Supplementary Information for

Total Synthesis of (±)-Hedychenone: Trimethyldecalin Terpene Systems via

Stepwise Allenoate Diene Cycloaddition

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90095-1569

Experimental Details	
Proton and Carbon NMR's of 6a	
Proton and Carbon NMR's of 6b	
Proton and Carbon NMR's of 7	
Proton and Carbon NMR's of 7a	
Proton and Carbon NMR's of 7b	
Proton and Carbon NMR's of 2	
Proton and Carbon NMR's of 4x	
Proton and Carbon NMR's of 4n	
Proton and Carbon NMR's of 5	
Proton and Carbon NMR's of 9n	
Proton and Carbon NMR's of 9x	
Proton and Carbon NMR's of 9a	
Proton and Carbon NMR's of 9b	
Proton and Carbon NMR's of 1	

Supporting Information Total Synthesis of (±)-Hedychenone: Trimethyldecalin Terpene Systems via Stepwise Allenoate Diene Cycloaddition

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General Experimental. All ¹H NMR and ¹³C NMR spectra were obtained on a Bruker ARX-400 spectrometer operating at 400.132 MHz and at 100.625 MHz, respectively. All ¹H and ¹³C NMR data were reported in parts per million (d) downfield from tetramethylsilane. Coupling constants are reported in hertz (Hz), with the following abbreviations used: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. When appropriate, the multiplicities are preceded with br., indicating that the signal is broad. Thin-layer chromatography (TLC) was carried out using Baker Si250F₂₅₄ silica gel plates and visualization was facilitated by the use of ultraviolet light, anisaldehyde stain, and permanganate stain methods. Flash column chromatography was performed using E. Merck silica gel 60 (230-400 mesh) with compressed air as the source. All solvent mixtures used are indicated as volume/volume. The following solvents were dried and distilled from the indicated drying agent under an argon atmosphere: tetrahydrofuran (THF) and diethyl ether (ether) from sodium benzophenone ketyl radical; dichloromethane, benzene, and triethylamine from calcium hydride; diisopropylamine from sodium hydroxide; methanol from magnesium methoxide. All other solvents and reagents were purified and dried before use as necessary by standard technique. All reactions were performed under an argon atmosphere unless otherwise noted.

2,2,6-Trimethylcyclohexanone, 6a. A 2 M solution of n-butyllithium in pentane (104 mL, 1.06 eq) was added dropwise to a solution of freshly distilled diisopropylamine (33.3 mL, 1.24 eq) in dry THF (250 mL) under Dry Ice/acetone cooling conditions. This solution was cooled in ice and stirred for 40 min. A solution of 2,6-dimethylcyclohexanone (24.7 g, 196 mmol) in dry THF (50 mL) was added to the lithium diisopropylamide (LDA) solution over 30 min under Dry Ice/acetone cooling conditions and reaction mixture was stirred for 1.5 h under the same conditions. Methyl iodide (18.3 mL, 1.5 eq) was added dropwise to the reaction mixture under the same conditions and stirred for 1 h. The mixture was allowed to warm to room temperature and stirred overnight. The reaction mixture was poured into a mixture of saturated ammonium chloride solution (500 mL), water (50 mL) and ether (250 mL) with vigorous stirring.

The layers were separated and the aqueous layer was added to ether (250 mL) and extracted. The organic layers were combined, washed with saturated sodium chloride solution (250 mL), dried over anhydrous sodium sulfate, filtered, and evaporated in vacuo. The residue was distilled (84-85 °C/16 mmHg) to afford 2,2,6-trimethyl-cyclohexanone (25.26 g, 92.0%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 2.64 (1H, m), 2.06 (1H, m), 1.89 (1H, m), 1.78 (1H, m), 1.65 (1H, m), 1.58 (1H, m), 1.33 (1H, m), 1.18 (3H, s), 1.04 (3H, s), 0.99 (3H, d, *J* = 6.5 Hz). ¹³C-NMR (100 MHz, CDCl₃) δ 217.4, 45.2, 41.8, 40.8, 36.8, 25.7, 25.3, 21.6, 15.0.

2,2,6-Trimethylcyclohexanone Hydrazone, 6b. Triethylamine (30 mL, 1.5 eq) and hydrazine monohydrate (124 mL, 17.9 eq) were added to a solution of 2,2,6-trimethyl-cyclohexanone **6a** (20.0 g, 143 mmol) in absolute ethanol (100 mL) and the mixture was refluxed for 3 d. After evaporation of ethanol in vacuo, the residue (*ca.* 130 g) was extracted with ether (120 mL x 3). The organic layers were combined, dried over anhydrous sodium sulfate, and evaporated in vacuo. The crystalline residue (ca. 24 g) was recrystallized from hexane (20 mL) to afford the hydrazone (18.80 g, 85.4%) as colorless needles. ¹H-NMR (400 MHz, CDCl₃) δ 4.98 (2H, br. s), 2.99 (1H, m), 1.78 (1H, m), 1.40-1.69 (5H, m), 1.17 (3H, d, *J* = 7.5 Hz), 1.13 (3H, s), 1.12 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 162.4, 40.4, 37.6, 31.7, 29.5, 28.9, 26.5, 17.4, 17.2.

