H}_{24}\text{O}$ $[\text{M}+1]^+$ 293.1905, found 293.1911.

(E)-2-Benzylidene-1-ethyl-4-phenethylcyclobutanol (23: more polar). TLC: SiO_2 , hexane/EtOAc (10:3), $R_f \approx 0.33$; ^1H NMR (300 MHz, CDCl_3) δ 7.34-7.16 (m, 5H), 6.38 (t, $J = 2.0$ Hz, 1H), 2.93-2.85 (m, 2H), 2.70-2.57 (m, 2H), 2.32-2.25 (m, 2H), 2.05-1.96 (m, 1H), 1.85 (br s, OH), 1.80-1.66 (m, 3H), 1.03 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.61, 142.37, 137.14, 128.69, 128.64, 128.60, 128.03, 126.85, 126.07, 120.18, 81.66, 48.68, 34.43, 31.78, 31.62, 26.25, 7.79; HRMS calcd for $\text{C}_{21}\text{H}_{24}\text{O}$ $[\text{M}+1]^+$ 293.1905, found 293.1909.

(E)-8-Benzylidenebicyclo[4.2.0]octan-1-ol (25). Viscous oil. TLC: SiO_2 , benzene/ CH_2Cl_2 (5:1), $R_f \approx 0.3$; ^1H NMR (300 MHz, CDCl_3) δ 7.33-7.17 (m, 5H), 6.38 (t, $J = 2.2$ Hz, 1H), 2.78-2.72 (m, 1H), 2.57-2.50 (m, 1H), 2.40-2.34 (m, 1H), 1.88-1.56 (m, 6H), 1.43-1.38 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 150.03, 137.46, 128.61, 127.90, 126.69, 117.00, 77.02, 43.33, 33.86, 29.46, 24.07, 21.10, 20.92; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{O}$ $[\text{M}+1]^+$ 215.1436, found 215.1442.

9-Chloro-12-phenyldodeca-9(Z),11(E)-dien-6-one (28). A solution of (E)-(4,4,4-trichlorobut-1-enyl)benzene (**8**, 0.2 mmol) and oct-1-en-3-one (**6**, 0.24 mol.) in dry THF (4 mL) was added to

a room temperature, suspension of chromous chloride (1.2 mmol, 6 equiv; Aldrich Chem. Co.) and copper(I) triflate (0.24 mmol, 1.2 equiv; Aldrich Chem. Co.) in dry THF (4 mL). After 12 h, the reaction was quenched with sat. aq. NaHCO₃ (3 mL) and extracted with ether (3 × 30 mL). The combined ethereal extracts were washed with water (2 × 30 mL), dried, and concentrated under reduced pressure. The residue was purified by SiO₂ column chromatography using a gradient of hexane to hexane/ethyl acetate (10:1) affording **28** (35%) as a white solid, mp 84-86 °C. TLC: SiO₂, EtOAc/hexane (1:90, R_f ≈ 0.4; ¹H NMR (300 MHz, CDCl₃) δ 7.45-7.23 (m, 5 H), 7.05 (dd, *J* = 15.9, 10.1 Hz, 1H) 6.60 (d, *J* = 15.9, 1H), 6.29 (d, *J* = 10.1 Hz, 1H), 2.70 (s, 4H), 2.42 (t, *J* = 7.3 Hz, 2H), 1.61-1.57 (m, 2H), 1.27-1.20 (m, 4H), 0.89 (t, *J* = 6.7 Hz, 3H); ¹H NMR (300 MHz, C₆D₆) δ 7.28-7.00 (m, 5 H), 6.37 (d, *J* = 15.9 Hz, 1H), 6.05 (dd, *J* = 10.9, 0.6 Hz, 1H), 2.57 (t, *J* = 7.3 Hz, 2H), 2.23 (t, *J* = 7.3 Hz, 2H), 1.88 (t, *J* = 7.3 Hz, 2H), 1.47-1.37 (m, 2H), 1.18-1.01 (m, 4H), 0.79 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 207.07, 137.26, 134.93, 134.45, 128.79, 128.23, 128.03, 127.90, 127.58, 126.84, 126.48, 124.00, 42.61, 40.12, 34.06, 31.51, 23.54, 22.68, 14.01; HRMS calcd for C₁₈H₂₃ClO [M+1]⁺ 291.1516, found 291.1519.

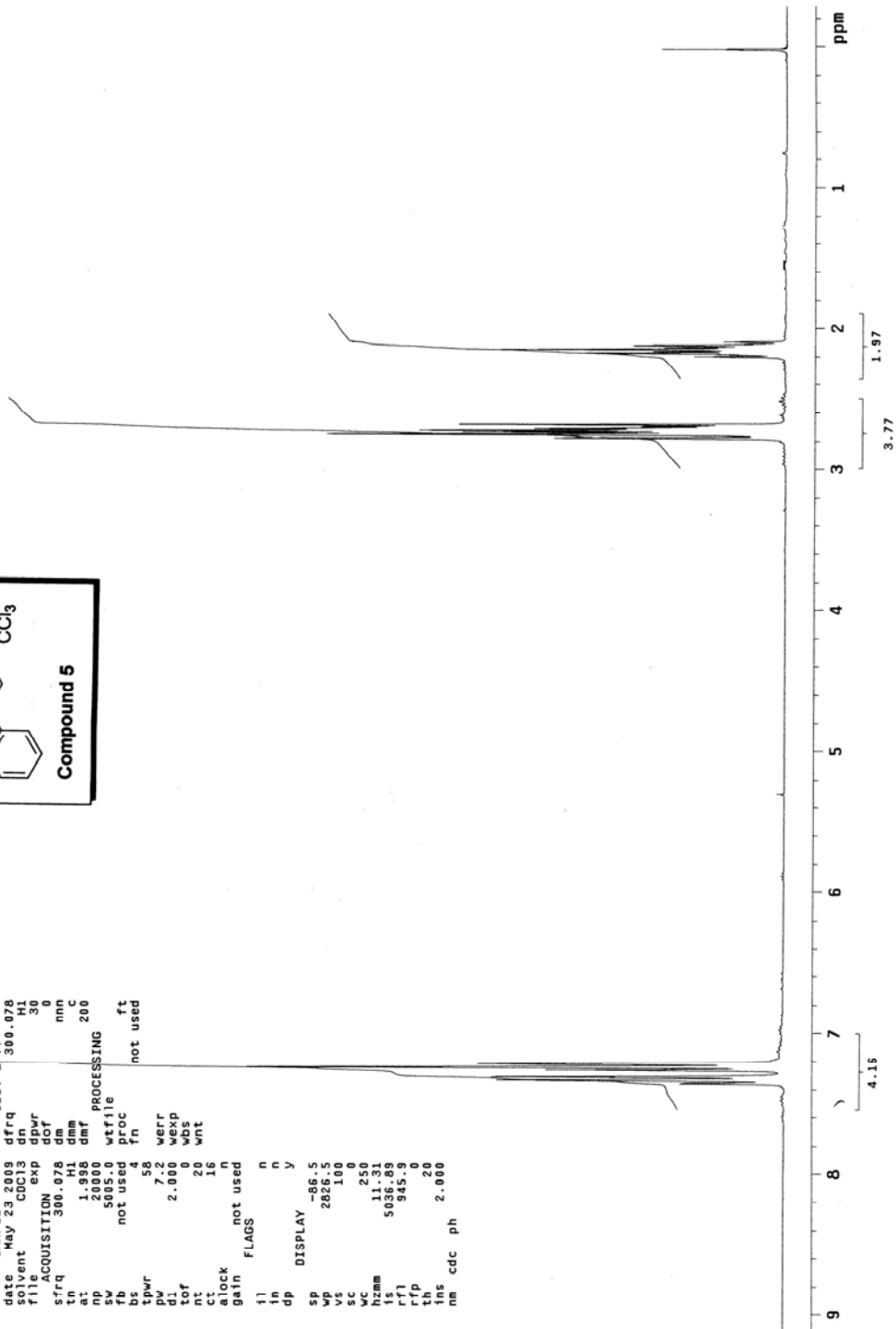
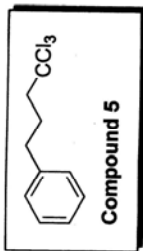
(E)-1-(5-hydroxypentyl)-2-(naphthalen-2-ylmethylene) cyclobutanol (Desilylated adduct for compound 17). White crystals. TLC: SiO₂, EtoAc/hexanes (5:5), R_f ≈ 0.25; ¹H NMR (300 MHz, CDCl₃) δ 7.82-7.75(m, 3H), 7.69-7.66 (m, 1H), 7.49-7.40 (m, 3H), 6.55 (t, *J* = 2.4 Hz, 1H), 3.65 (t, *J* = 6.6 Hz, 2H), 2.97-2.73 (m, 2H), 2.36-2.25 (m, 1H), 2.17-2.07 (m, 1H), 1.80-1.73 (m, 2H), 1.64-1.28 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 151.05, 134.73, 133.79, 132.50, 128.20, 128.14, 127.85, 126.99, 126.40, 126.06, 125.95, 120.63, 80.21, 63.13, 39.09, 35.99, 32.95, 26.33, 25.50, 23.72.