1,3,3-Trimethyl-2-iodocyclohexene (7). A solution of iodine (13.82 g, 2.1 eq) in dry ether (80 mL) was added dropwise to a solution of 2,2,6-trimethylcyclohexanone hydrazone **6b** (4.0 g, 25.9 mmol) and 1,5-diazabicyclo[4.3.0]-5-nonene (DBN, 20.2 mL, 6.35 eq) in dry ether (80 mL). After the reaction stirred for 3.5 h, saturated sodium bicarbonate solution (80 mL) was added. The layers were separated and the aqueous layer was extracted with ether (160 mL). The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and evaporated in vacuo. Dry benzene (80 mL) and DBN (1.6 mL, 0.5 eq) were added to the residue and the mixture was refluxed for 2 h. After cooling to room temperature, ether (160 mL) was added and the solution was washed with 1 M sodium thiosulfate solution (3 x 80 mL). The organic layer was dried over anhydrous sodium sulfate, evaporated in vacuo, and the residue was chromatographed through silica gel (40 g, pentane) to afford **7a** (5.53 g, 85.2%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 2.12 (2H, t, *J* = 6.2 Hz), 1.87 (3H, s), 1.60-1.75 (4H, m), 1.09 (6H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 137.7, 117.4, 39.6, 37.9, 33.7, 31.6, 31.1, 19.4.

α,2,6,6-Tetramethylcyclohexenemethanol (7a). A 1.6 *M* solution of *t*-butyllithium in pentane (30.5 mL, 2.1 eq) was added dropwise to a solution of **7** (5.82 g, 23.3 mmol) in dry ether (116 mL) under Dry Ice/acetone cooling conditions and stirred for 1 h. Freshly distilled acetaldehyde (13.6mL, 10.5 eq) was added dropwise to the mixture and the mixture was stirred for 2 h under the same conditions. The reaction was quenched by adding of saturated ammonium chloride solution (116 mL), water (23 mL), and ether (116 mL). The layers were separated and the aqueous layer was added to ether (116 mL) and extracted. The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and then evaporated in vacuo. The residue was chromatographed through silica gel (60 g, pentane/ether = 6/1) to afford **7a** (3.53 g, 90.2%) as colorless crystals. ¹H-NMR (400 MHz, CDCl₃) δ 4.51 (1H, q, *J* =6.6 Hz), 1.93 (2H, m), 1.86(3H, s), 1.55 (2H, m), 1.42 (2H, m), 1.41 (3H, d, *J* = 6.6 Hz), 1.09 (3H, s), 0.98 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 141.2, 130.8, 39.9, 34.5, 34.0, 28.6, 27.9, 23.0, 20.9, 19.3.

2-Acetyl-1,1,3-trimethylcyclohexene (**7b**). 4-Methylmorpholine N-oxide (NMO, 4.20 g, 1.5 eq), 4Å molecular sieves (12 g), and tetra-*n*-propylammonium perruthenate (TPAP, 192 mg, 2.3 mol%) were added successively to a solution of **7a** (4.00 g, 23.8 mmol) in dry dichloromethane (48 mL) at room temperature. The reaction mixture was stirred for 2 h under water bath cooling conditions to keep around 25 °C and then filtered through silica gel (10 g). The filtrate was evaporated in vacuo and the residue was chromatographed through silica gel (40 g, dichloromethane) to afford **7b** (3.88 g, 98.2%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 2.28 (3H, s), 1.95 (2H, t, *J* = 6.5 Hz), 1.65 (2H, m), 1.58 (3H, s), 1.43 (2H, m), 1.07 (6H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 210.1, 143.6, 128.5, 38.8, 33.4, 33.2, 31.1, 28.5, 20.8, 18.8.

1-(1,1-Dimethylethyl(dimethyl)silyloxy)ethenyl-2,6,6-trimethylcyclohexene (2). Triethylamine (1.31 mL, 3.0 eq) was added to a solution of **7b** (0.52 g, 3.1 mmol) in dry THF (10 mL) under Dry Ice/acetone cooling conditions. TBSOTf (1.44 mL, 2 eq) was added dropwise under the same conditions. The mixture was allowed to warm to room temperature and stirred for 2 h. The reaction was quenched by adding saturated sodium bicarbonate solution (30 mL) and pentane (40 mL). The layers were separated and the organic layer was washed with water (3 x 30 mL). The aqueous layers were combined and extracted with pentane (40 mL). The organic layers were combined and dried over anhydrous magnesium sulfate, filtered, and evaporated in vacuo to yield a colorless oil as a crude product. The crude product was distilled (113 °C/0.8 mmHg) to afford **2** (871 mg, 99.2%) as a clear oil. ¹H-NMR (400 MHz, CDCl₃) δ 4.25 (1H, s), 3.87 (1H, s), 1.94 (2H, t, J = 6.4 Hz), 1.66 (3H, s), 1.58-1.66 (2H, m), 1.40-1.44 (2H, m), 1.04 (6H, s), 0.91 (9H, s), 0.19 (6H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 156.4, 138.8, 129.6, 92.9, 39.3, 33.2, 31.6, 29.0, 25.7, 21.3, 19.2, 18.1, -4.6.

(±) Ethyl (1R,8aS)-4-[1,1-Dimethylethyl(dimethyl)silyloxy]-5,5,8a-trimethyl-2methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenecarboxylate (4x), (±) Ethvl (1R*,8aR*)-4-[1,1-Dimethylethyl(dimethyl)silyloxy]-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenecarboxylate (4n), and (±) Ethyl 2-[3-[1,1-Dimethylethyl(dimethyl)silyloxy]-3-(2,6,6-trimethyl-1-cyclohexenyl)cyclobutylidene]acetate (5). The diene 2 (1.45 g, 5.17 mmol), the allenoate 3 (1.45 g, 2.5 eq), and hydroquinone (5 mg) were added to a sealed tube. The tube was heated in an oil bath at 110 °C for 14 d. The tube was cooled to room temperature and the reaction mixture was chromatographed through silica gel (75 g, pentane/ether = 100/1-50/1) to afford a 2:1 mixture of 4x and 4n (718 mg, 35.4%) as a colorless oil and the cyclobutane 5 (184 mg, 9.1%) as a colorless oil. Analytical samples of 4x and 4n were prepared by silica gel column chromatographic separation (silica gel 5 g, pentane/ether = 200/0 to 200/1) by use of a small amount of the mixture of 4x and 4n.

Exo [4+2] cycloadduct, **4x**. ¹H-NMR (400 MHz, CDCl₃) δ 4.91 (1H, m), 4.82 (1H, s), 4.14 (2H, q, *J* = 7.2 Hz), 3.07 (1H, m), 3.06 (1H, m), 2.86 (1H, d, *J* = 19.6 Hz), 1.15-1.70 (6H, m), 1.25 (3H, t, *J* = 7.2 Hz), 1.24 (3H, s), 1.20 (3H, s), 1.19 (3H, s), 0.95 (9H, s), 0.20 (3H, s), 0.19 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 172.1, 142.2, 139.9, 126.6, 108.6, 59.8, 59.7, 40.3, 39.4, 38.6, 34.7, 33.7, 29.9, 29.7, 26.4, 22.8, 18.7, 16.9, 14.3, -2.3, -2.5. IR (neat) 2931, 2860, 1736, 1654, 1626, 1473, 1464, 1375, 1321, 1254, 1202, 1161, 1067, 1043, 837, 779 (cm⁻¹).

Endo [4+2] cycloadduct **4n**. ¹H-NMR (400 MHz, CDCl₃) δ 4.92 (1H, m), 4.89 (1H, m), 4.09 (2H, m), 3.22 (1H, m), 2.85 (1H, s), 2.79 (1H, d, *J* = 19.3 Hz), 1.26 (3H, s), 1.22 (3H, s), 1.15-1.70 (6H, m), 0.97 (9H, s), 0.22 (6H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 171.8, 142.7, 140.4, 121.2, 112.4, 62.0, 60.0, 40.3, 38.7, 37.8, 34.0, 33.6, 30.3, 29.81, 29.78, 26.4, 18.7, 17.3, 14.3, -2.1, -2.6.

[2+2] cycloadduct **5**. ¹H-NMR (400 MHz, CDCl₃) δ 5.67 (1H, m), 4.16 (2H, q, J = 7.2 Hz), 3.68 (1H, br. d, J = 17.8 Hz), 3.42 (2H, m), 3.09 (1H, br. d, J = 17.8 Hz), 1.96 (2H, t, J = 6.3 Hz), 1.66 (3H, s), 1.53-1.59 (2H, m), 1.35-1.40 (2H, m), 1.28 (3H, t, J = 7.2 Hz), 1.15 (3H, s), 1.14 (3H, s), 0.84 (9H, s), 0.08 (3H, s), 0.07 (3H, s). ¹³C-NMR (100

MHz, CDCl₃) δ 166.5, 162.3, 140.8, 132.5, 112.6, 78.7, 59.6, 43.5, 34.3, 34.2, 29.0, 28.9, 26.1, 25.6, 21.9, 19.6, 18.8, 18.3, 14.4, -2.7, -2.8.

(\pm) (1*R*,8a*R*)-4-[1,1-Dimethylethyl(dimethyl)silyloxy]-5,5,8a-trimethyl-2-methyl-ene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenemethanol (9n) and (\pm) (1*R*,8a*S*)-4-[1,1-Dimethylethyl(dimethyl)silyloxy]-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8aoctahydro-1-naphthalenemethanol (9x). A solution of 1 *M* diisobutylaluminum hydride (DIBAL) in hexane (6.0 mL, 3.3 eq) was added dropwise to a solution of 2:1 mixture of 4x and 4n (710 mg, 1.8 mmol) in dry dichloromethane (14 mL) under Dry Ice/acetone cooling conditions. Reaction mixture was stirred for 1h and 50% sodium hydroxide solution (1.0 mL) was added dropwise. The mixture was allowed to warm to room temperature. After stirring for 30 min, the solution was filtered through Celite. The filtrate was evaporated in vacuo and the residue was chromatographed through silica gel (80 g, deactivated by triethylamine, pentane/ether = 50/4 then 7/1) to afford 9n (207 mg, 32.7%) as colorless needles and 9x (404 mg, 63.7%).