STANDARD 1H OBSERVE

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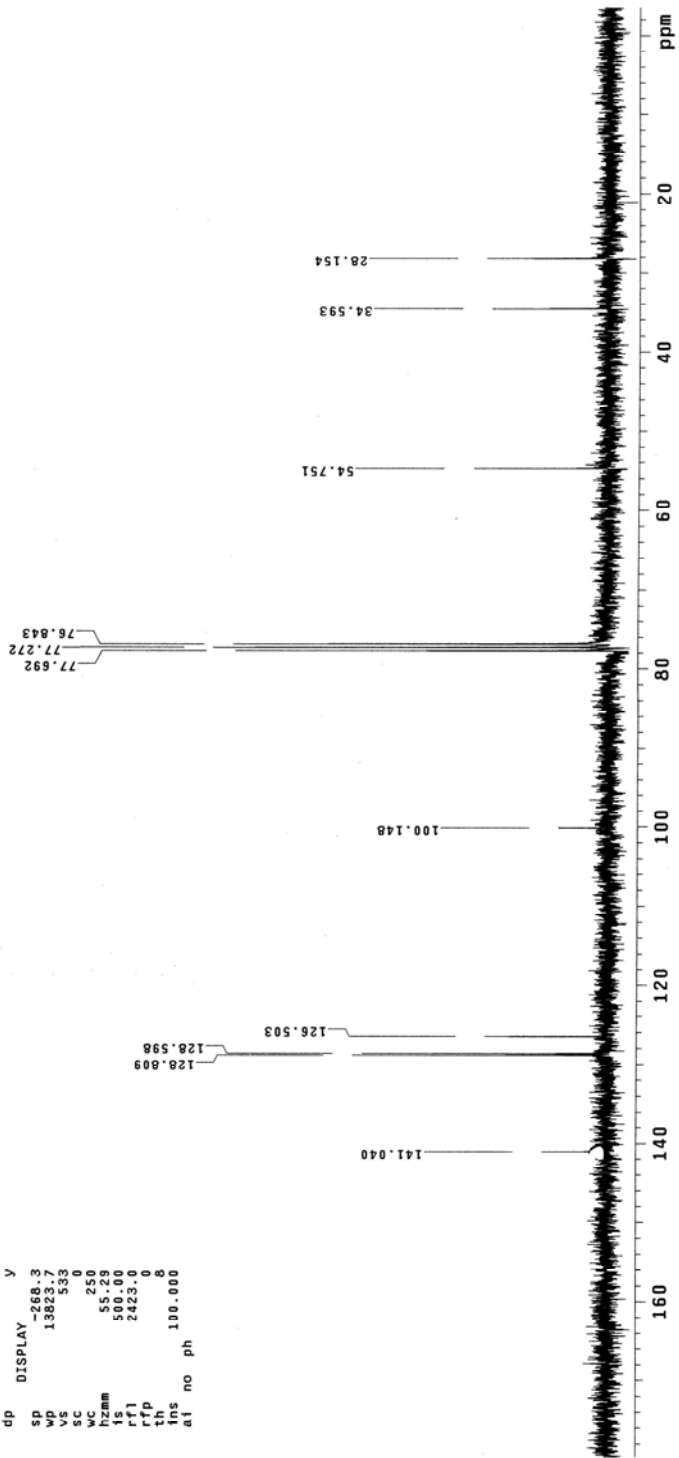
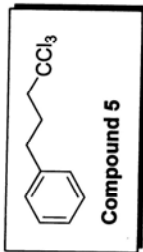
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bs 4 fn not used
tpwr 58
pw 7.2 werr
d1 2.000 wexp
tof 0 wds
ct 20 wnt
16
allock n
gain not used
FLAGS
f1 n
in n
dp n y
SP DISPLAY -86.5
wp 2826.5
vs 1.00
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wc 11250
hzmm 11.38
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rff 845.9
th 20
ins cdc ph 2.000

```



13C OBSERVE

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f1file CDexp d1pwr 37
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sfrq 75.462 dm nvy W
tn C13 dmm W
at 1.000 dmf 10400 W
cp 20000 lb PROCESSING
fb not used wtf1file 1.00
bs 20 proc ft
tpwr 56 fn not used
pw 4.7 werr
d1 0 wexp
d2 1.000 wds
nt 1000 wnt
ct 120
alock n
gain not used
FLAGS n
ll n
in n
dp n y
DISPLAY
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vs 533
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wc 250
fzmm 500.00
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rfp 0
th 8
ins 100.000
at no ph
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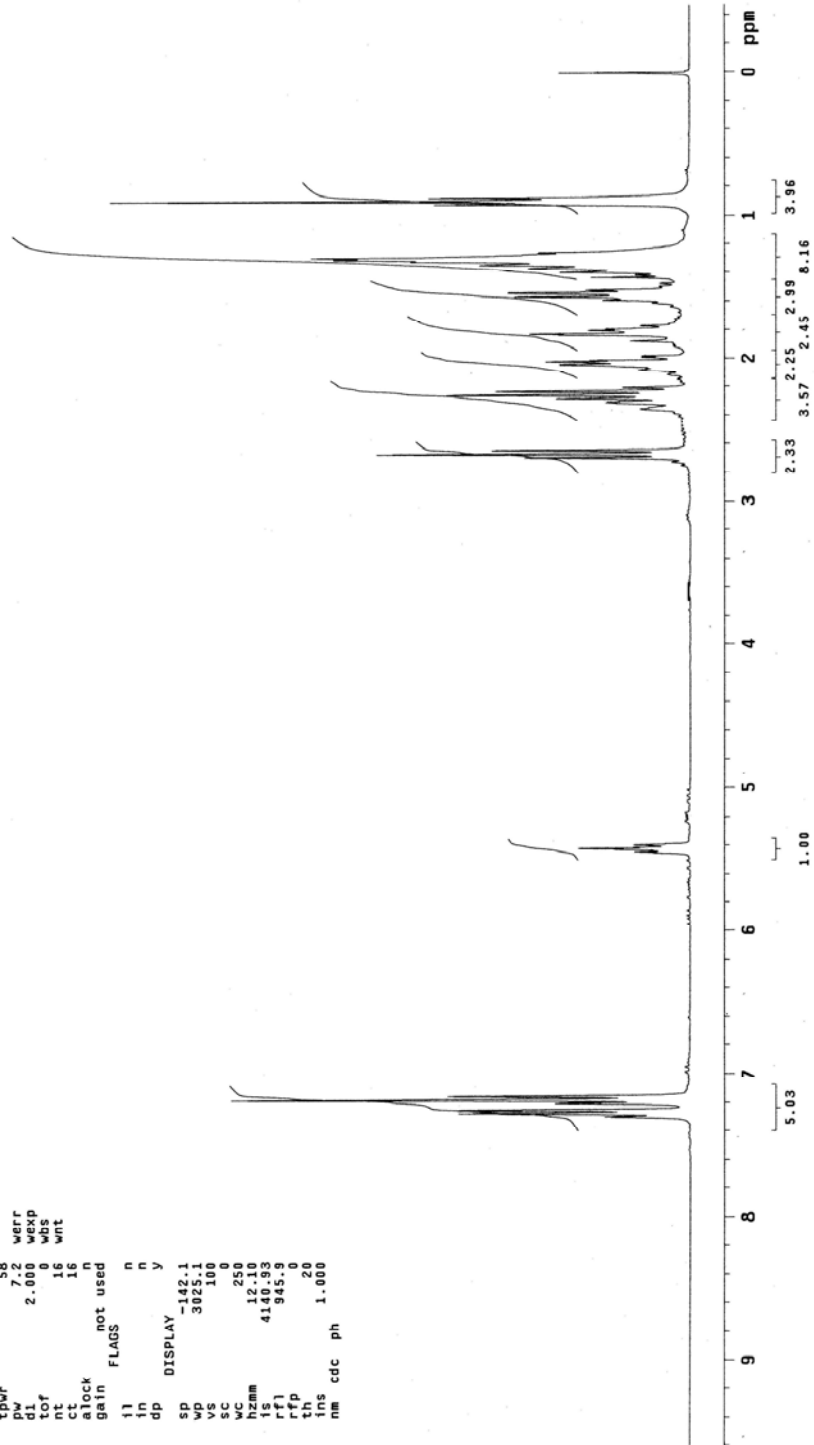
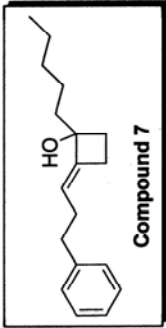


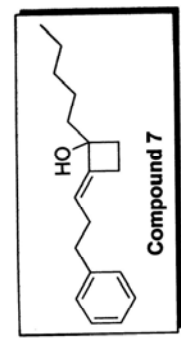
STANDARD 1H OBSERVE

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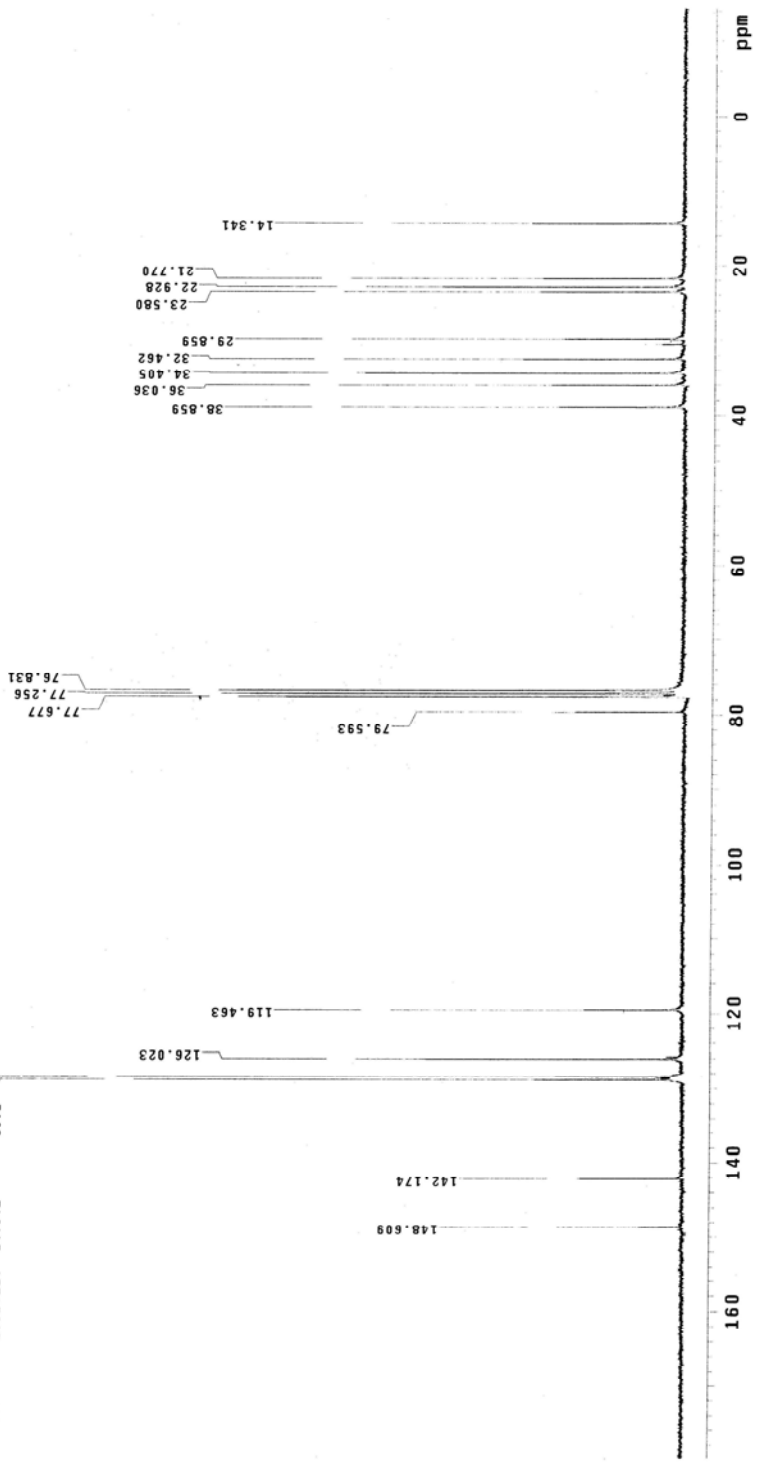
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at 1.988 dmf 200
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fb not used fn not used
t1 55
t2 55
t3 7.2 werr
d1 2.000 wexp
nt 0 wbs
ct 16 wnt
alock not used
gain FLAGS
{ } n n
in n
dp n
DISPLAY
sp -182.1
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ins 1.000
na cdc ph

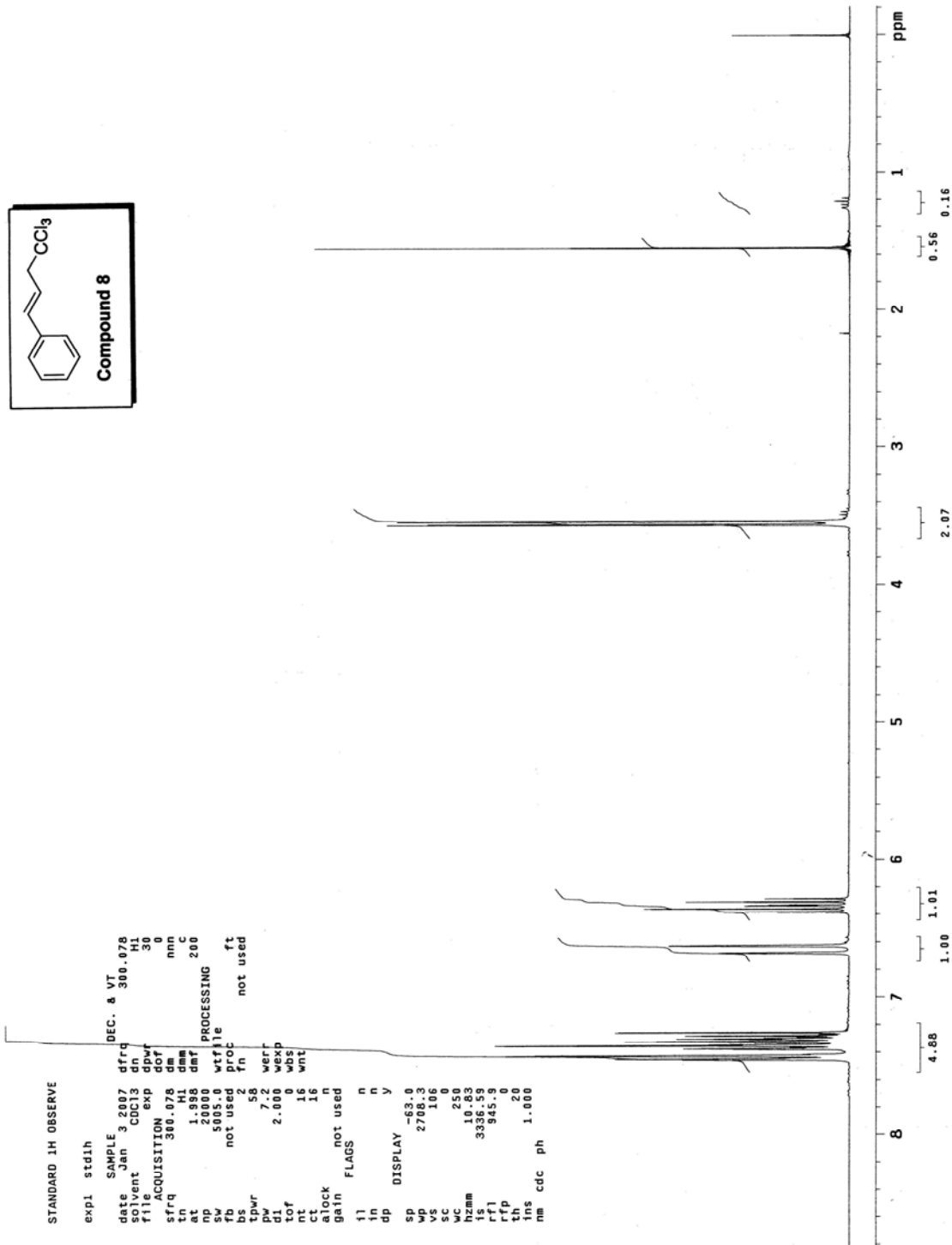
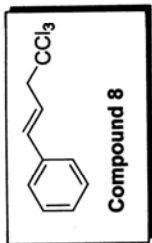
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5	9508.930	126.023	55.3
6	9013.942	119.463	49.4
7	6005.653	79.593	22.4
8	5861.079	77.677	75.9
9	5823.501	77.256	76.9
10	5797.237	76.831	79.3
11	2332.091	38.859	58.7
12	2719.895	36.036	59.1
13	2595.892	34.405	56.0
14	2449.414	32.462	55.3
15	2253.022	29.859	57.0
16	1779.219	23.560	58.3
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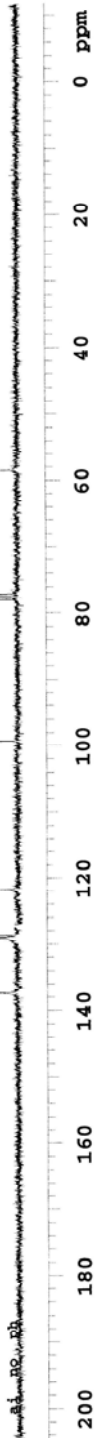
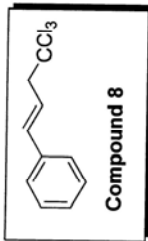


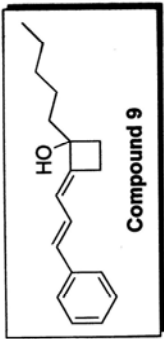


13C OBSERVE

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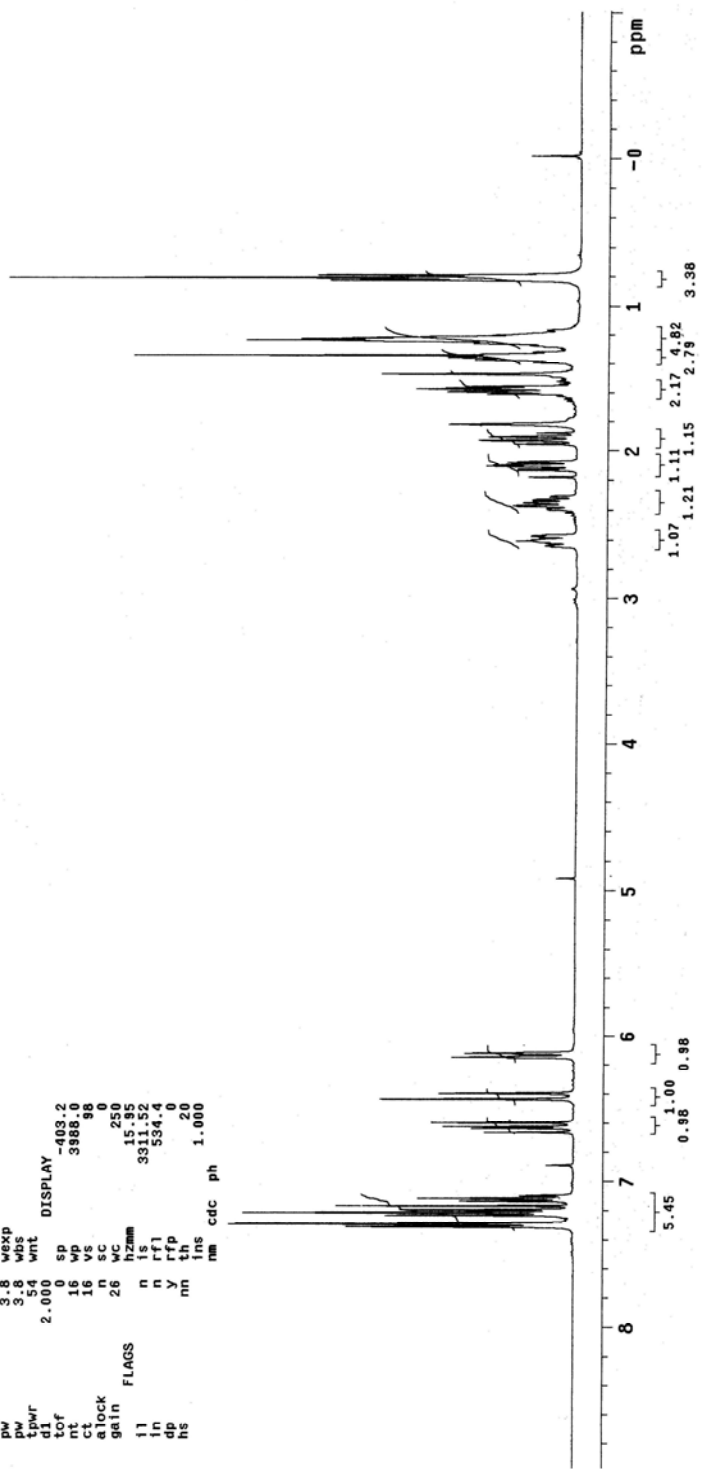
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sw 20000.0 lb PROCESSING
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bs 10 proc ft
tpwr 56 fn not used
pw 4.7
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d2 1.000 wexp