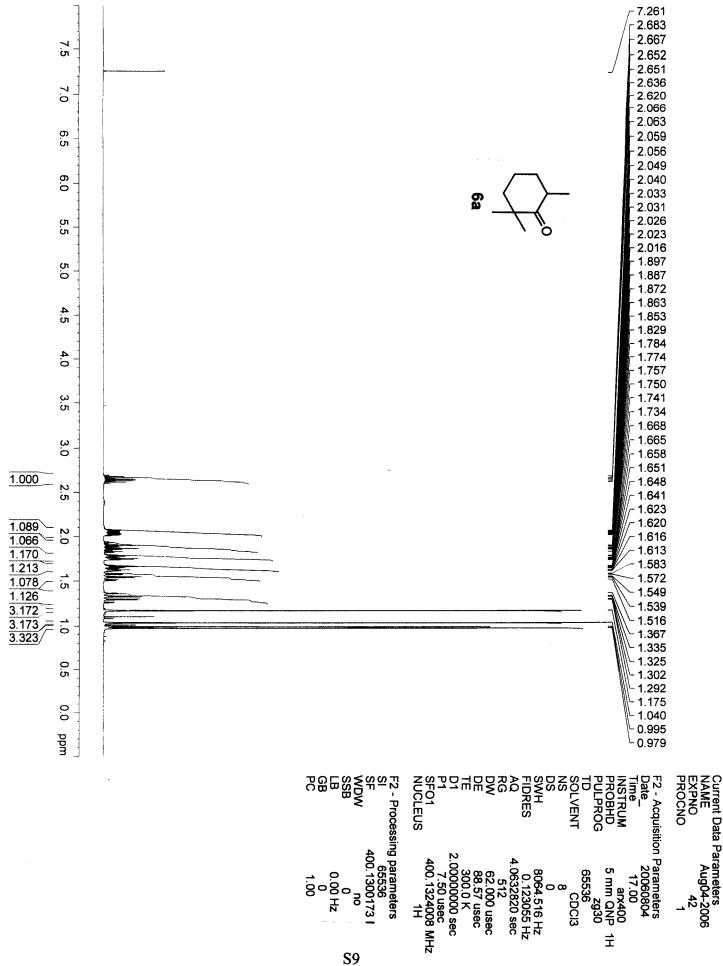
Endo product **9n**: ¹H-NMR (400 MHz, CDCl₃) δ 5.05 (1H, m), 4.89 (1H, m), 3.85 (2H, m), 3.03 (1H, m), 2.81 (1H, d, *J* = 18.1 Hz), 2.21 (1H, m), 1.60-1.78 (4H, m), 1.38-1.49 (2H, m), 1.24 (3H, s), 1.22 (3H, s), 0.97 (9H, s), 0.89 (3H, s), 0.22 (3H, s), 0.20 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 143.4, 142.1, 123.3, 112.3, 60.6, 58.1, 40.6, 37.3, 36.8, 33.6, 33.5, 31.0, 30.1, 29.6, 26.4, 18.6, 17.4, -2.3, -2.5.

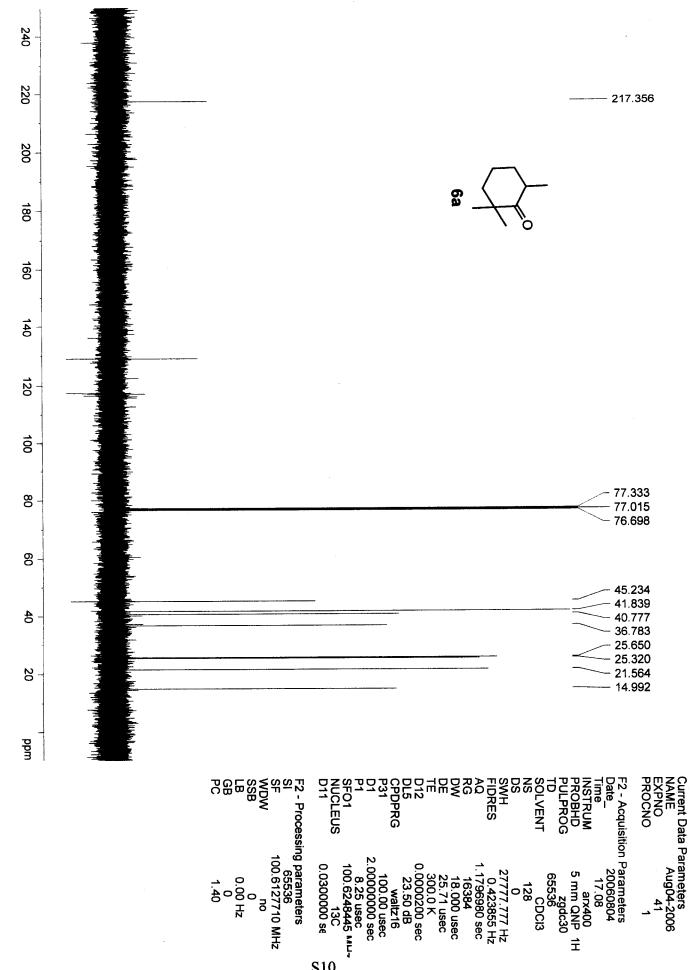
Endo product **9**x: ¹H-NMR (400 MHz, CDCl₃) δ 4.99 (1H, m), 4.89 (1H, m), 3.77 (1H, m), 3.42 (1H, t, *J* = 10.2 Hz), 2.97 (1H, dt, *J* = 19.8, 2.5 Hz), 2.82 (1H, d, *J* = 19.8 Hz), 2.01 (1H, dd, *J* = 10.2, 4.9 Hz), 1.50-1.72 (4H, m), 1.22 (3H, s), 1.20 (3H, s), 1.15-1.35 (2H, m), 0.96 (9H, s), 0.21 (3H, s), 0.20 (3H,s). ¹³C-NMR (100 MHz, CDCl₃) δ 143.3, 142.8, 126.6, 106.9, 59.0, 55.3, 42.4, 38.4, 37.3, 33.9, 33.5, 30.1, 28.6, 26.5, 22.8, 18.7, 16.7, -2.1, -2.6.

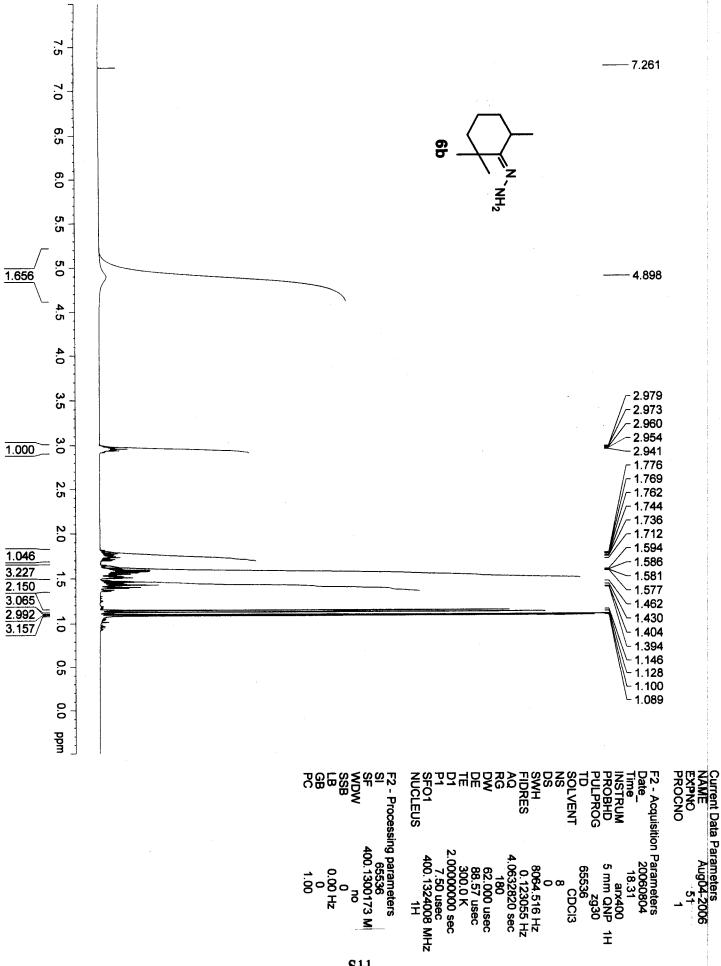
(±) (1R,8aS)-4-[1,1-Dimethylethyl(dimethyl)silyloxy]-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenecarbaldehyde (9a). Dess-Martin periodinane (DMP, 1.27 g, 1.5 eq) was added to a solution of 9x (700 mg, 2.0 mmol) in dry dichloromethane (28 mL) at room temperature, successively. The reaction mixture was stirred for 1 h under water bath cooling conditions to keep the temperature around 25 °C. Ether (28 mL), a 1M solution of sodium thiosulfate (14 mL), and saturated sodium bicarbonate solution (14 mL) were added successively to the reaction mixture. The layers were separated and the organic layer was washed with saturated sodium bicarbonate solution (14 mL) and water (14 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and then evaporated in vacuo. The residue obtained here was chromatographed through silica gel (60 g, pentane/ether = 40/1) to afford **9a** (481 mg, 69.1%) as colorless needles. ¹H-NMR (400 MHz, CDCl₃) δ 9.84 (1H, d, *J* = 5.0 Hz), 5.02 (1H, m), 4.64 (1H, m), 3.05 (1H, m), 2.92 (1H, dd, *J* = 19.7, 0.7 Hz), 2.74 (1H, m), 1.52-1.69 (4H, m), 1.42 (1H, m), 1.28 (3H, s), 1.25 (3H, s), 1.22 (3H, s), 1.20-1.30 (1H, m), 0.97 (9H, s), 0.22 (3H, s), 0.21 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 205.4, 142.8, 140.8, 126.0, 109.8, 64.4, 40.1, 39.5, 38.5, 36.0, 33.8, 29.9, 29.8, 26.4, 23.9, 19.0, 17.0, -2.35, -2.44.