tof 0 wbs
nt 300 wnt
ct 130
alock n
gain not used
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in n
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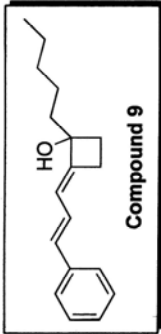




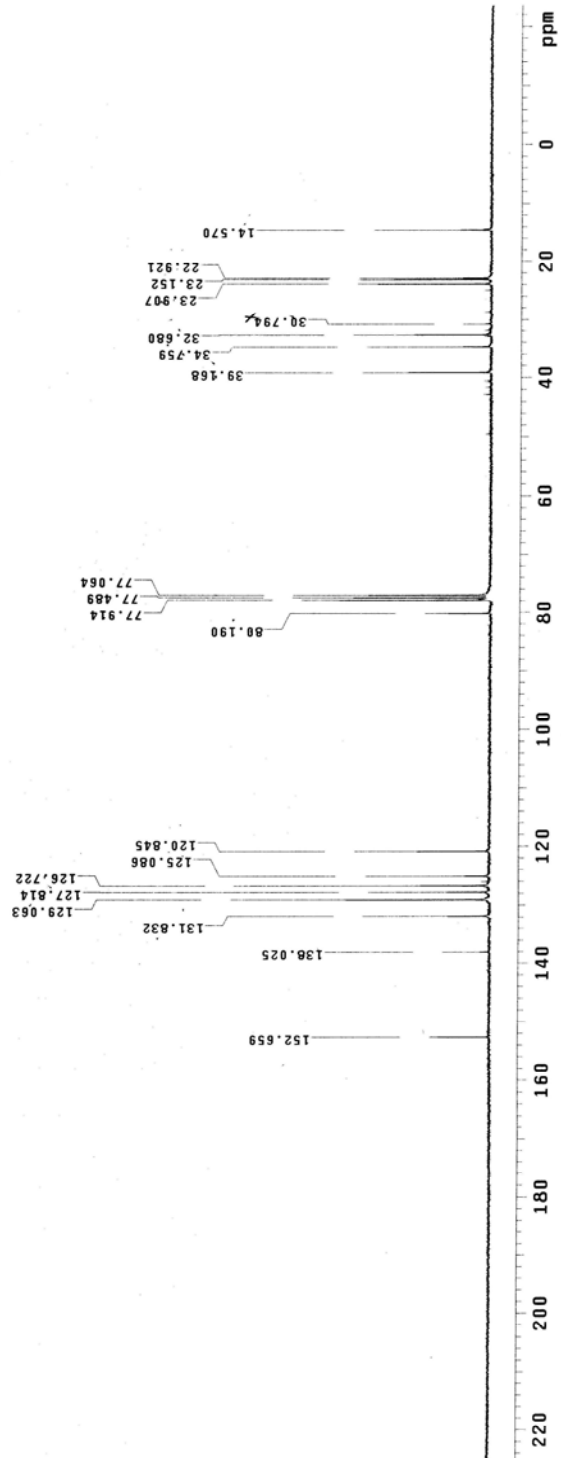
1H SENSITIVITY
 0.1% ETHYLBENZENE
 UTSW INOVA 400
 Sensitivity
 November 19, 1997
 Abe Revilla
 exp1 s2pu1

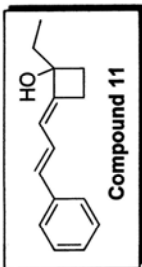
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 fb 3000
 ss 2 werr
 pw 3.8 wexp
 ps 3.8 wps
 tpr 54 wnt
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 gain 28 hzmm 25.95
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INDEX	FREQUENCY PPM	HEIGHT
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13	2955.352	39.168
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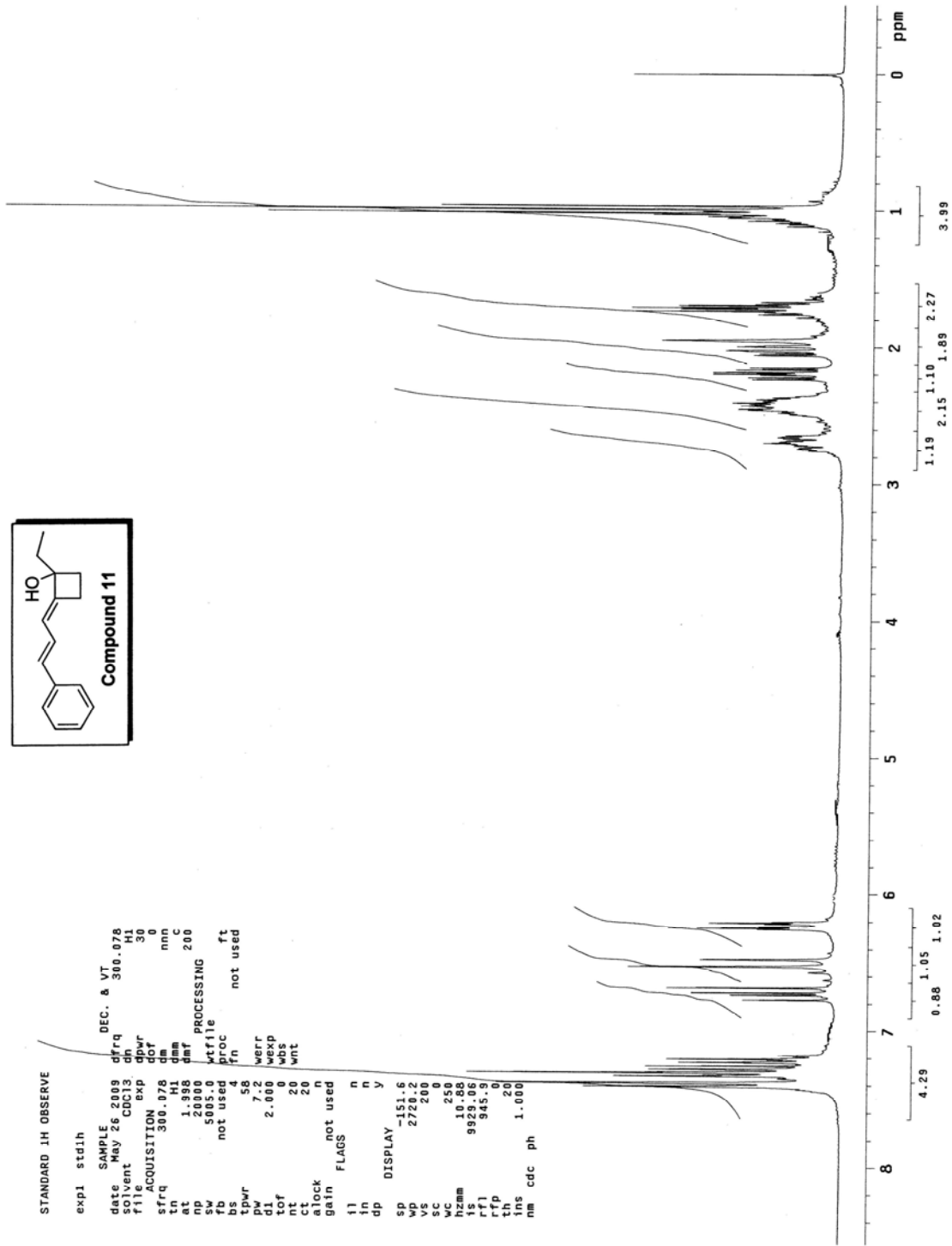


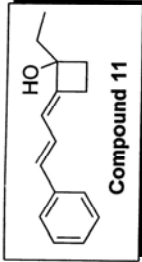


STANDARD 1H OBSERVE

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d1 2.000        wexp
tof 0           wbs
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ct 20
alock
gain not used  n
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DISPLAY
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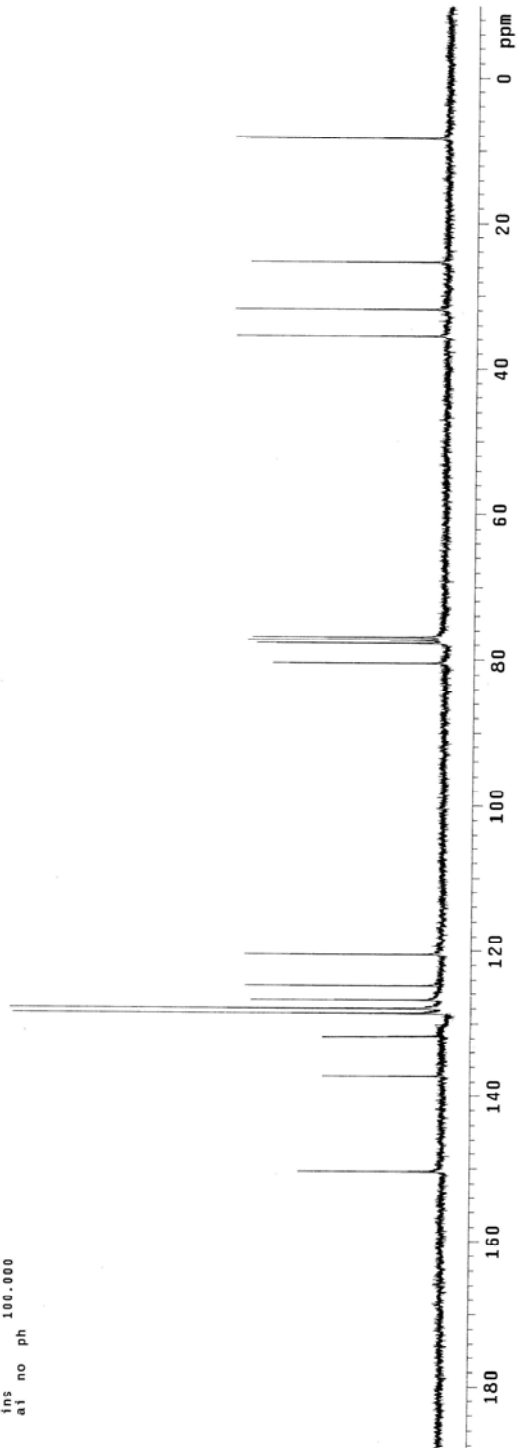




13C OBSERVE

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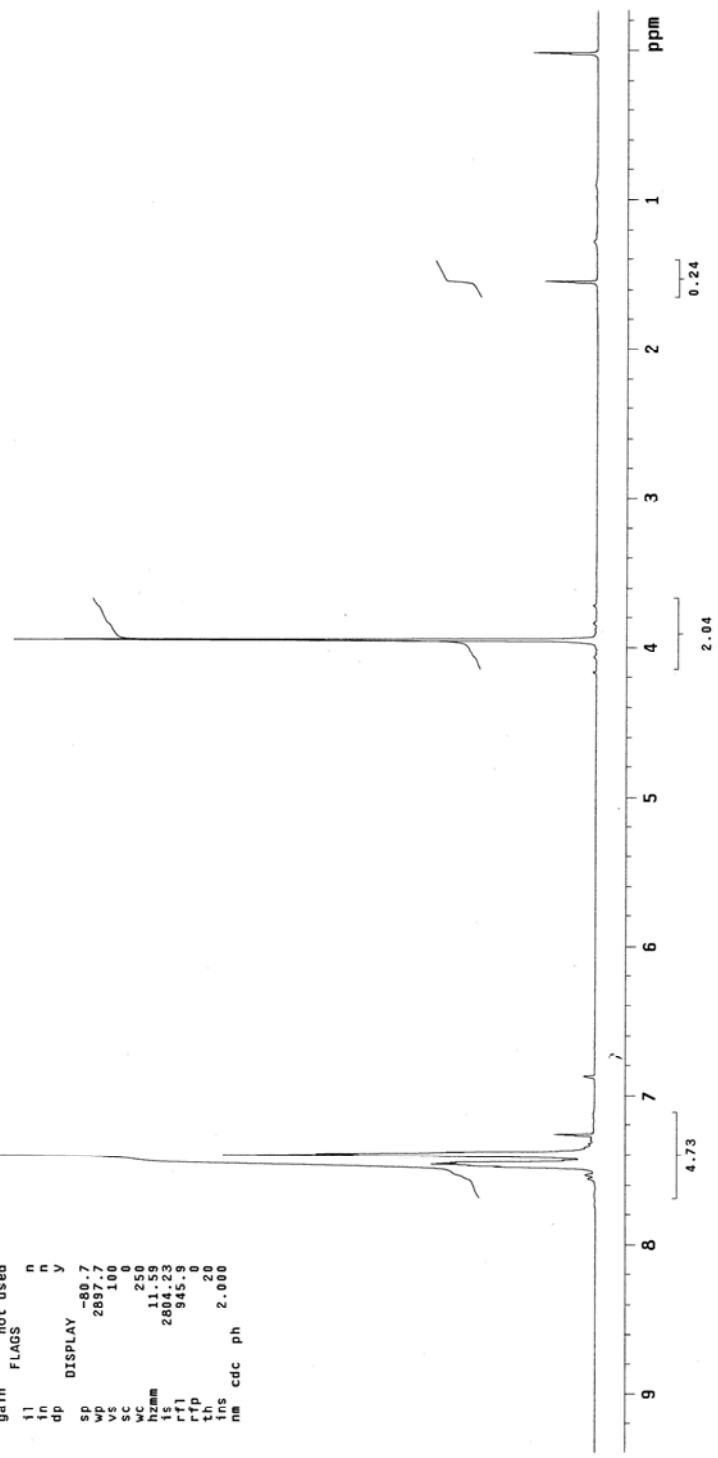
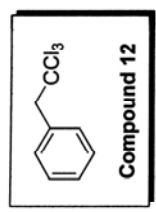
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bs not used
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pw 56 fn not used
d1 4.7
d2 1.000 werr
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ln n
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STANDARD 1H OBSERVE

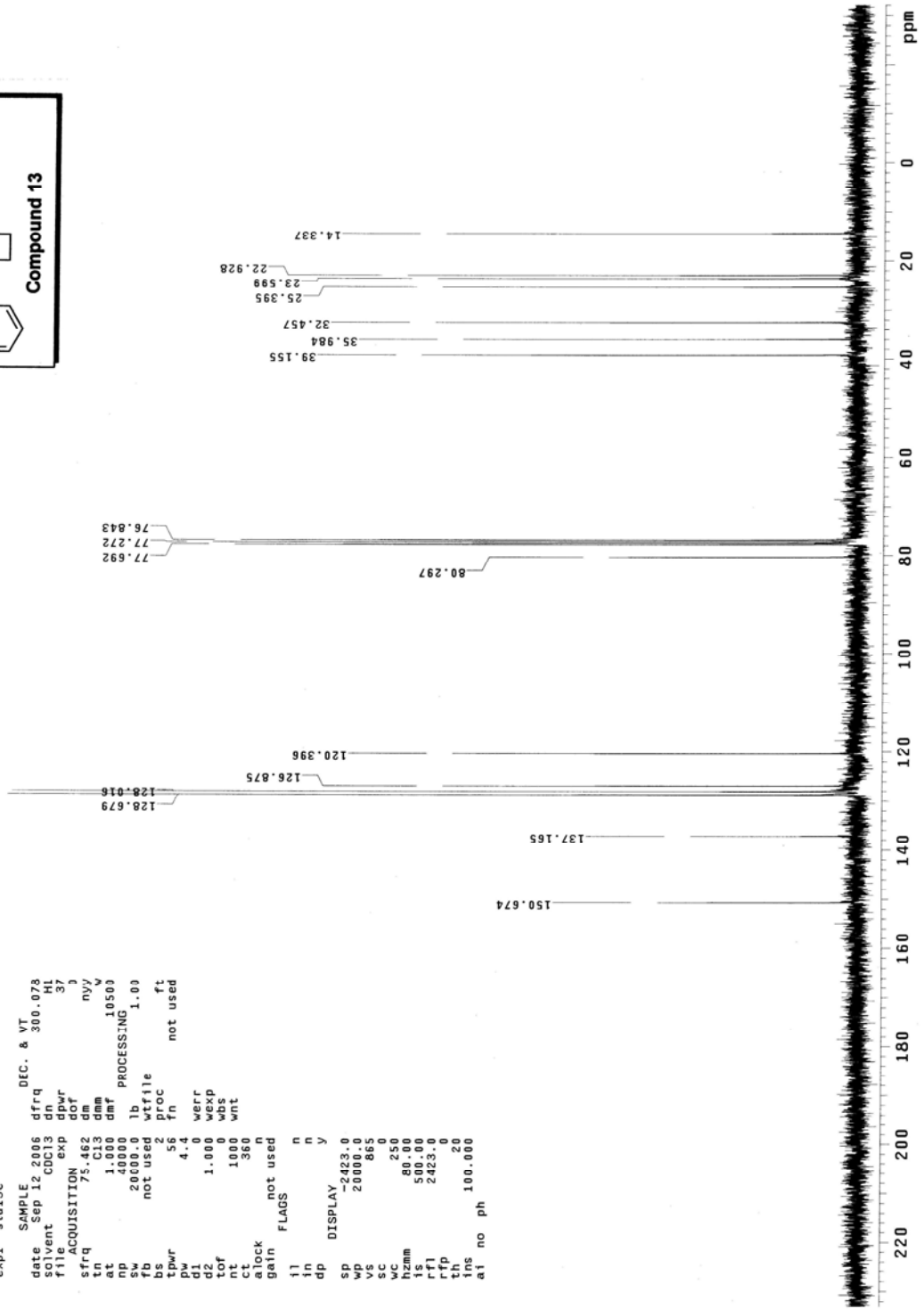
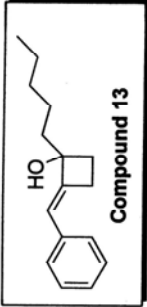
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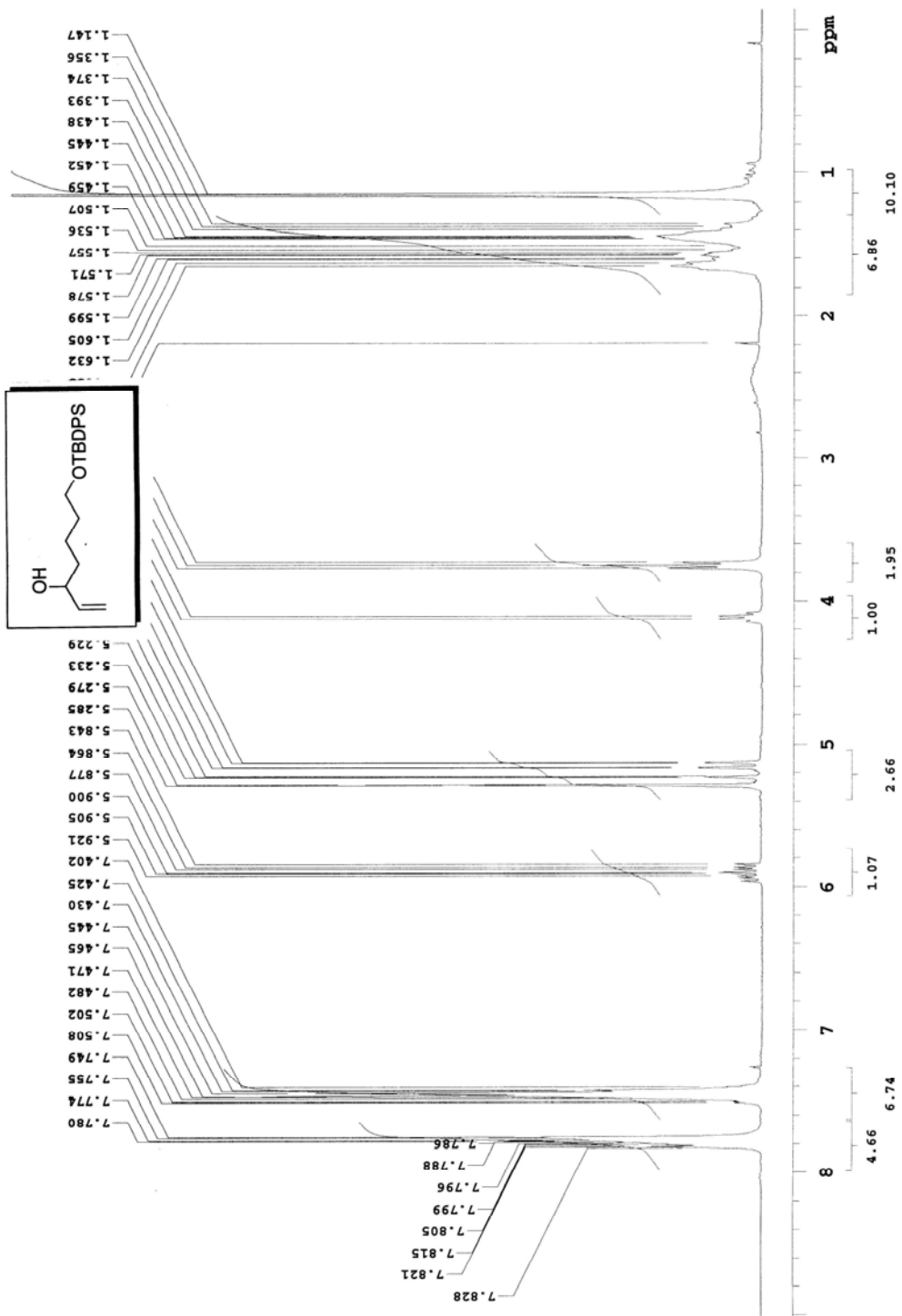
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nm cdc ph 2.000
    
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13C OBSERVE

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in n
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at no ph
100.000
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13C OBSERVE

exp1 std13c

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bs 10 proc ft
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dp y
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141.533

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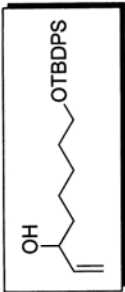
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27.223

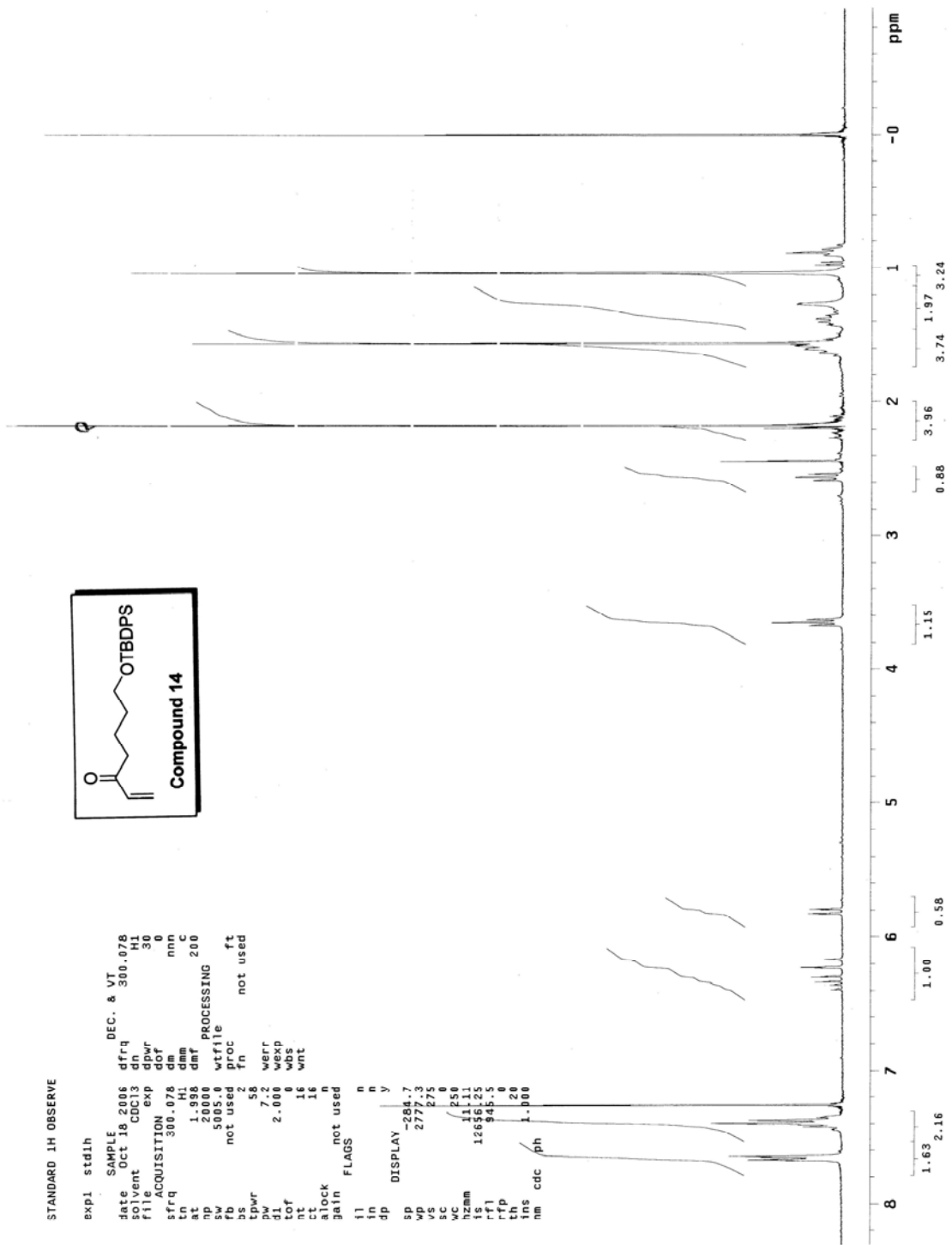
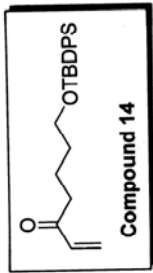


180 160 140 120 100 80 60 40 20 ppm

STANDARD 1H OBSERVE