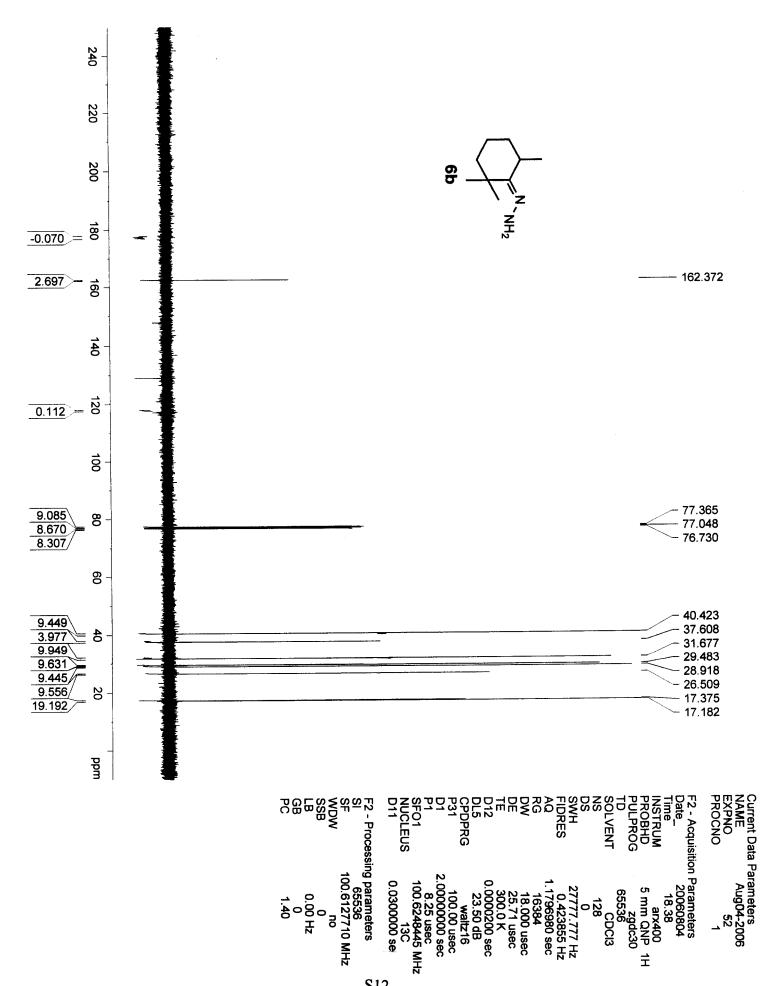
1,2,3,5,6,7,8,8a-octahydronaphthalen-1-yl)methanol, 9b. A solution of 2 M n-butyllithium in pentane (0.10 mL, 2 eq) was added to a slurry of furylmethyltriphenylphosphonium chloride (10, 84 mg, 2.2 eq.) in dry THF (1 mL) under Dry Ice/acetone cooling conditions. The resulting slurry was stirred for 2 h under Dry Ice/acetone cooling conditions. A solution of **9a** (34 mg, 97 mmol) in dry THF (1 mL) was added dropwise to the reaction mixture. The resulting slurry was stirred for 2 h under Dry Ice/acetone cooling conditions and for additional 1 h under ice cooling conditions. Water (2 mL) and ether (4 mL) were added to reaction mixture, successively. The layers were separated and the aqueous layer was extracted with ether (4 mL x 2). The organic layers were combined and dried over anhydrous sodium sulfate, filtered, and then evaporated in vacuo. The crude product was chromatographed through silica gel (5 g, pentane/ether = 100/1 to 50/1) to afford **9b** (15 mg, 37.3%) as a colorless oil and to recover **9a** (4 mg, 11.8%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.34-7.37 (2H, m), 6.56 (1H, dd, J = 1.2, 0.8Hz), 6.23 (1H, d, *J* = 15.7 Hz), 5.95 (1H, dd, *J* = 15.7, 9.8 Hz), 4.86 (1H, d, *J* = 1.6 Hz), 4.70 (1H, dd, J = 4.0, 1.6 Hz), 3.09 (1H, m), 2.91 (1H, d, J = 9.6 Hz), 2.69 (1H, dd, J =9.8, 1.0 Hz), 1.51-1.69 (4H, m), 1.27 (3H, s), 1.23 (3H, s), 1.17-1.32 (2H, m), 1.00 (3H, s), 0.98 (9H, s), 0.20 (3H, s), 0.19 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 146.1, 143.3, 142.4, 139.7, 128.4, 126.7, 124.5, 108.2, 107.6, 57.6, 40.5, 39.4, 38.7, 36.2, 34.0, 29.9, 29.7, 26.5, 26.5, 22.8, 18,7, 17.1, -2.3.

(\pm) (4*S*,4a*R*,8a*S*)-4-((*E*)-2-(Furan-3-yl)ethenyl)-3,4a,8,8-tetramethyl-4a,5,6,7,8,8ahexahydronaphthalen-1(4*H*)-one, (\pm)-Hedychenone (1). The silyl ether 9b (12 mg, 29 mmol) was dissolved in dry THF (0.2 mL) and a solution of 1 *M* tetra *n*-butylammonium fluoride in THF (58 mL, 2 eq) was added under ice cooling conditions. The solution was stirred for 45 min under ice cooling conditions. Saturated sodium bicarbonate (1 mL) and ether (2 mL) were added to reaction mixture, successively. The layers were separated and aqueous layer was extracted with ether (2 mL x 3). The organic layers were combined and dried over anhydrous magnesium sulfate, filtered, and then evaporated in vacuo. The crude product was chromatographed through silica gel (1.5 g, pentane/dichloromethane = 50/50 then 0/100) to afford **1** (7 mg, 80.7%) as colorless needles. ¹H-NMR (400 MHz, CDCl₃) δ 7.43 (1H, m), 7.38 (1H, m), 6.53 (1H, m), 6.36 (*J* = 15.6 Hz), 5.86 (1H, q, *J* = 1.4 Hz), 5.77 (1H, dd, *J* = 15.6, 10.1 Hz), 2.91 (1H, d, *J* = 10.1 Hz), 2.09 (1H, s), 1.79 (3H, t, *J* = 1.4 Hz), 1.35-1.70 (4H, m), 1.10-1.27 (2H, m), 1.19 (3H, s), 1.15 (3H, s), 0.97 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 199.9, 157.2, 143.6, 140.2, 128.1, 126.0, 124.6, 123.8, 107.4, 63.4, 61.3, 43.3, 42.7, 40.2, 33.6, 32.5, 23.0, 21.7, 18.1, 15.7.

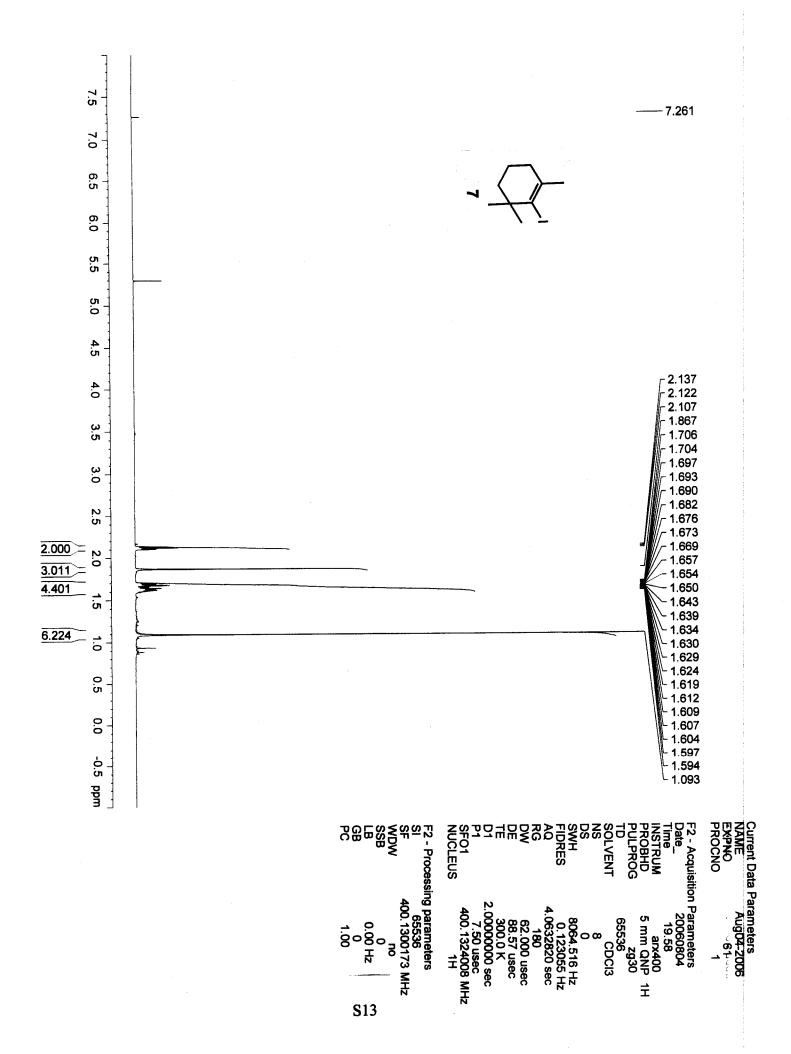


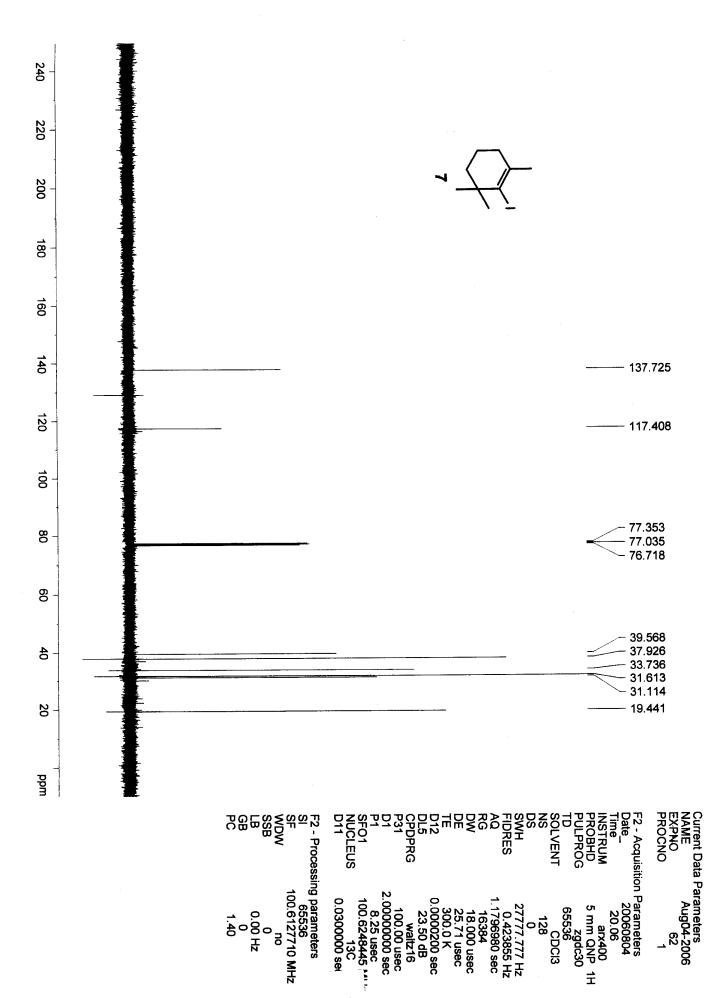


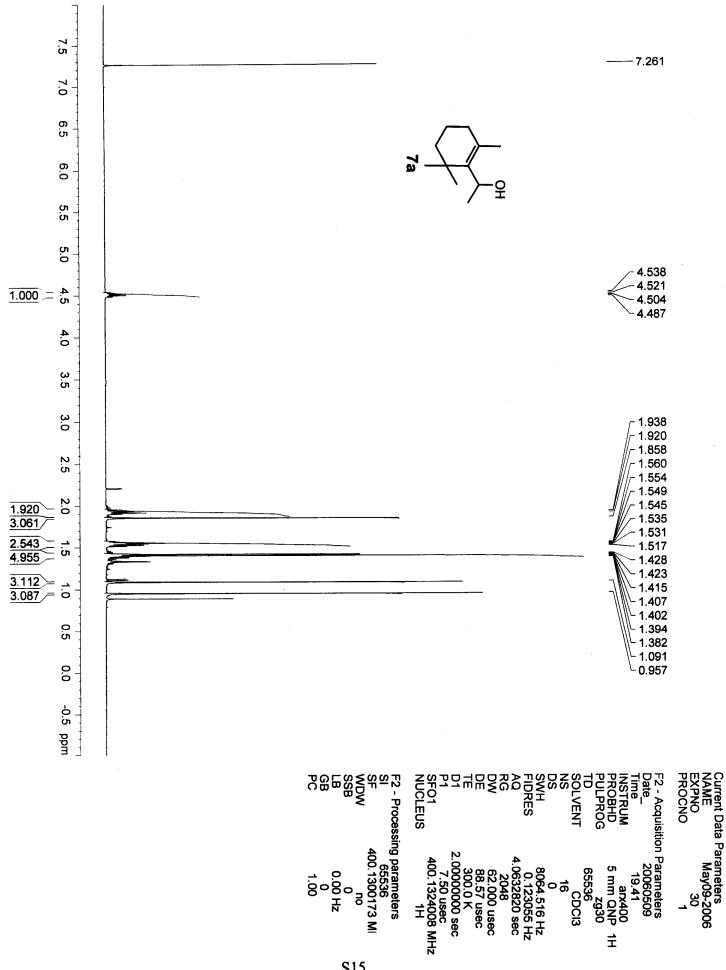