```

expl stdiH
SAMPLE
date Oct 18 2006          DEC. & VT
solvent CDC13            dfrq 300.078
fl 1                    dm 31
ACQUISITION              dpr 30
sfrq 300.078            dm  nnn
tn 1.998                dmf 200
at 20000                PROCESsing
pw 3005.0               wtfile
not used                proc
bs 2                     fn  not used
tpwr 58
pw 7.2                  werr
d1 2.000                wexp
tof 0                   wbs
nt 16                   wnt
clock 16
gain not used
FLAGS
il n
in n
dp n
SP DISPLAY -284.7
WD 2772.3
VS 275
SC 0
WC 250
hzmm 11.11
rf 1263623
rfl 341.0
rfp 20
th 1.000
ins cdc
nm cdc /ph
  
```



13C OBSERVE

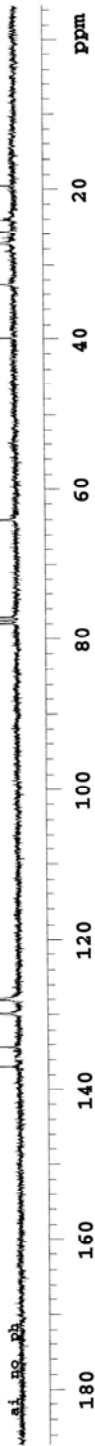
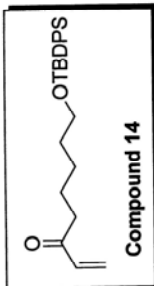
expl std13c

SAMPLE DEC. & VT
date Aug 17 2009 dfrq 300.078
solvent CDCl3 dn H1
file exp 37
ACQUISITION dof 0
sfrq 75.462 dm RNY
tn C13 dmm w
at 1.000 dmf 10400
np 40000 PROCESSING
sw 20000.0 lb 1.00
fb not used wfile
bs 10 proc ft
tpwr 56 fn not used
pw 4.7

d1 0 werr
d2 1.000 wexp
tof 0 wbs
nt 300 wnt
ct 300
alock n
gain not used

FLAGS
il n
in n
cp y

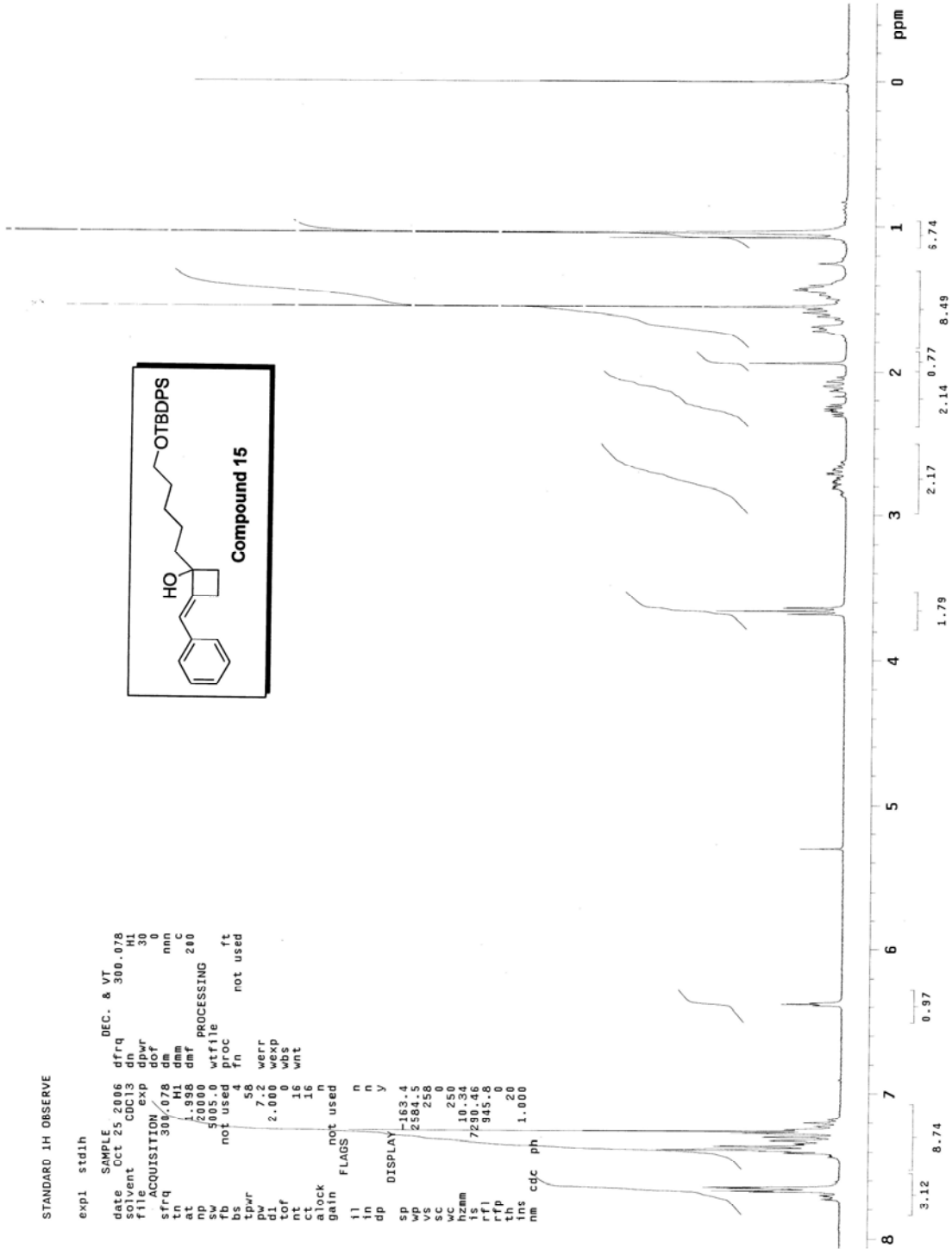
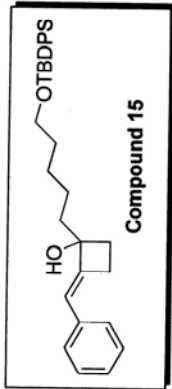
DISPLAY
sp -347.1
wp 14480.4
vs 228
sc 0
wc 250
hzmm 57.92
ls 500.00
rf1 2423.0
rfp 0
th 8
ins 100.000



STANDARD 1H OBSERVE

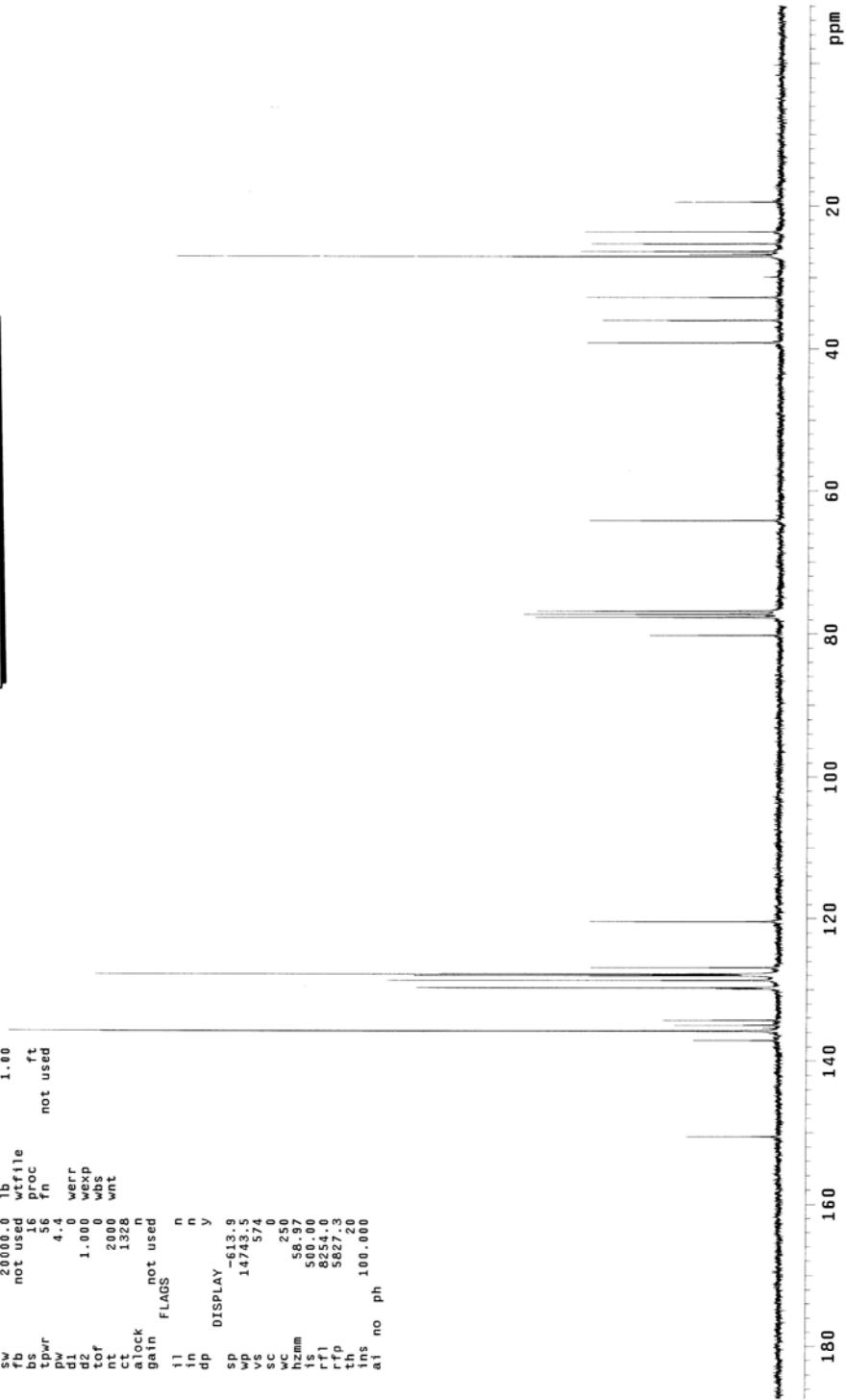
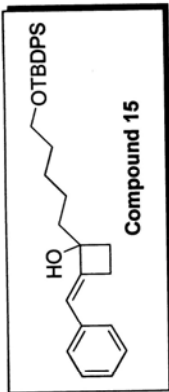
```

exp1 stdih
SAMPLE
date Oct 25 2006 DEC. & VT
file C0016 dfrq 300.078
file C0016 d1 16
file C0016 d2 31
file C0016 d3 31
file C0016 d4 31
file C0016 d5 31
file C0016 d6 31
file C0016 d7 31
file C0016 d8 31
file C0016 d9 31
file C0016 d10 31
file C0016 d11 31
file C0016 d12 31
file C0016 d13 31
file C0016 d14 31
file C0016 d15 31
file C0016 d16 31
file C0016 d17 31
file C0016 d18 31
file C0016 d19 31
file C0016 d20 31
file C0016 d21 31
file C0016 d22 31
file C0016 d23 31
file C0016 d24 31
file C0016 d25 31
file C0016 d26 31
file C0016 d27 31
file C0016 d28 31
file C0016 d29 31
file C0016 d30 31
file C0016 d31 31
file C0016 d32 31
file C0016 d33 31
file C0016 d34 31
file C0016 d35 31
file C0016 d36 31
file C0016 d37 31
file C0016 d38 31
file C0016 d39 31
file C0016 d40 31
file C0016 d41 31
file C0016 d42 31
file C0016 d43 31
file C0016 d44 31
file C0016 d45 31
file C0016 d46 31
file C0016 d47 31
file C0016 d48 31
file C0016 d49 31
file C0016 d50 31
file C0016 d51 31
file C0016 d52 31
file C0016 d53 31
file C0016 d54 31
file C0016 d55 31
file C0016 d56 31
file C0016 d57 31
file C0016 d58 31
file C0016 d59 31
file C0016 d60 31
file C0016 d61 31
file C0016 d62 31
file C0016 d63 31
file C0016 d64 31
file C0016 d65 31
file C0016 d66 31
file C0016 d67 31
file C0016 d68 31
file C0016 d69 31
file C0016 d70 31
file C0016 d71 31
file C0016 d72 31
file C0016 d73 31
file C0016 d74 31
file C0016 d75 31
file C0016 d76 31
file C0016 d77 31
file C0016 d78 31
file C0016 d79 31
file C0016 d80 31
file C0016 d81 31
file C0016 d82 31
file C0016 d83 31
file C0016 d84 31
file C0016 d85 31
file C0016 d86 31
file C0016 d87 31
file C0016 d88 31
file C0016 d89 31
file C0016 d90 31
file C0016 d91 31
file C0016 d92 31
file C0016 d93 31
file C0016 d94 31
file C0016 d95 31
file C0016 d96 31
file C0016 d97 31
file C0016 d98 31
file C0016 d99 31
file C0016 d100 31
ACQUISITION
sfrq 300.078 dm nnn
tn 16 d16
at 2.998 dm
np 50000 dmf
fb not used wfile
bs not used proc
tpwr 58 fn
pw 7.2 werr
d1 2.000 wexp
tof 0 wbs
ct 16 wnt
gain not used
alock n
gain not used
FLAGS
ll n
ln n
dp y
SP -163.4
WP 2584.5
VS 258
SC 0
WC 1.250
IC 10.34
IC2 728.0
IC3 945.8
rfl 0
rfp 0
th 20
ins 1.000
nm cdc ph
  
```



13C OBSERVE

```
exp1 std13c
SAMPLE      DEC. & VT
date Oct 31 2006 dfrq 300.076
solvent CDCl3  dr 37
file C0C13  exp 0
ACQUISITION  nvy
sfrq 75.462  dm
tr 1.000  dnm
at 200000  dmf 10500
ns 200000  lb  PROCESSING 1.00
fb not used  wfile
bs not used  proc
tpwr 56  fn  not used
pw 4.4
d1 1.000  verr
d2 1.000  wexp
d3 1.000  wst
at 2000  wnt
ct 1328
alock not used
gain not used
FLAGS      n
l1 n
n u
dp DISPLAY y
SP -613.9
wp 14743.5
vs 574
sc 0
Kcmm 25.7
Kcmm 56.37
ls 500.00
rf1 8254.0
rfp 5827.3
th 20
lms 100.000
at no ph
```



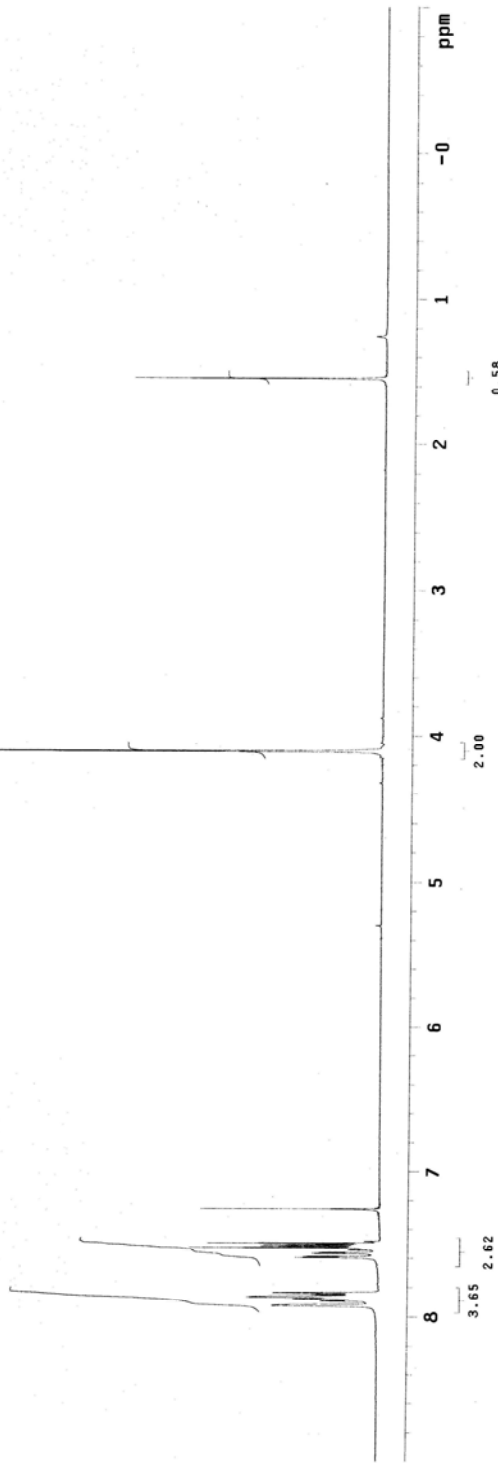
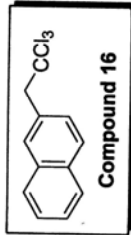
1H SENSITIVITY
0:1XETHYLBENZENE

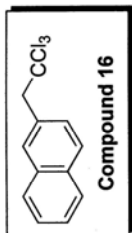
UTSW 300 Mercury
November 20, 1997

Ken Kezeor

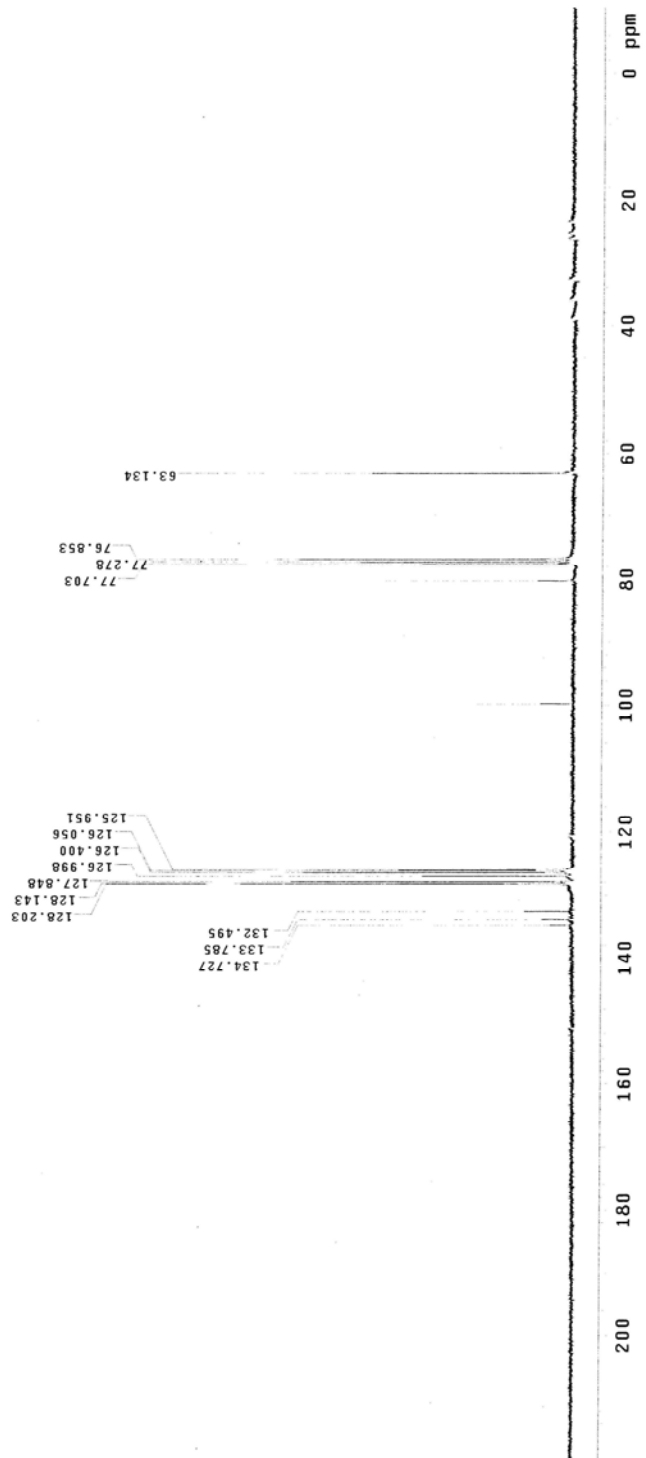
exp1 s2pu1

SAMPLE DEC. & VT
date Oct 29 2002 dn
solvent cdc13 dof -776.0
fil nm
ACQUISITION exp dmh
sfrq 300.078 dmf PROCESSING 200
tn H1 32768
at 2.000 fn
np 16000 werr
pw 4000.0 wexp
pr 2500 wpc
pt 6.0 wnt
tpwr 58 DISPLAY
dl 2.000 sp -301.8
tof 0 wp 3001.2
nt 16 vs 90
sl 16 sc 250
slock 0
gain 8 hzmm
ll n rfl 11409.24
in y rfp 443.7
dp y th 20
nm cdc ph 2.000





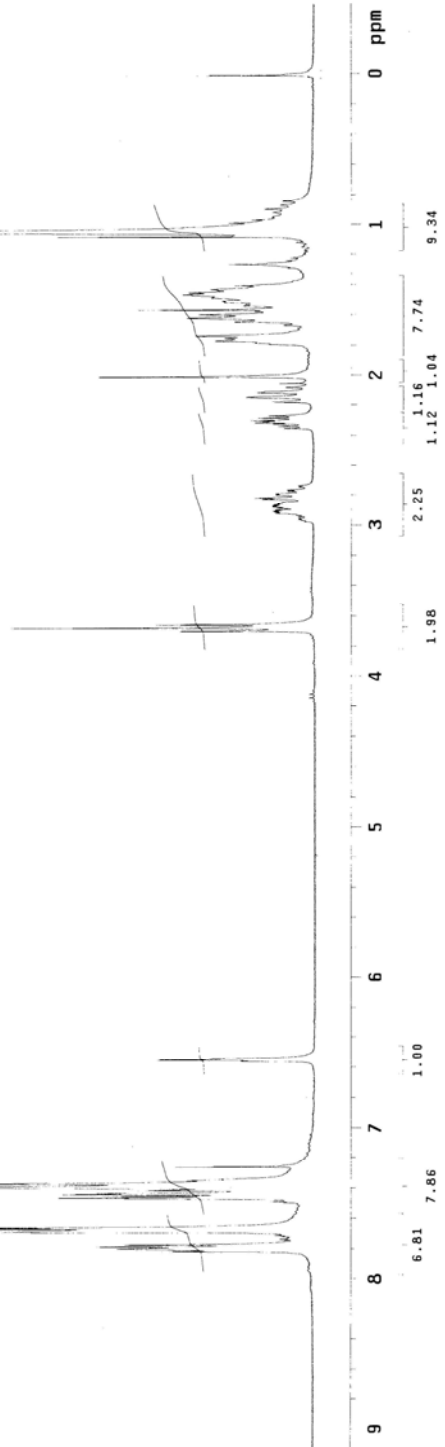
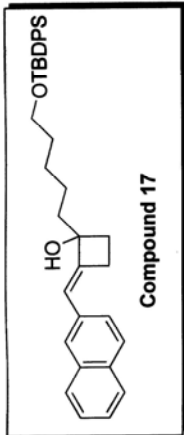
INDEX	FREQUENCY PPM	HEIGHT
1	11397.459	151.051
2	10165.740	134.727
3	10094.633	133.785
4	9997.281	132.495
5	9673.484	128.203
6	9668.907	128.143
7	9646.628	127.848
8	9582.540	126.998
9	9537.374	126.400
10	9511.433	126.056
11	9503.499	125.951
12	9102.186	120.632
13	6056.791	80.271
14	5863.001	77.703
15	5830.957	77.278
16	5798.913	76.853
17	4763.741	63.134
18	2949.749	39.093
19	2715.370	35.987
20	2485.875	32.945
21	1986.294	26.325
22	1924.343	25.503
23	1789.758	23.720



STANDARD 1H OBSERVE

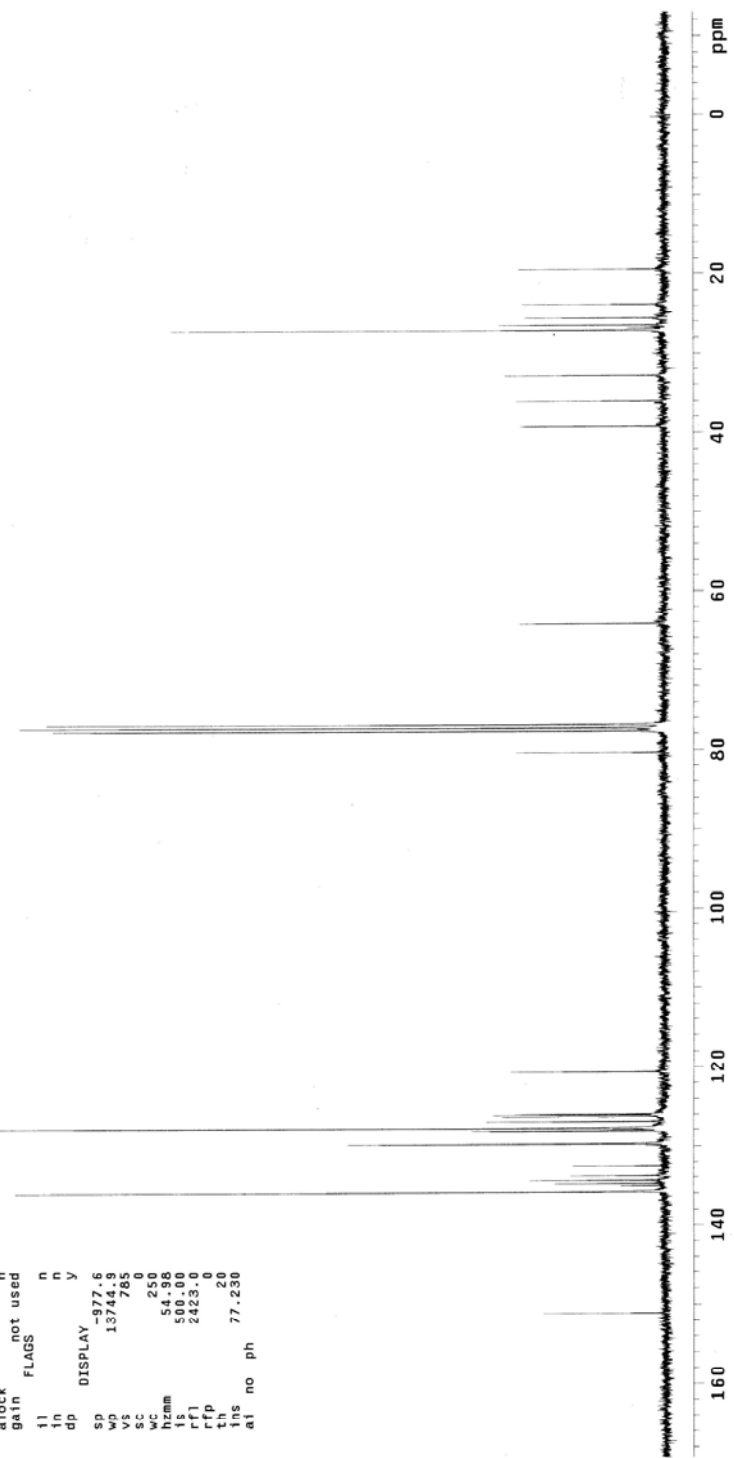
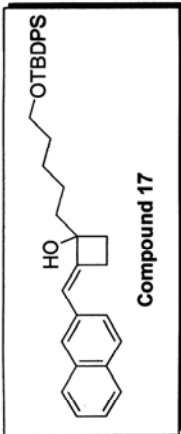
```

exp1 stdih
SAMPLE
date Oct 26 2006 DEC. & VT
file CD013 dfrq 300.078
exp H1 dpr 30
ACQUISITION exp 0
sfrq 300.078 dm nnn
at 1.998 dm C
ap 2000 dmf 200
bs not used wtf 16
proc not used ft
fn not used
tpwr 58
pw 7.2 werr
d1 2.000 wexp
tof 0 wbs
ct 16 wnt
alock n
gain not used
FLAGS
il n
in n
dp n
DISPLAY
sp -139.6
wp 2885.8
vs 512
sc 0
wc 250
hzmm 11.54
rf 50.00
rf1 945.9
rff 0
th 20
ins 1.000
nm cdc ph
  
```



13C OBSERVE

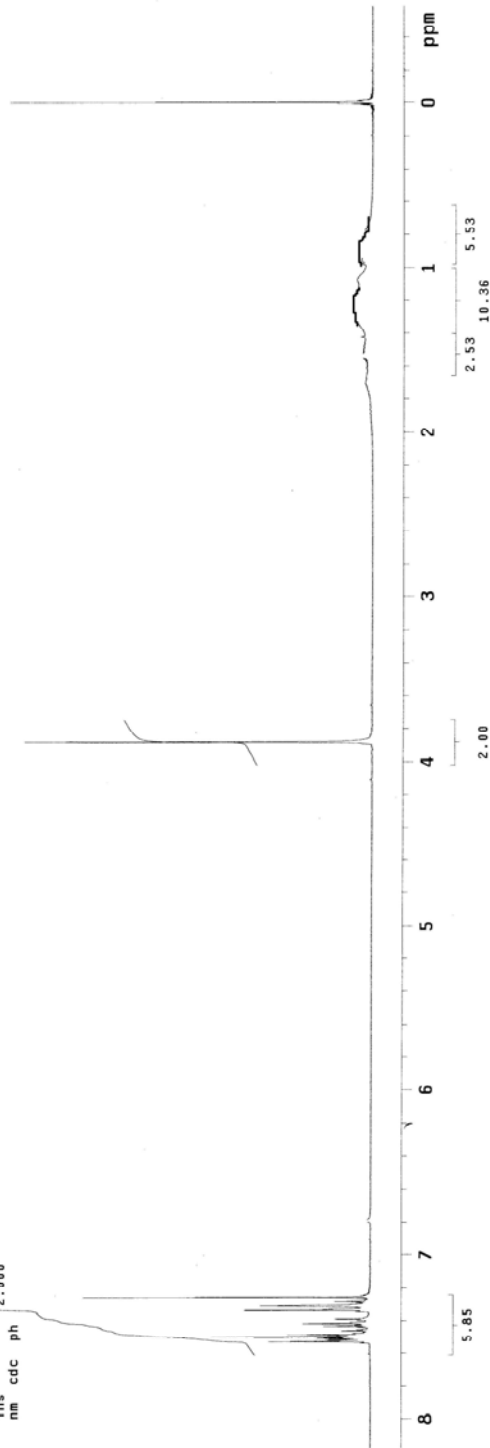
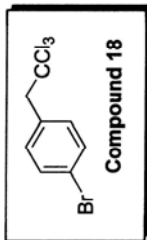
```
exp1 std13c
SAMPLE DEC. & VT
date Nov 1.2006 dfrq 300.078
solvent CDCl3 dn 31
f1 100.625 dpr 30
ACQUISITION exp 0
sfrq 75.462 dm nvy
tn 1.000 dmm
at 40000 dmf 10500 w
sv 20000.0 lb PROCESSING
bc not used v file 1.00
pc 2 proc
tpr 56 fn not used
dvr 4.4
d1 1.000 werr
d2 200 wbs
nt 1102 wnt
clock
gain not used
FLAG n
ll n
ln n
dp y
SP -977.6
W0 13744.9
V5 785
SC 0
WC 250
hzmm 54.86
rf1 2423.0
rff 0
th 20
ins 77.230
at no ph
```



STANDARD 1H OBSERVE

```

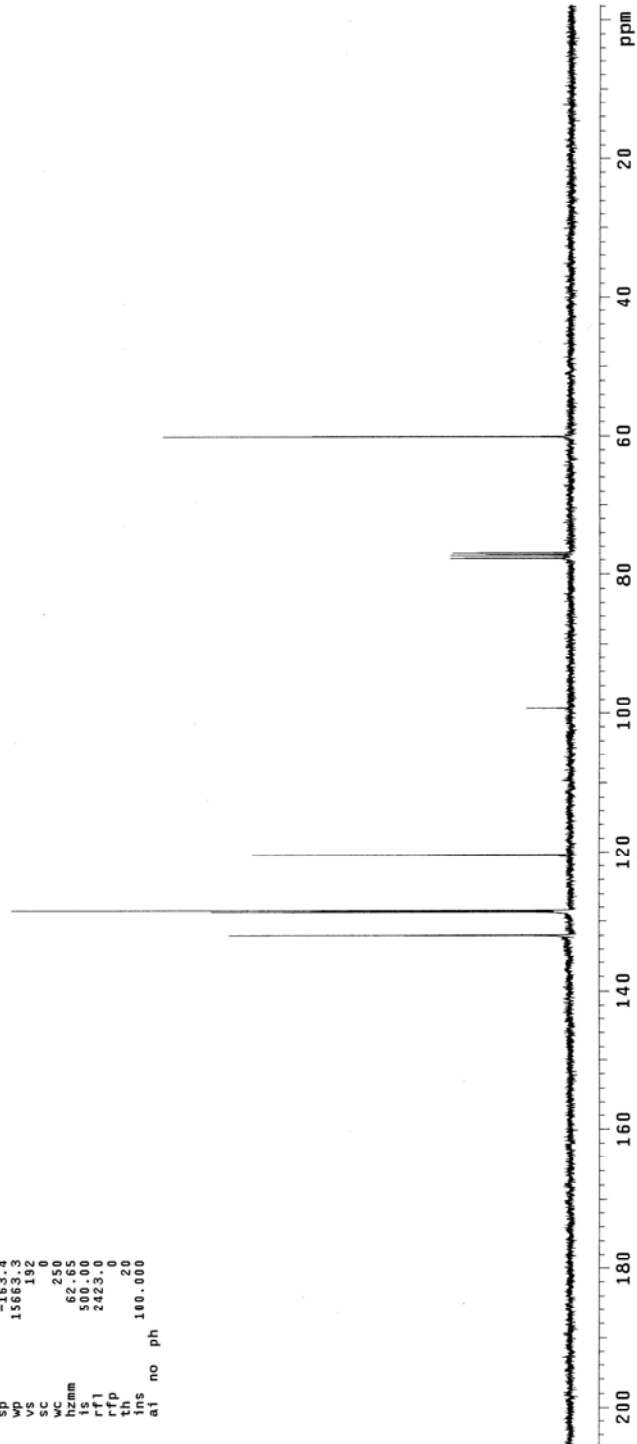
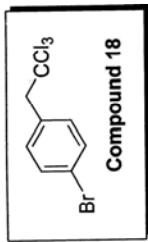
exp1 stdih
SAMPLE
date Oct 16 2006 dfrq DEC. & VT 300.078
solvent CDCl3 dmvr 31
fil ACQUISITION exp 31
sfrq 300.078 dm nnn
tn H1 dmm
at 1.998 dmf
np 20000 wffle PROCESSING 200
fw 3003.0 wffle
bs not used2 fn not used
tpwr 58
pw 7.2 weff
d1 2.000 wexp
tof 0 wos
rt 16 wnt
in
alock in
gain not used
FLAGS
il n
in n
dp y
SP DISPLAY -175.6
wp 2630.7
vs 62
sc 0
wc 250
hzmm 0.52
rf 3955.0
rf1 945.0
rfp 0
th 20
ins cdc ph
nm cdc ph 2.000
  
```



13C OBSERVE

expt1 std13c

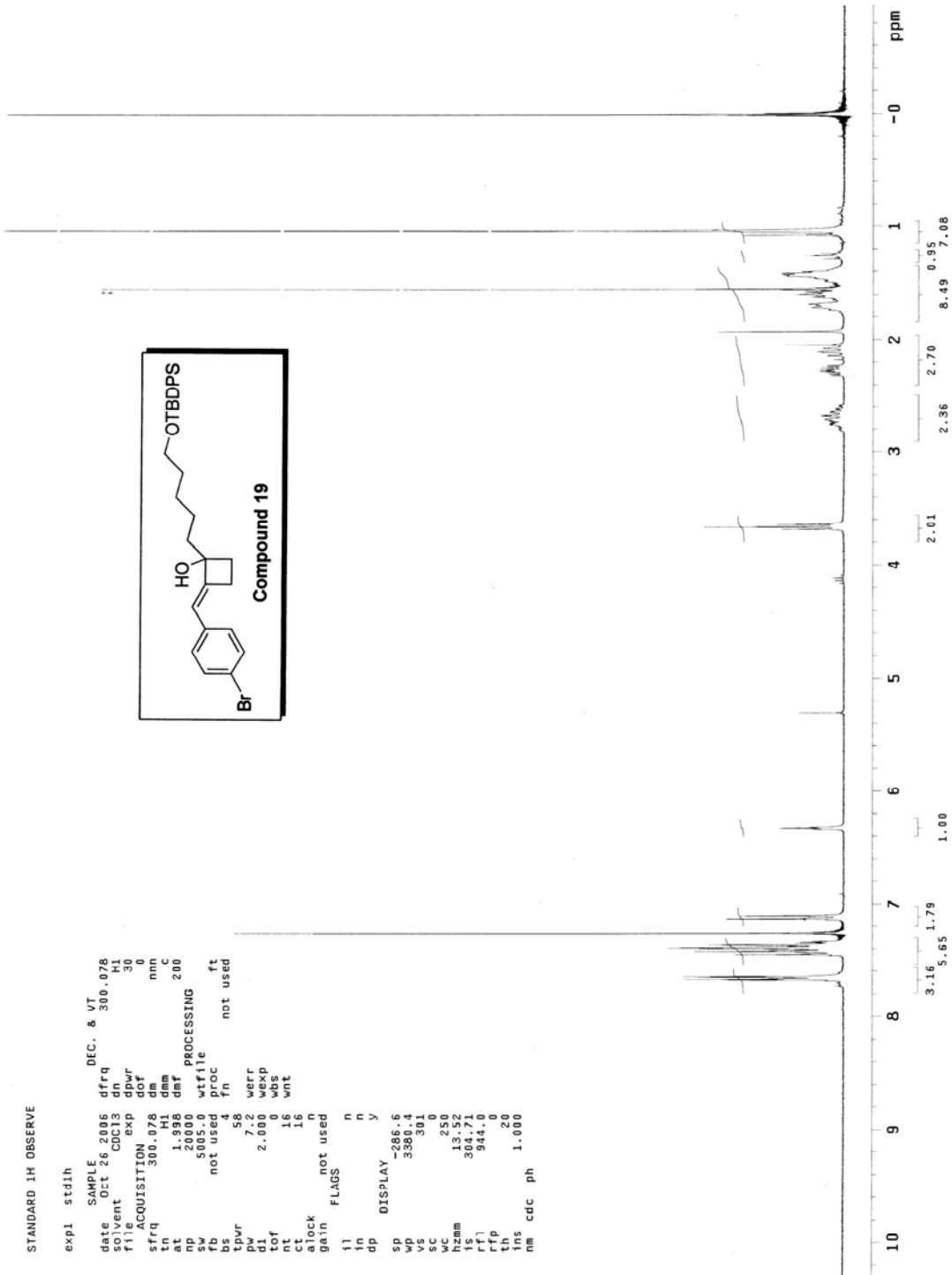
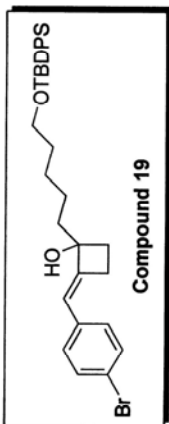
date SAMPLE Mar 7 2008 dfrq DEC. & VT
solvent Mar CCl3 dn 300.078
file CCl3 exp HI
ACQUISITION dpr 0
de 0
sfrq 75.462 dm nyv
tn C13 dm nyw
at 1.000 dmf 10400
np 40000 lb PROCESSING 1.00
sw 28000.0 lb
bd not used wfile
bs 58 frc
tpwr 4.4 ft
pw 4.4 not used
d1 0 werr
d2 1.000 wexp
tof 0 wbs
nt 1000 wnt
l0 10
alock
gain not used
flags
il n
in n
dp y
SD DISPLAY -163.4
WD 15663.3
VS 192
SC 0
WC 250
hzmm 62.65
rs 500.00
ls 1
rfp 2423.0
th 20
ins 180.000
at no ph



STANDARD 1H OBSERVE

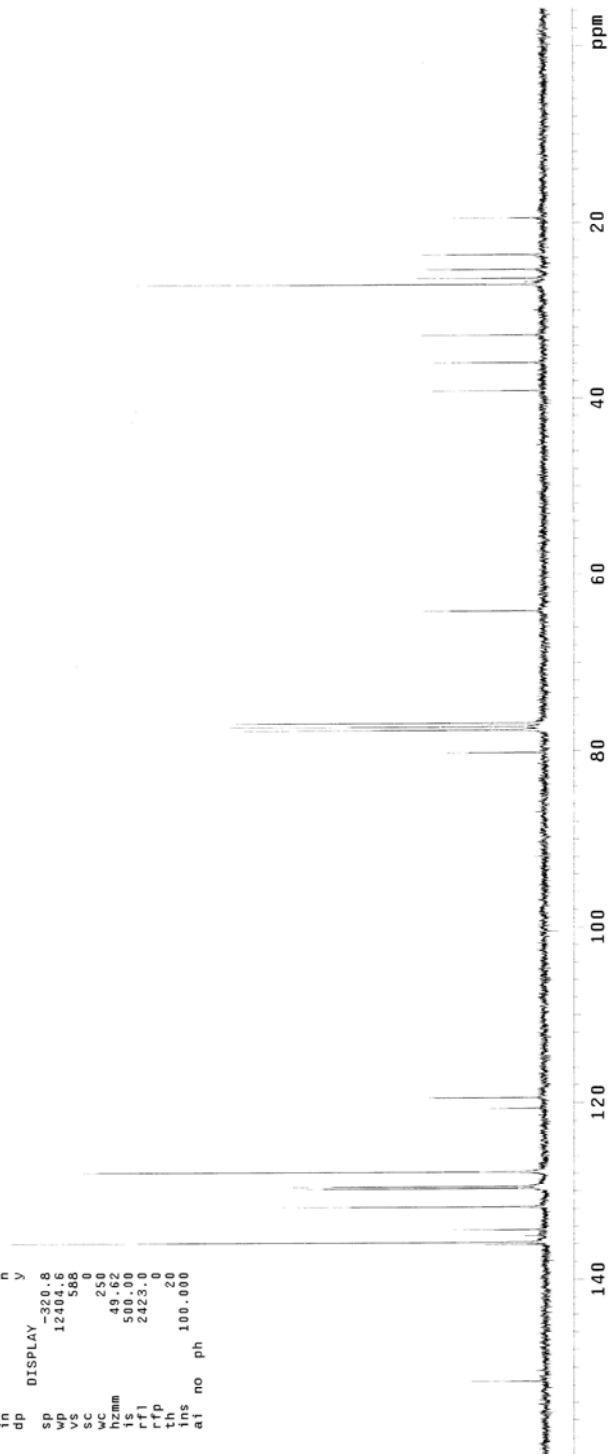
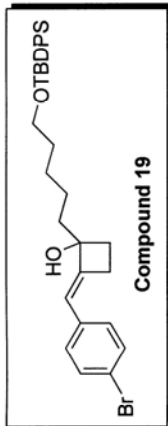
```

exp1 std1h
SAMPLE DEC. & VT 300.078
date Oct 26 2006 dfrq
solvent CDC13 d1
fl1 31
fl2 31
ACQUISITION exp 31
sfrq 300.078 dm nnn
tn H1 dmm
at 1.998 dmf C
np 20000 dmf PROCESSING
pw 3005.0 wrflie
bs not used 4 fnc ft
not used 4 fn not used
tpwr 58
pw 7.2 werr
d1 2.000 wexp
tof 0 wbp
nt 16 wnt
in 16
slck 16
gain not used
FLAGS
il n
in n
dp y
DISPLAY -286.6
sp 3380.4
vs 301
sc 0
wc 250
hzmm 13.52
rf 804.71
rfg 944.0
rfp 0
th 20
ins 1.000
nm cdc ph
  
```



13C OBSERVE

```
exp1 std13c
date SAMPLE DEC. & VT
  Oct 30 2006 dfrq 300.078
  solvent CDCl3 d1 42
  f1 101.625 d1pwr 37
  ACQUISITION Exp 0
  sfrq 75.462 dm nvy
  tn 1.000 dmm 10500 w
  np 40000 lb PROCESSING
  pw 20000.0 vtfile 1.00
  bs not used
  bs2 not used
  tpwr 56 fn
  pw 4.4 werr
  d1 0 wexp
  d2 1.000 wbs
  tor 10000 wnt
  ct 892
  alock gain
  gain not used
  FLAGS
  ll n
  in n
  dp n y
  SP DISPLAY -320.8
  wp 12404.6
  vs 588
  sc 0
  wc 250
  hzmm 49.62
  rf 1
  rfp 2423.0
  th 20
  fns 100.000
  ai no ph
```



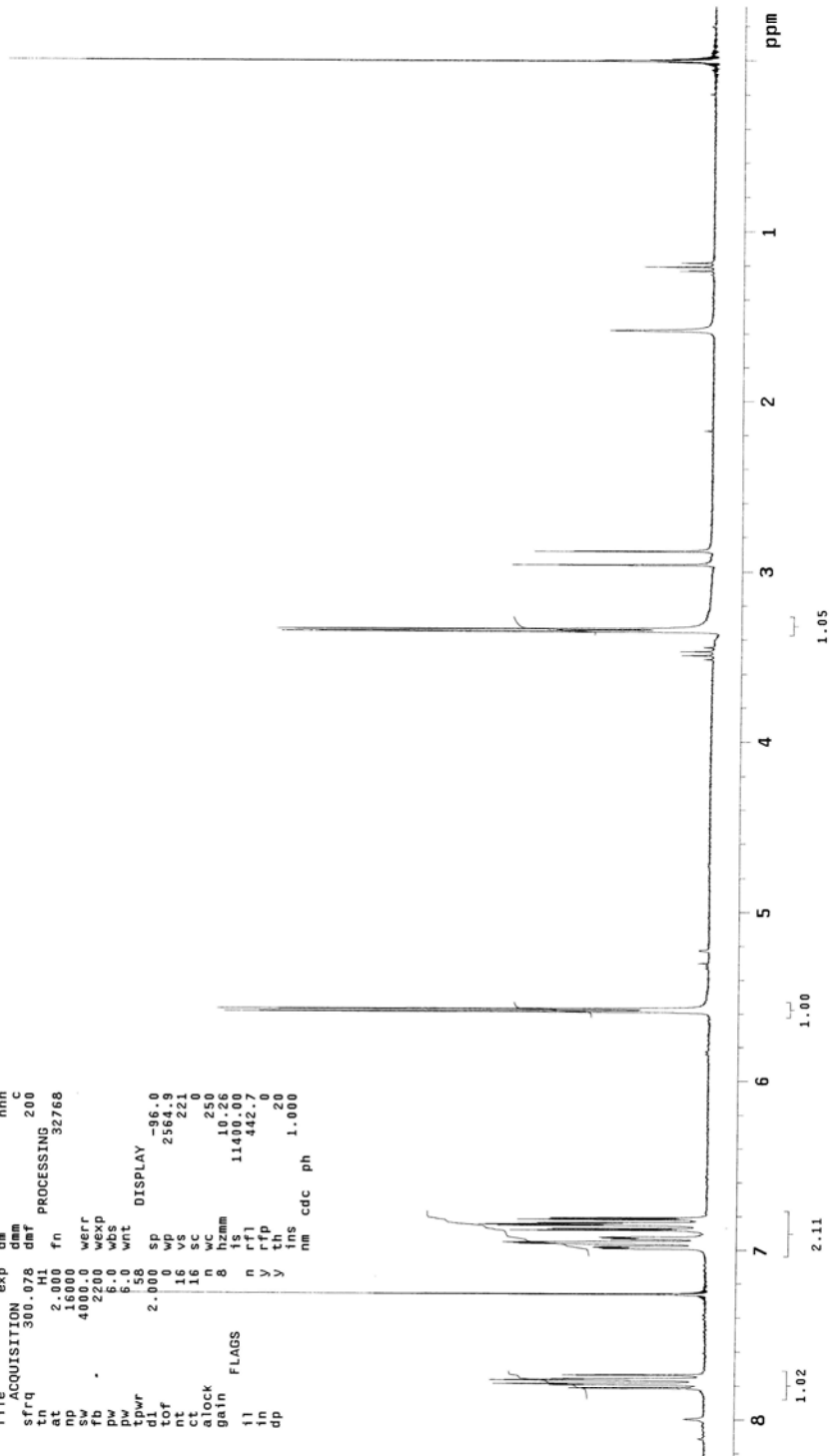
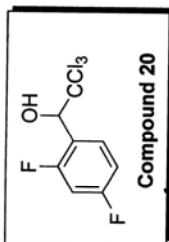
1H SENSITIVITY
0.1-METHYLBENZENE

UTSW 300 Mercury
November 20, 1997

Ken Kezeor

exp1 s2pu1

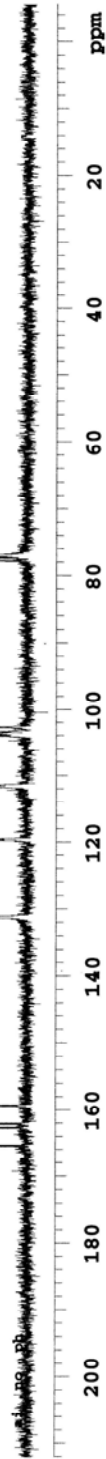
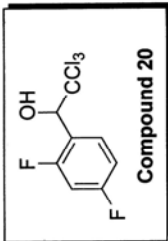
date Nov 6 2001 DEC. & VT H1
solvent cdc13 dof -776.0
file exp nnn
dam c
ACQUISITION
sfrq 300.078 dmf PROCESSING 200
at 4.00 fn 32768
nt 2.000
np 16000
sw 4000.0 weff
fb 2200 wexp
pw 6.0 wbs
pr 5.0 wnt
pwr 2.000 SP DISPLAY -96.0
d1 0.000 WP 2564.9
tof 0.000 VS 221
nt 16 VS 221
ct 16 SC 0
alock n hzmm 250
gain 8 hzmm 10.26
fl n fl 11400.00
in y rfp 442.0
dp y th 20
ins 1.000
nm cdc ph



13C OBSERVE

exp2 std13c

SAMPLE	DEC. & VT
date Aug 22 2009	dfrq 300.078
solvent CDCl3	dn H1
file	exp dpr 37
ACQUISITION	dof 0
sfrq 75.462	dm nvy
tn C13	dmm w
at 1.000	dmf 10400
np 40000	PROCESSING
sw 20000.0	lb 1.00
fb not used	wtfile
bs 10	proc ft
tpwr 56	fn not used
pw 4.7	
d1 .	0 weir
d2 1.000	wexp
tof 0	wbs
nt 3000	wnt
ct 210	
alock	n
gain	not used
FLAGS	
il n	
in n	
dp y	
DISPLAY	
sp -425.8	
wp 16425.7	
vs 398	
sc 0	
wc 250	
hzmm 65.70	
is 500.00	
rf1 2423.0	
rfp 0	
th 13	



1H SENSITIVITY
0.1% ETHYLBENZENE

UTSV INOVA 400

Sensitivity

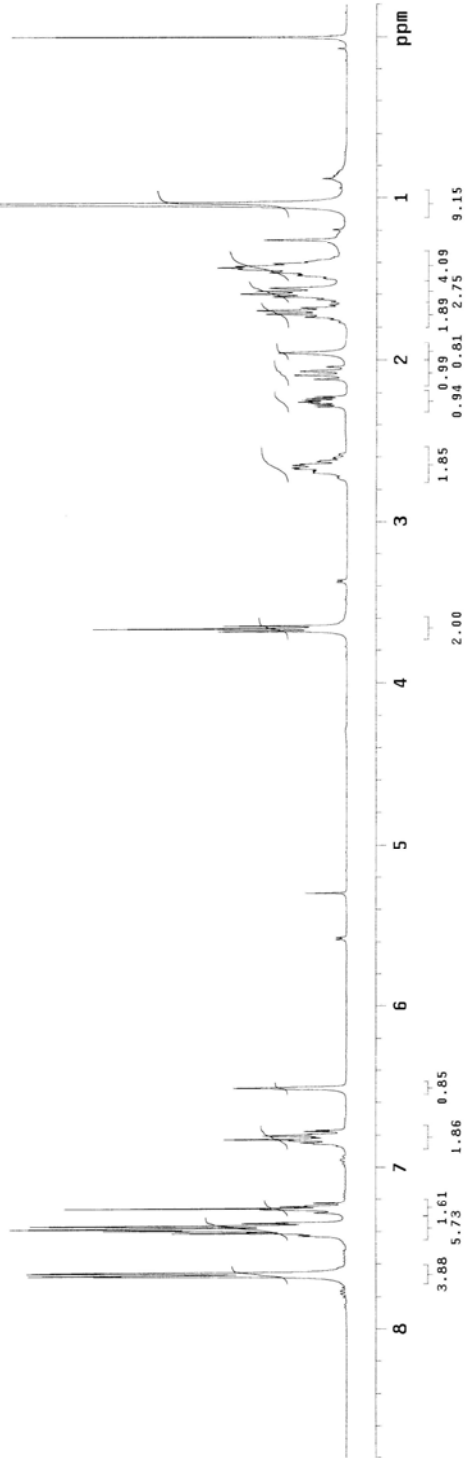
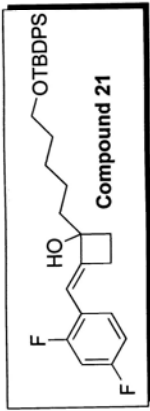
November 19, 1997

Abe Revilla

exp1 s2pu1

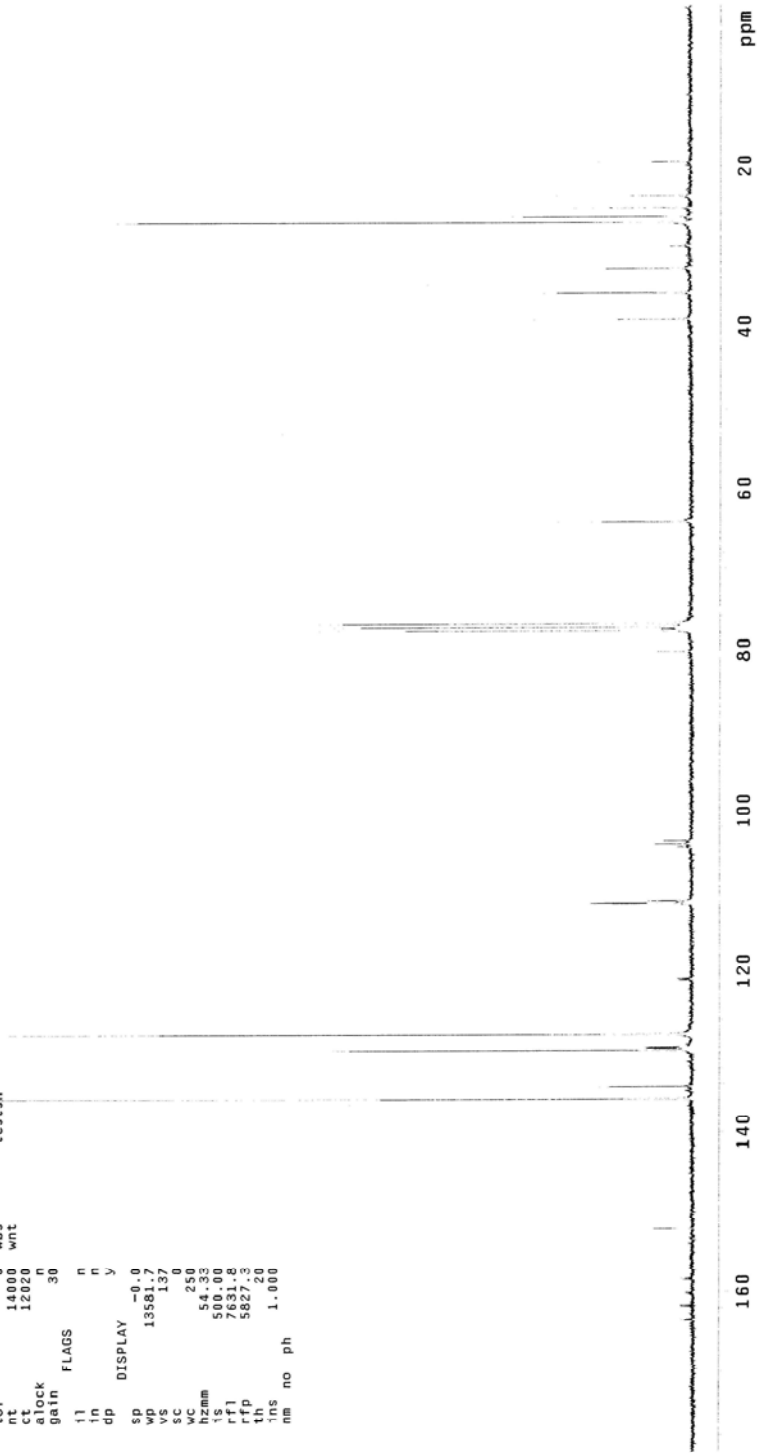
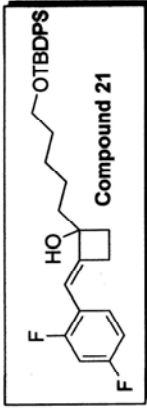
```

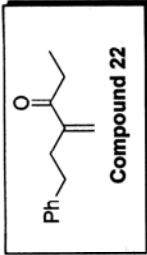
SAMPLE          DEC. & VT
date Nov 3 2003 dn
solvent cdc13 dof -1425.0
file exp dm nnn
ACQUISITION 399.781 dmm dm c
sfrq 2003 lb 0.50
nt 20032 fn 32768
sw 5000.0 math
bs 3000 4
ss 2 weff
pw 3.8
wds 54
tdwr 54
d1 2.000 sp -80.0
tof 0 wp 3597.7
nt 32 vs 390
ct 32 sc 0
atlock n
gain 26 n mcmm 14.30
11 n rff 2062.82
in n rfp 498.4
dp y th 20
hs nn ins nm cdc ph 2.000
  
```



13C OBSERVE

```
text
expl std13c
SAMPLE
date Nov 3 2003   dfrq DEC. & VT 300.078
chem: C6C13      dm   H1
file ACQUISITION exp 37
sfrq 75.462      dm   nyv 0
in    1.815      dmf  10400
ap    90099      lb   PROCESSING 1.00
pw    18000      wf   wfile
bs    10400     proc  ft
tpwr  56        fn   not used
pj    7.6
d1    2.000     werr react
d2    2.000     wexp procpilot
rt    14000     wnt  testsh
ct    12000
alock n
gain  30
FLAOS
l1    n
n     n
dp    y
SP    DISPLAY  -0.0
wp    13581.7
vs    137
sc    0
hzmm  250
ts    54.23
rf1   500.00
rf1   7631.8
rff   5827.3
th    20
tms   1.000
nm    no ph
```

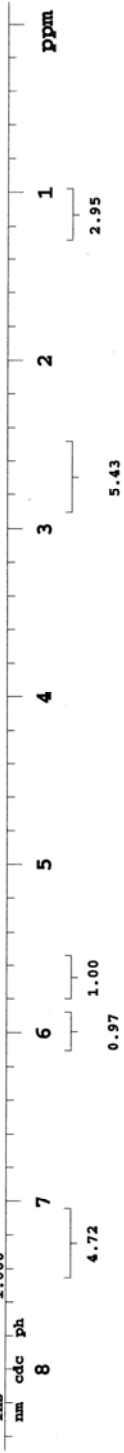


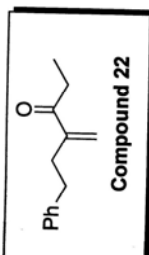


STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Aug 17 2009	dfrq	300.078
solvent	CDCl3	dp	H1
file	exp	dpwr	30
ACQUISITION			
dof	0	nmr	
sfrq	300.078	dm	nmr
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	20	wnt	
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	-39.1		
vp	2584.2		
vs	100		
sc	0		
wc	250		
hzmm	10.34		
is	3434.43		
rfl	945.9		
rfp	0		
th	20		
ins	1.000		
nm	cdc		
ph			

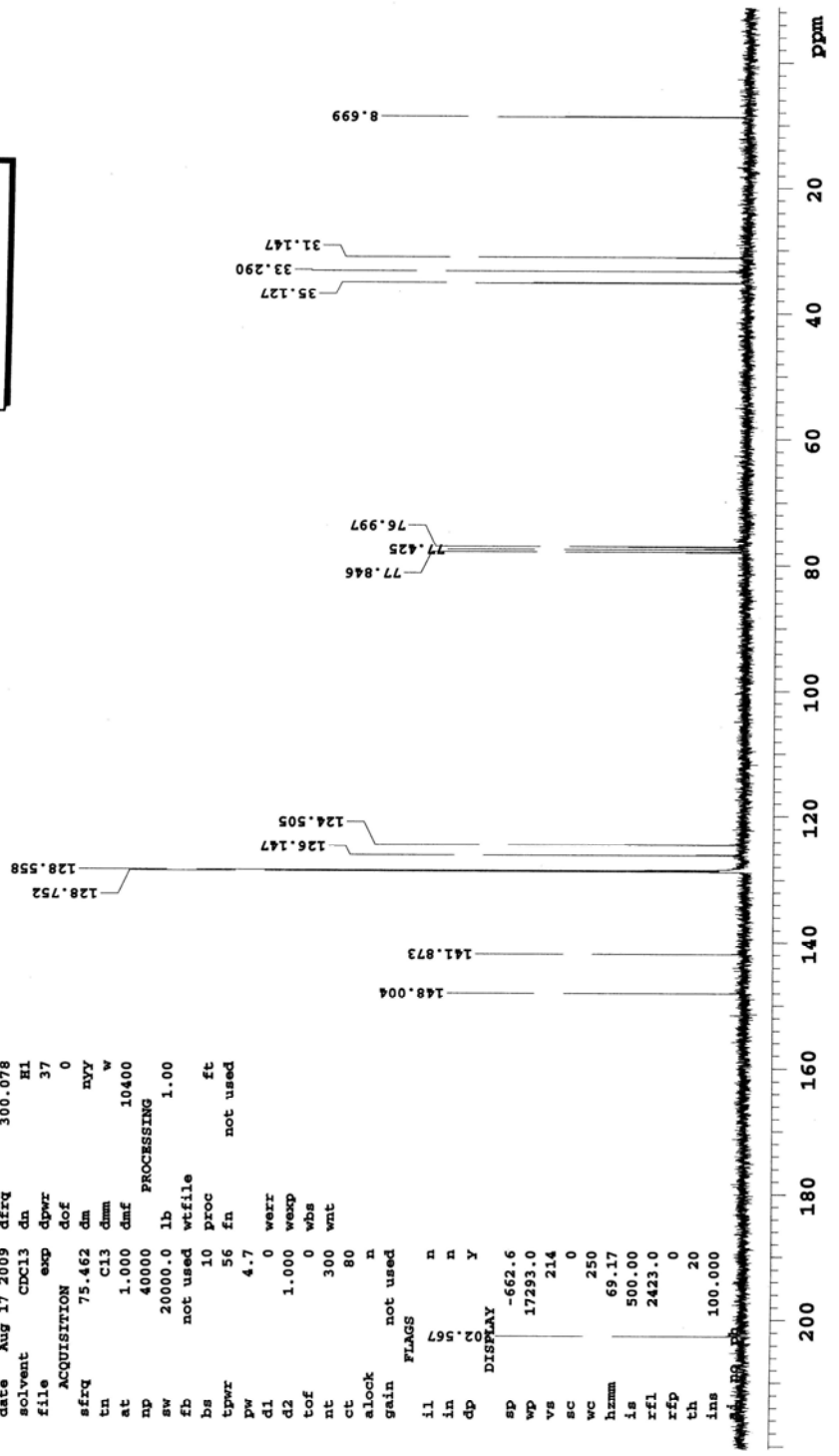


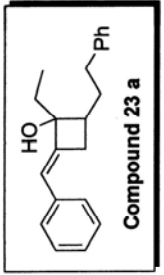


13C OBSERVE

exp1 std13c

SAMPLE DEC. & VT
 date Aug 17 2009 dfrq 300.078
 solvent CDCl3 dn H1
 file exp dpr 37
 ACQUISITION dof 0
 sfrq 75.462 dm nyf w
 tn C13 dmm w
 at 1.000 dmf 10400
 np 40000 PROCESSING
 sw 20000.0 lb 1.00
 fb not used wfile
 bs 10 proc ft
 tpxr 56 fn not used
 pw 4.7
 d1 0 weir
 d2 1.000 wexp
 tof 0 wbs
 nt 300 wnt
 ct 80
 gain not used
 alock n
 il n
 in n
 il 2.567
 dp Y
 DISPLAY
 sp -662.6
 vp 17293.0
 vs 214
 sc 0
 wc 250
 hzmm 69.17
 is 500.00
 rf1 2423.0
 rfp 0
 th 20
 ins 100.000

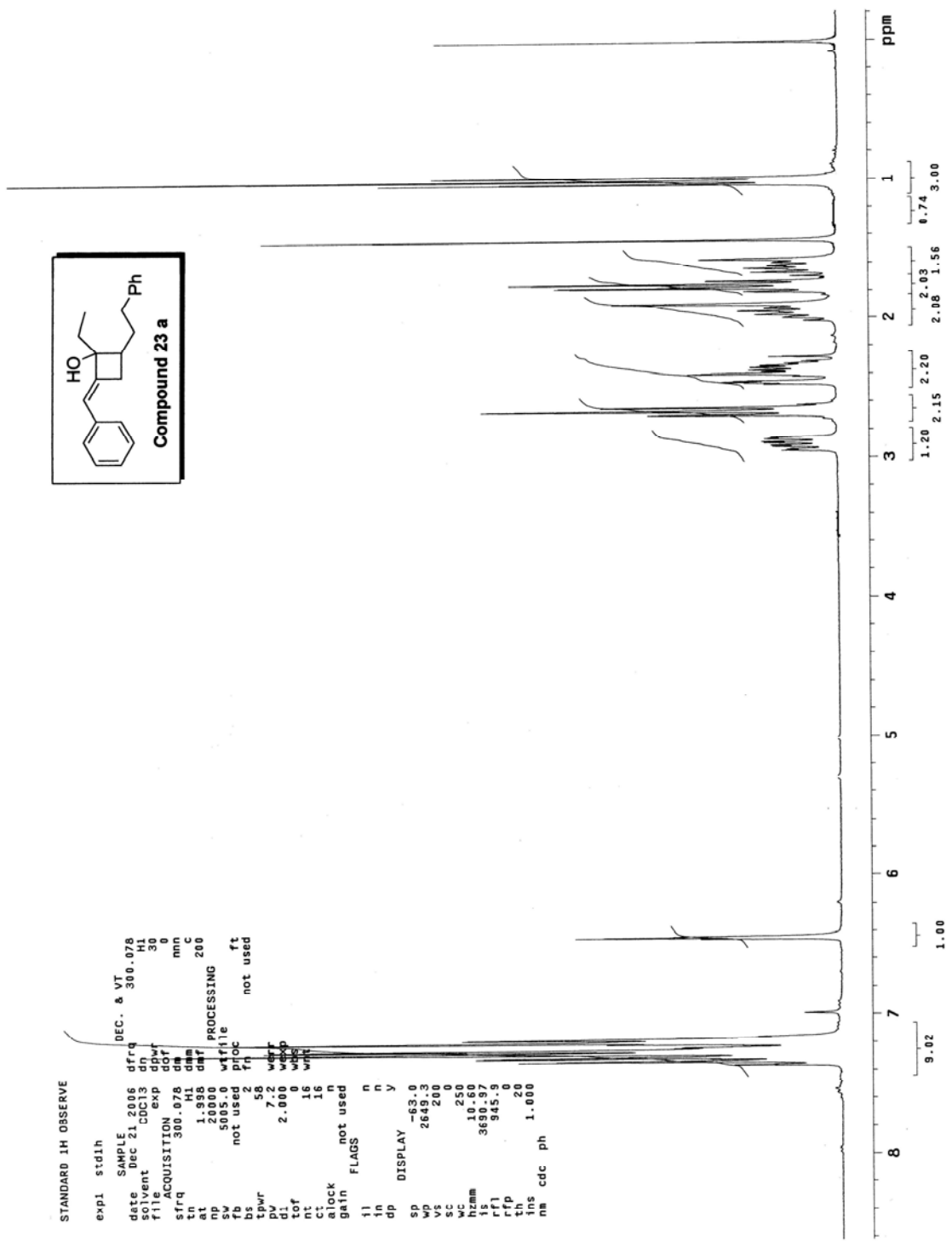


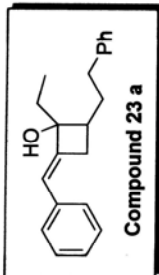


STANDARD 1H OBSERVE

```

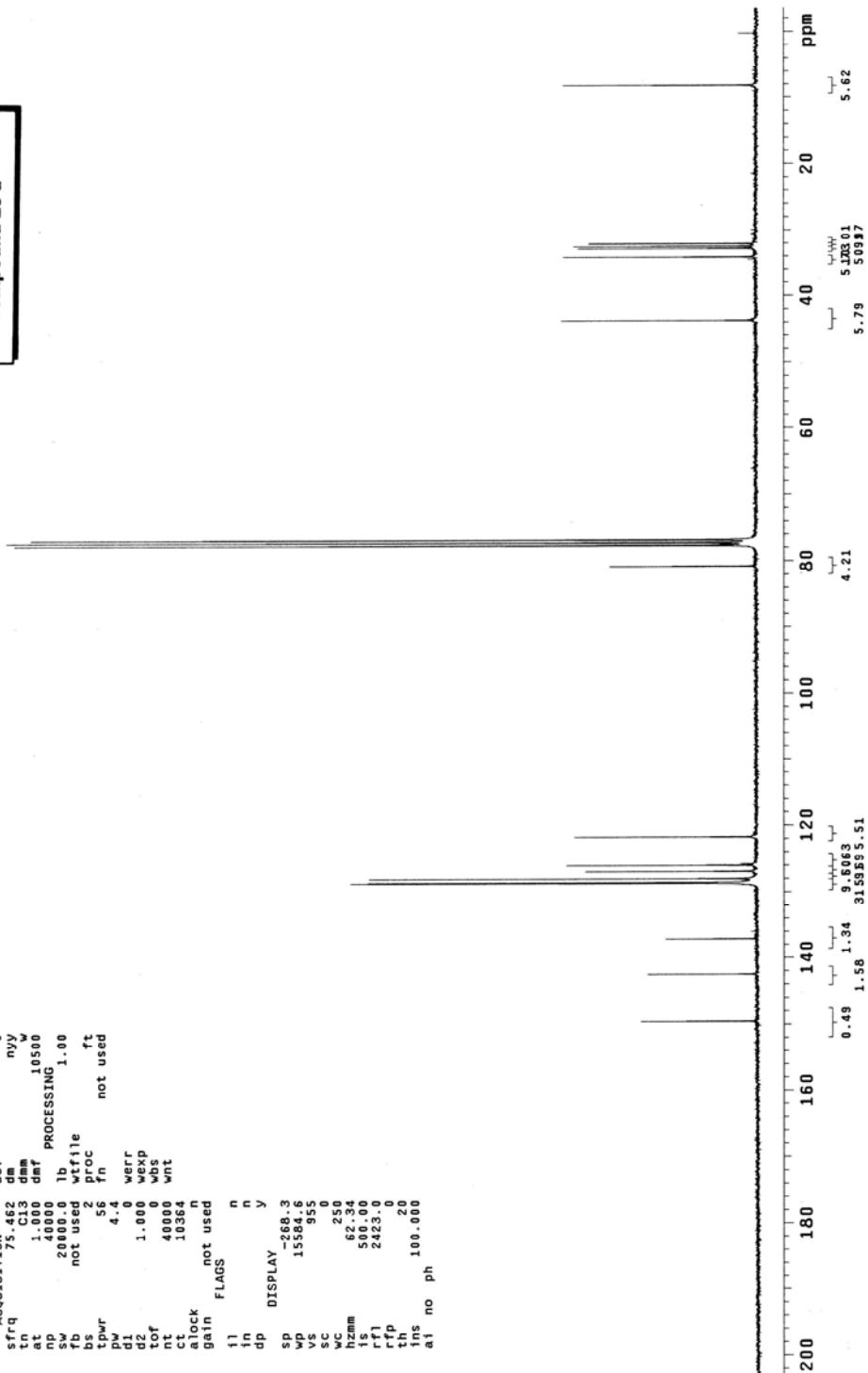
exp1 stdih
SAMPLE
date Dec 21 2006 dfrq DEC. & VT
solvent cdc13 dn 300.078 H1
file exp 30
ACQUISITION
sfrq 300.078 dm 0
tn 1.891 dmp
at 2000.0 wf file PROCESSING 200
sv 5005.0 wf file
fb not used proc ft
bs 2 fn not used
tpwr 58
pv 7.02 wpr
si 2.000 wpcp
rf 16 wpt
ct 16
alock not used n
gain not used n
FLAGS
ll n
hh n
dp Y
DISPLAY
sp -63.0
wp 2649.3
vs 200
sc 250
hzmm 10.60
rf1 3630.97
rfp 845.9
th 0
ins cdc ph 1.000
  
```





```

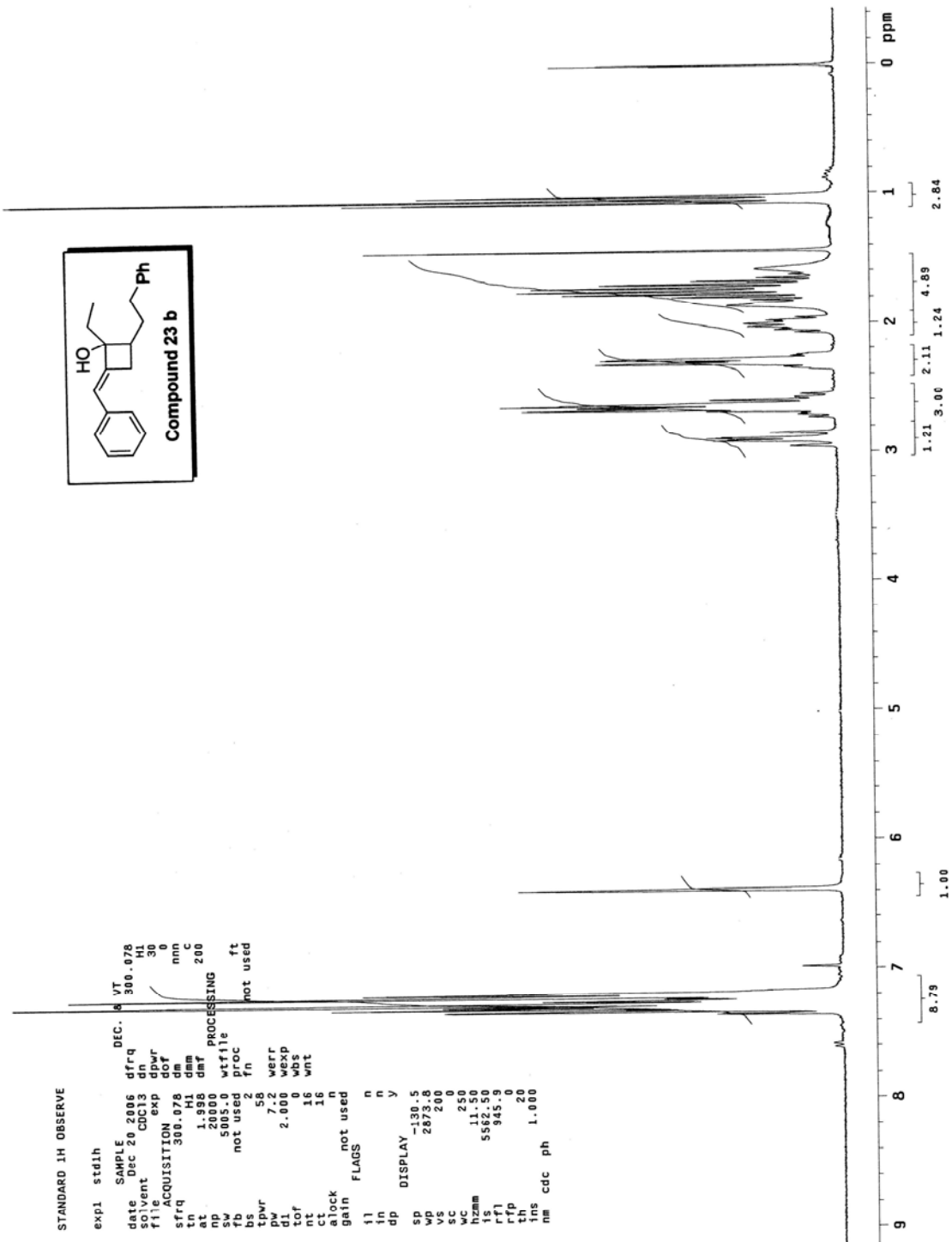
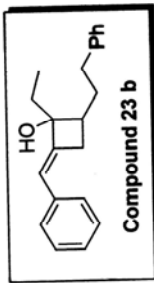
13C OBSERVE
expl std13c
SAMPLE DEC. & VT
date Dec 21 2006 dfrq 300.078
solvent CDCl3 dn 37
F1 ACQUISITION exp dof 0
sfrq 75.462 dm nvy w
tn 1.000 dmm dmf 10500
at 40000 lb PROCESSING
sw 20000.00 wfile 1.00
bs not used 2 proc ft
tdwr 56 fn not used
pv 4.4
d1 4.4 werrf
d2 1.000 wexp
tof 4000 wbs
nt 10384 wnt
clock 10384
gain not used
flags not used
t1 n
in n
dp n y
SP DISPLAY -268.3
WD 15564.6
VS 955
SC 0
WC 250
hzmm 62.34
f1 240.00
rfp 2423.0
th 20
ins 100.000
a1 no ph
  
```



STANDARD 1H OBSERVE

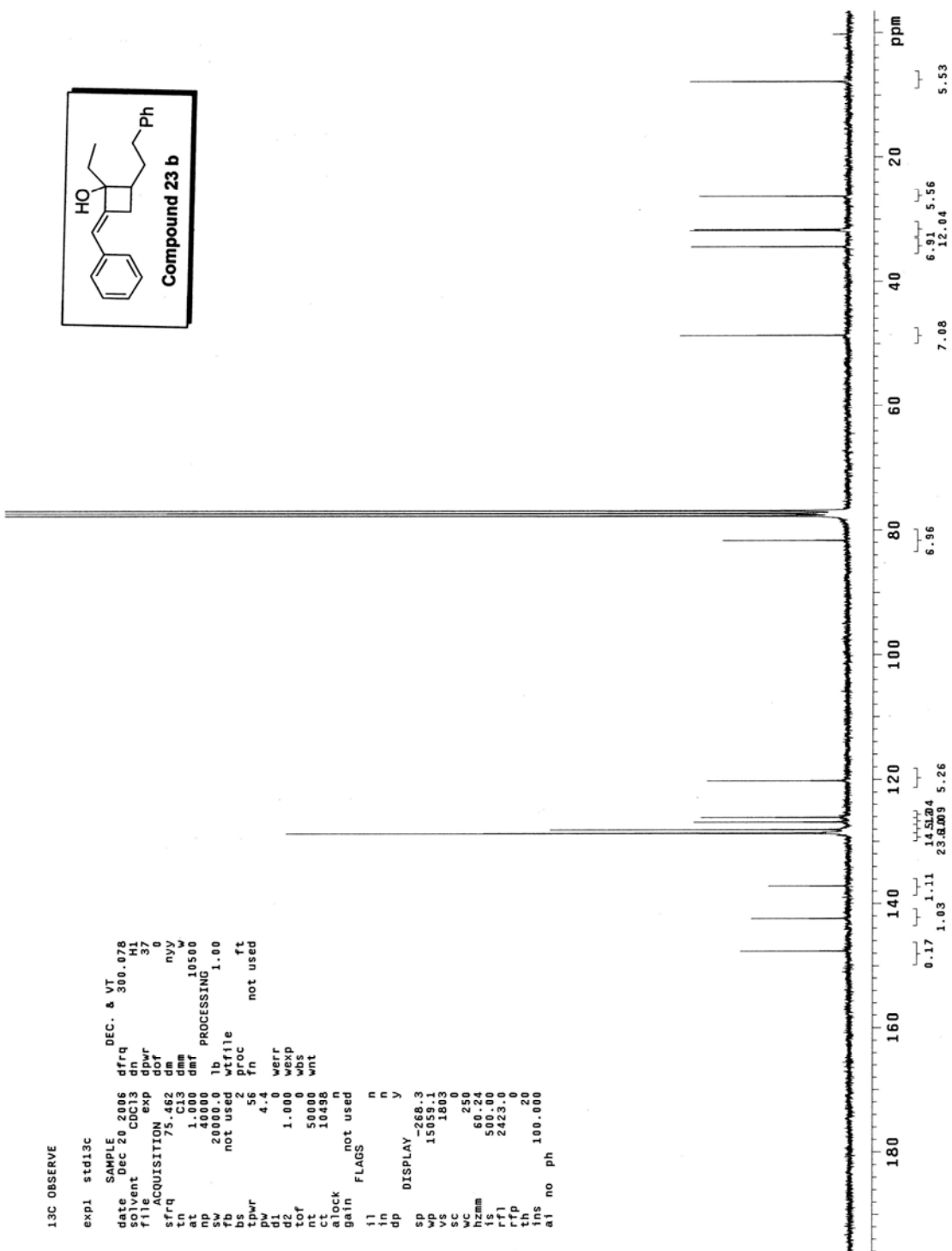
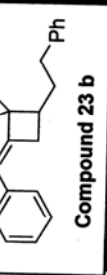
```

exp1 std1h
SAMPLE
date Dec 20 2006 dfrq DEC 300.078
solvent CDCl3 dm H1 30
f1 ACQUISITION exp dof 0
sfrq 300.078 dm mnn c
tn 1.998 H1 dm c
at 20000 dmf 200
np 5005.0 wfile PROCESSING
sw not used f1c ft
b not used fn not used
tpwr 58
pw 7.2 wepr
d1 2.000 wexp
tof 0 wss
nt 16 wnt
ci 16
c1 not used
gain not used
flags
ll n
in n
dp y
SP DISPLAY -130.5
wv 2073.8
vs 200
sc 0
wc 250
hzmm 11.50
fs 5582.50
rfi 345.0
rfp 0
th 20
tms 1.000
nm cdc ph
  
```



13C OBSERVE

```
exp1 std13c
SAMPLE
date Dec 20 2016
file ent CD016
file ACQUISITION exp 37
sfreq 75.462 dm nyv 0
tn 1.000 C13 dmw
np 200000 1.000 dmt 10500
bs not used lb wfile 1.00
tpwr 56 proc ft
pv 4.4 fn not used
d1 1.000 werr
tcf 50000 wds
ct 10498 wnt
alock n
gain not used
FLAGS
il n
in n
dp y
SP DISPLAY -268.3
wp 15059.1
vs 1803
wc 250
lzm 60.74
rfi 2423.0
rfp 0
th 20
ins 100.000
al no ph
```

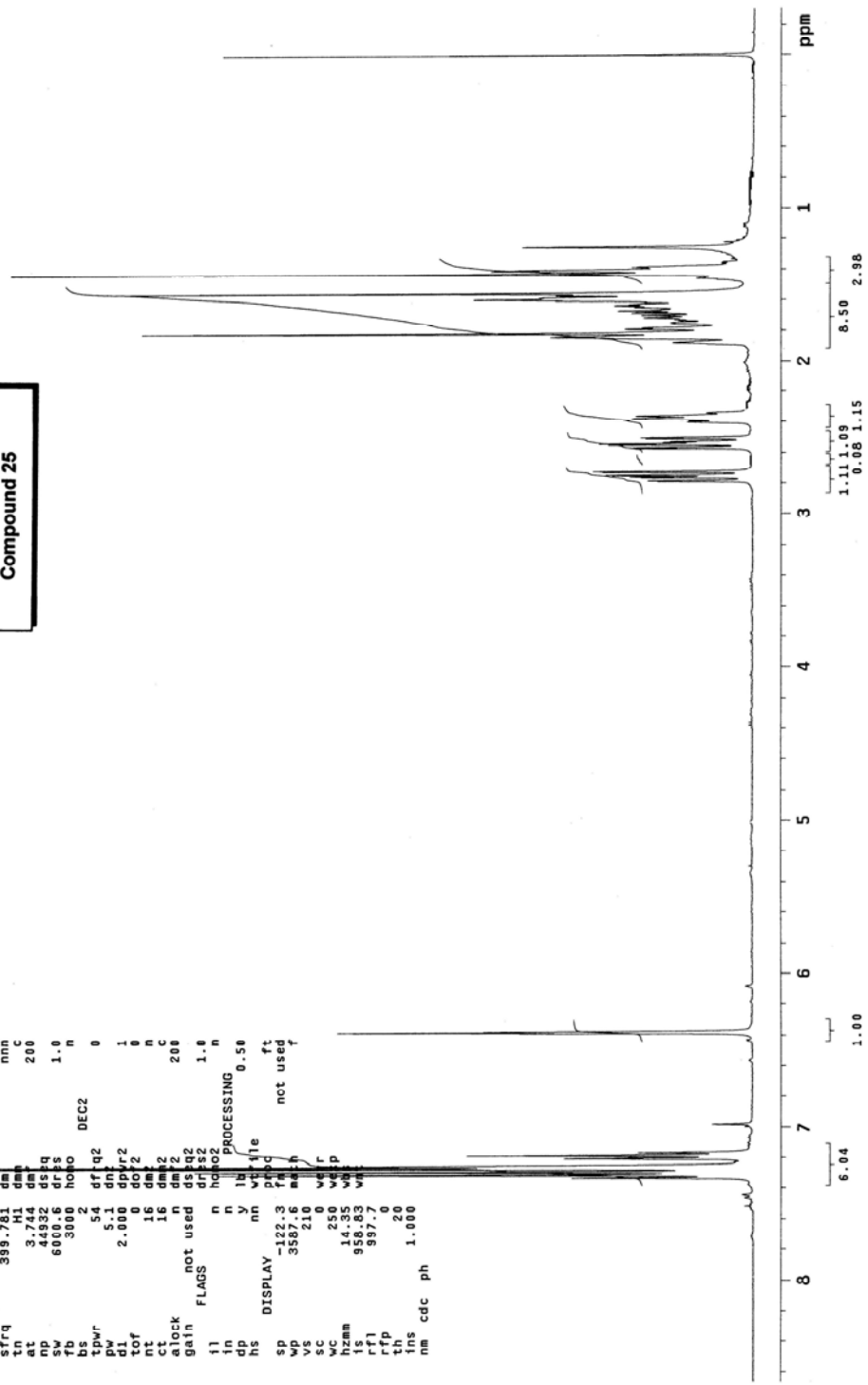
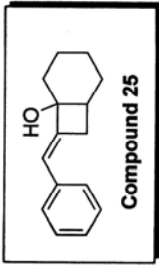


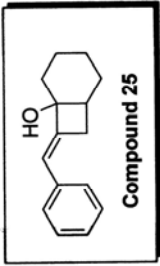
STANDARD 1H OBSERVE

```

expl stdih
date SAMPLE DEC. & VT
solvent Dec 6.2006 dffq 399.761
f1 H1 dnt
f2 H1 dnt
f3 H1 dnt
f4 H1 dnt
f5 H1 dnt
f6 H1 dnt
f7 H1 dnt
f8 H1 dnt
f9 H1 dnt
f10 H1 dnt
f11 H1 dnt
f12 H1 dnt
f13 H1 dnt
f14 H1 dnt
f15 H1 dnt
f16 H1 dnt
f17 H1 dnt
f18 H1 dnt
f19 H1 dnt
f20 H1 dnt
f21 H1 dnt
f22 H1 dnt
f23 H1 dnt
f24 H1 dnt
f25 H1 dnt
f26 H1 dnt
f27 H1 dnt
f28 H1 dnt
f29 H1 dnt
f30 H1 dnt
f31 H1 dnt
f32 H1 dnt
f33 H1 dnt
f34 H1 dnt
f35 H1 dnt
f36 H1 dnt
f37 H1 dnt
f38 H1 dnt
f39 H1 dnt
f40 H1 dnt
f41 H1 dnt
f42 H1 dnt
f43 H1 dnt
f44 H1 dnt
f45 H1 dnt
f46 H1 dnt
f47 H1 dnt
f48 H1 dnt
f49 H1 dnt
f50 H1 dnt
f51 H1 dnt
f52 H1 dnt
f53 H1 dnt
f54 H1 dnt
f55 H1 dnt
f56 H1 dnt
f57 H1 dnt
f58 H1 dnt
f59 H1 dnt
f60 H1 dnt
f61 H1 dnt
f62 H1 dnt
f63 H1 dnt
f64 H1 dnt
f65 H1 dnt
f66 H1 dnt
f67 H1 dnt
f68 H1 dnt
f69 H1 dnt
f70 H1 dnt
f71 H1 dnt
f72 H1 dnt
f73 H1 dnt
f74 H1 dnt
f75 H1 dnt
f76 H1 dnt
f77 H1 dnt
f78 H1 dnt
f79 H1 dnt
f80 H1 dnt
f81 H1 dnt
f82 H1 dnt
f83 H1 dnt
f84 H1 dnt
f85 H1 dnt
f86 H1 dnt
f87 H1 dnt
f88 H1 dnt
f89 H1 dnt
f90 H1 dnt
f91 H1 dnt
f92 H1 dnt
f93 H1 dnt
f94 H1 dnt
f95 H1 dnt
f96 H1 dnt
f97 H1 dnt
f98 H1 dnt
f99 H1 dnt
f100 H1 dnt

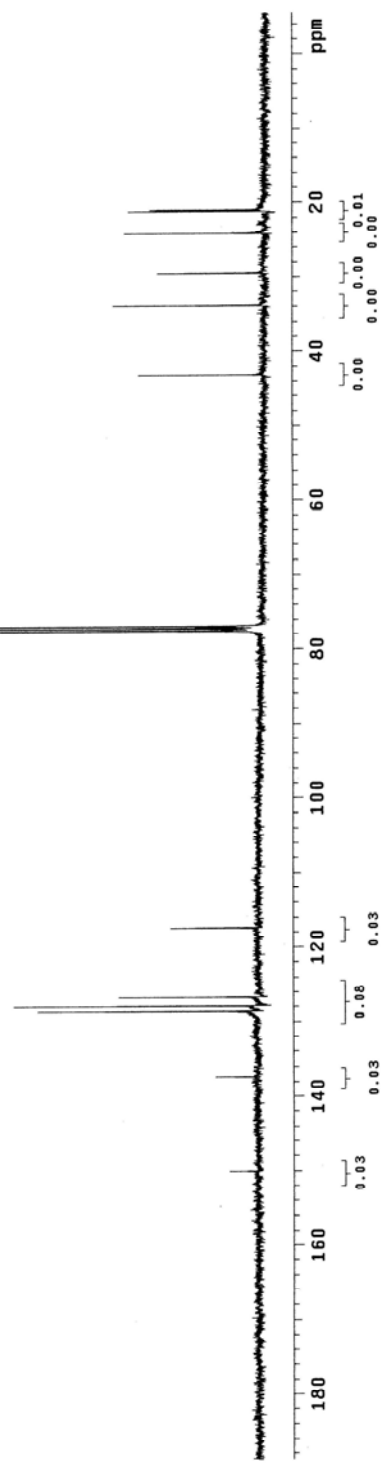
```

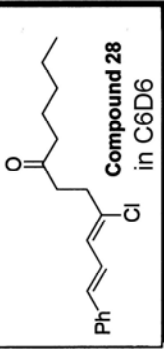




```

13C OBSERVE
expl std13c
SAMPLE
date Dec 7 2006 dfrq DEC. & VT 399.781
solvent CDC13 dn H1
file exp 33
ACQUISITION exp 33
sfrq 100.534 dm nyv
tn 0.13 dmp W
nt 2000 dseq 10000
sv 25000.0 dres 1.0
fb 14000 homo DEC2
bs 4
tpwr 58 dfrq2 0
pw 7.5 dn2 1
d1 2.00 dpr2 1
tof 0 dm2 0
nt 2000 dma2 C
ct 18788 dmf2 9900
alock n dseq2
gain 54 dres2 1.0
FLAGS n homo2
il n lb PROCESSING n
in y wtfile 1.50
dp y v proc
hs DISPLAY mn ft not used
f
sp -561.4 math
wp 19545.0 werr
vs 12 werr
vc 250 wbp
wzmm 78.18 wnt
ls 277888.48
rfl 2987.5
rfp 0
th 50
nms no ph 100.000
  
```

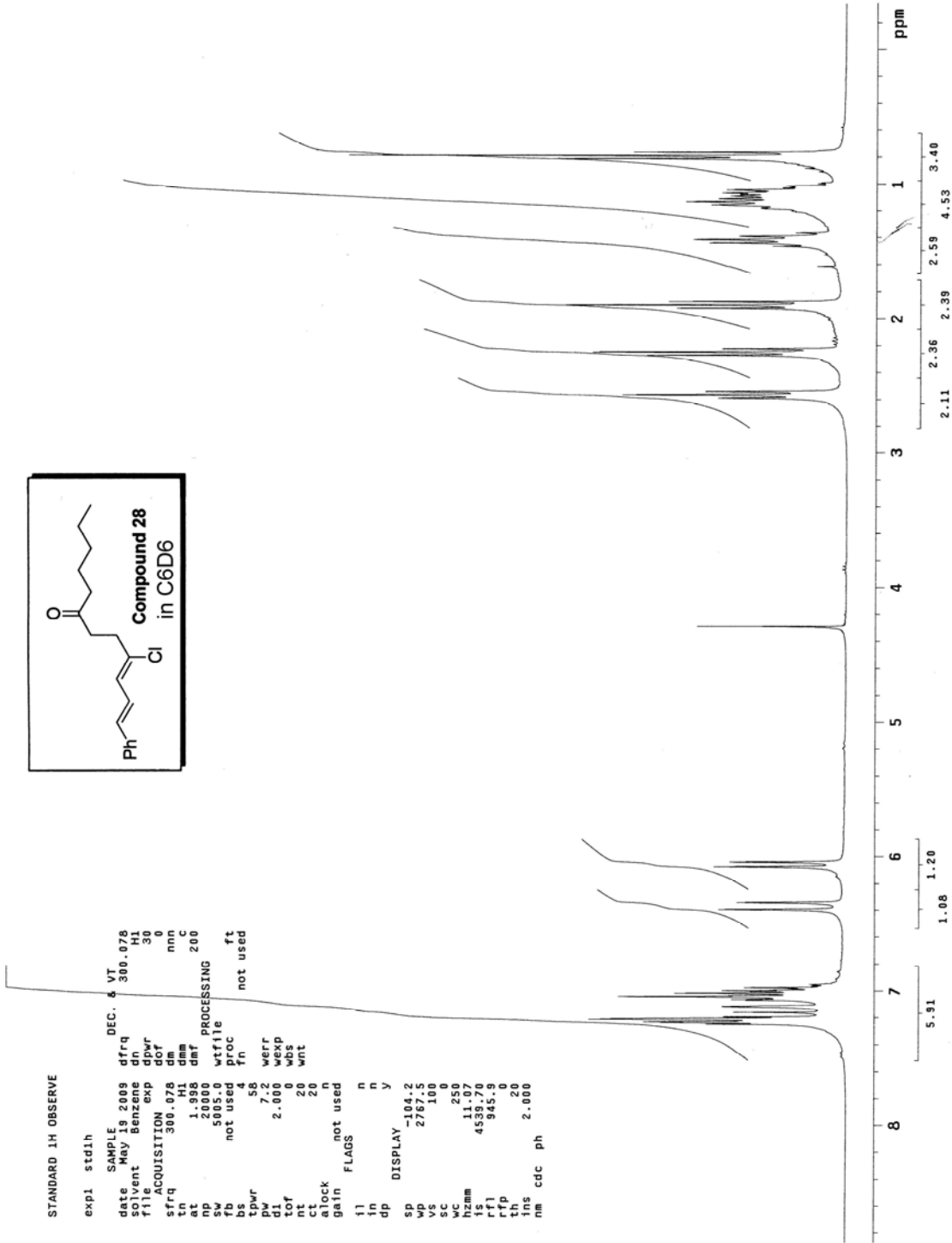




STANDARD 1H OBSERVE

```

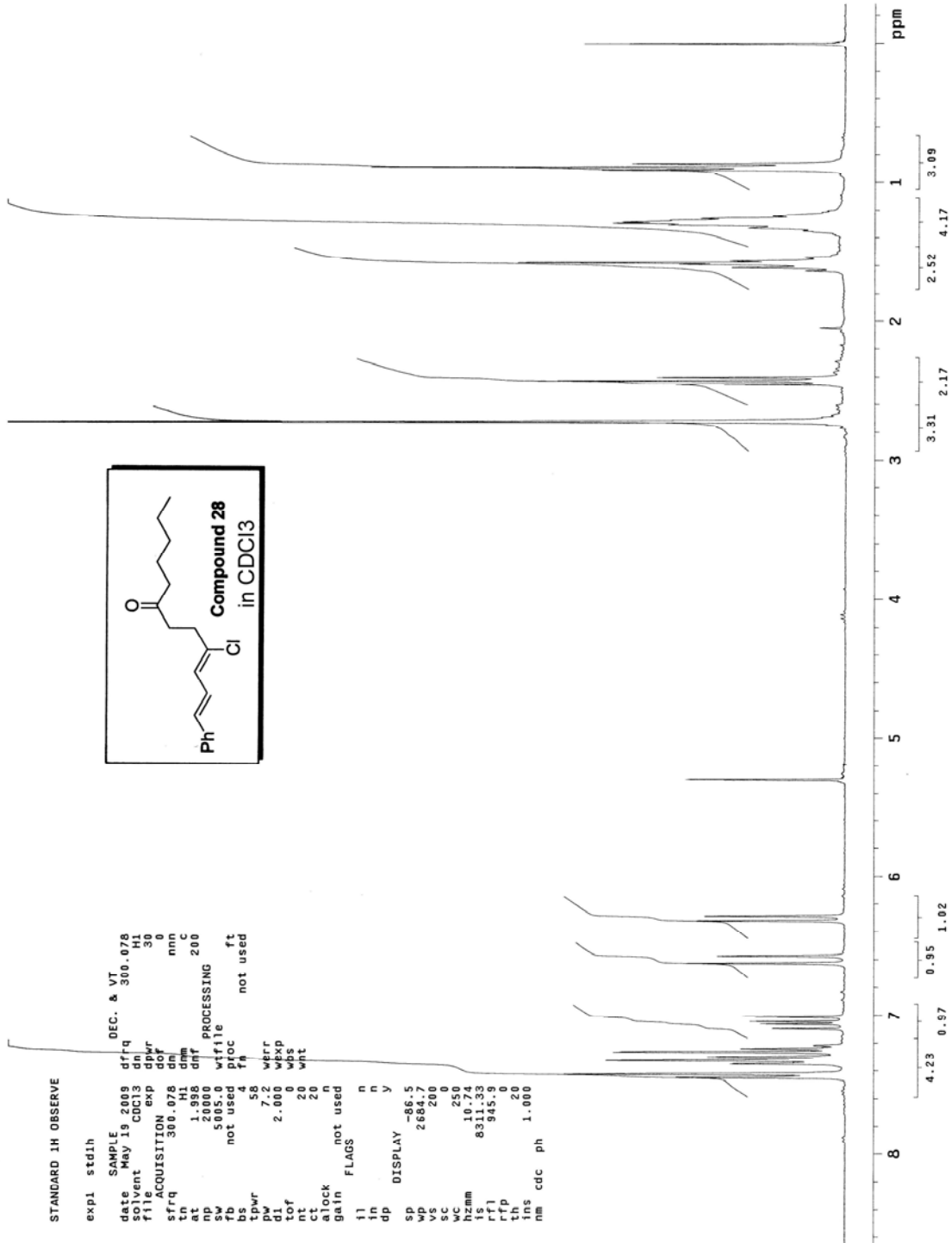
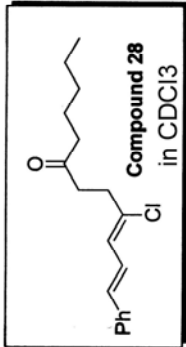
exp1 std1h
SAMPLE DEC. 4 VT
date May 19 2009 dfrq 300.078
solvent Benzene dn H1
file exp dpwr 30
ACQUISITION exp dof 0
sfrq 300.078 dm nnn
at 1.958 dmf PROCESSING 200
np 20000 wf file ft
sw 5005.0 not used proc
bs 4 fn not used
cpwr 58 wscr
di 700 wexp
dt 2.000 wbs
nt 0 wnt
ct 20
alock not used
gain n
in n
in n
dp DISPLAY
sp -104.2
wp 2767.5
vs 100
sc 250
hزام 11.07
is 4539.70
rfl 845.9
rff 0
th 20
ms cdc ph 2.000
  
```



STANDARD 1H OBSERVE

```

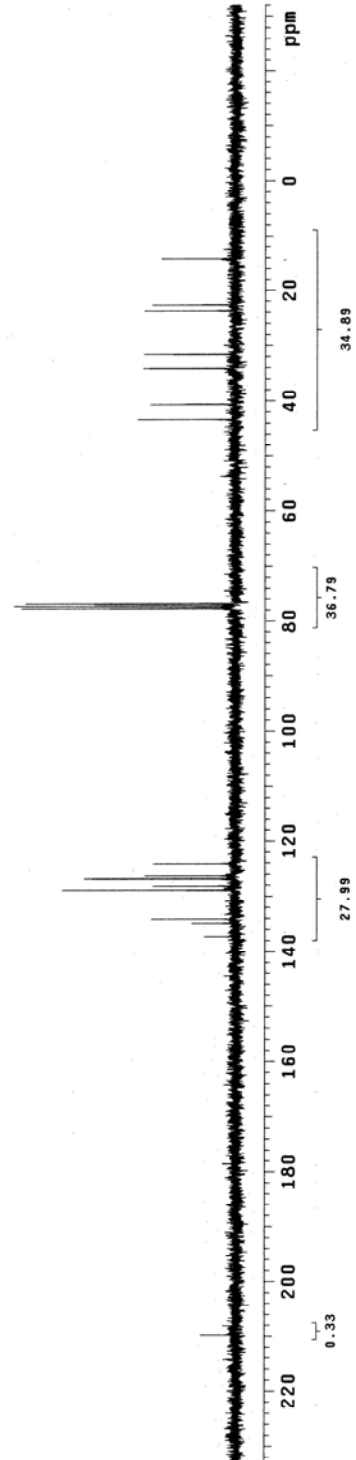
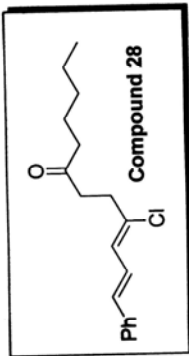
expl stdih
SAMPLE
date May 19 2009 DEC. & VT
file cdc13 dfrq 300.078
file cdc13 dpr 48
ACQUISITION exp dof 30
sfrq 300.078 dm nnn
tn H1 dnm
at 1.998 dnt 200
sv 5000.0 wf116
fb not used proc ft
bs not used fw
tpwr 58
pw 7.2 werr
di 2.000 wexp
nof 20 wps
ct 20 wnt
alock not used
gain n
ll n
ln n
dp n y
SP DISPLAY -86.5
wp 2684.7
vs 200
sc 0
nc 10.250
ts 8311.33
rf1 945.9
rff 0
th 20
ins 1.000
nm cdc ph
  
```

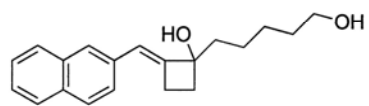


13C OBSERVE

```

expl std13c
date SAMPLE 8 2007 DEC. & VT
solvent Jan CDCl3 dn 300.078
file C0013 dn 37
ACQUISITION exp dpr 0
sfrq 75.462 dm nyv
tn C13 dm
at 1.000 dmt 10500
cp 20000 lb PROCESSING 1.00
fb not used wtf file
bs 4 proc ft
tpwr 56 fn not used
pv 4.4
d1 0 werr
d2 1.000 wexp
ncf 100 wds
ct 100 wnt
alock n
gain not used
FLAGS
ll n
ln n
dp y
DISPLAY
sp -2423.0
wp 20000.0
vs 297
sc 0
wc 250
lzmm 88.00
rfi 2423.0
rfp 0
th 20
ins 100.000
al no ph
  
```





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