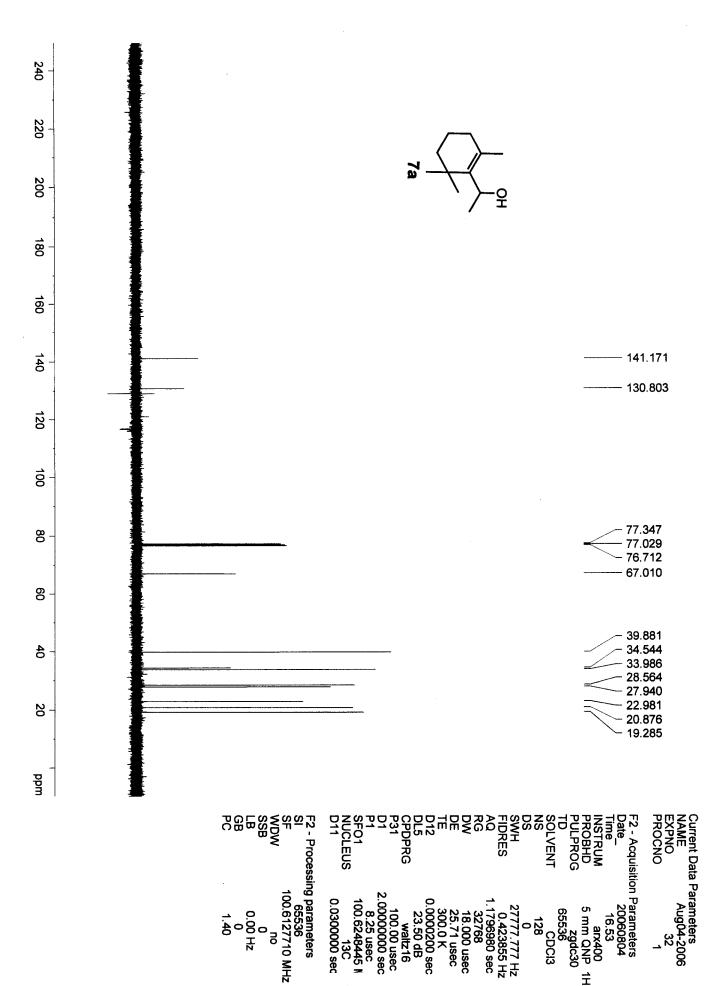


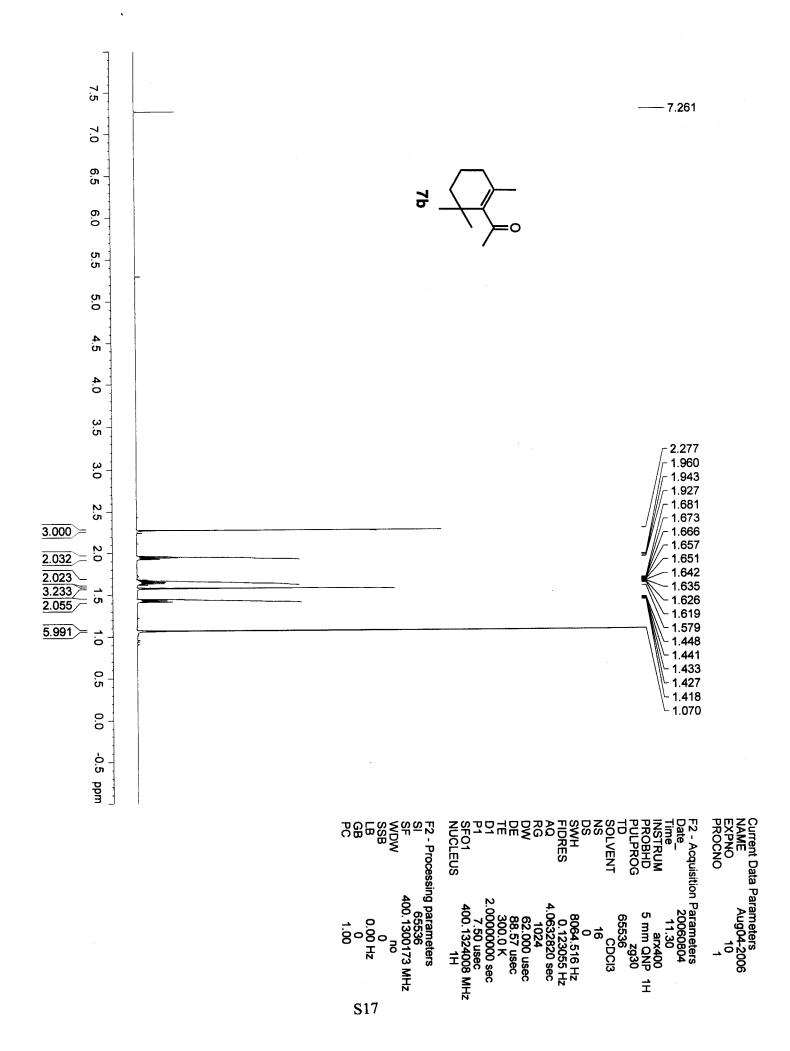
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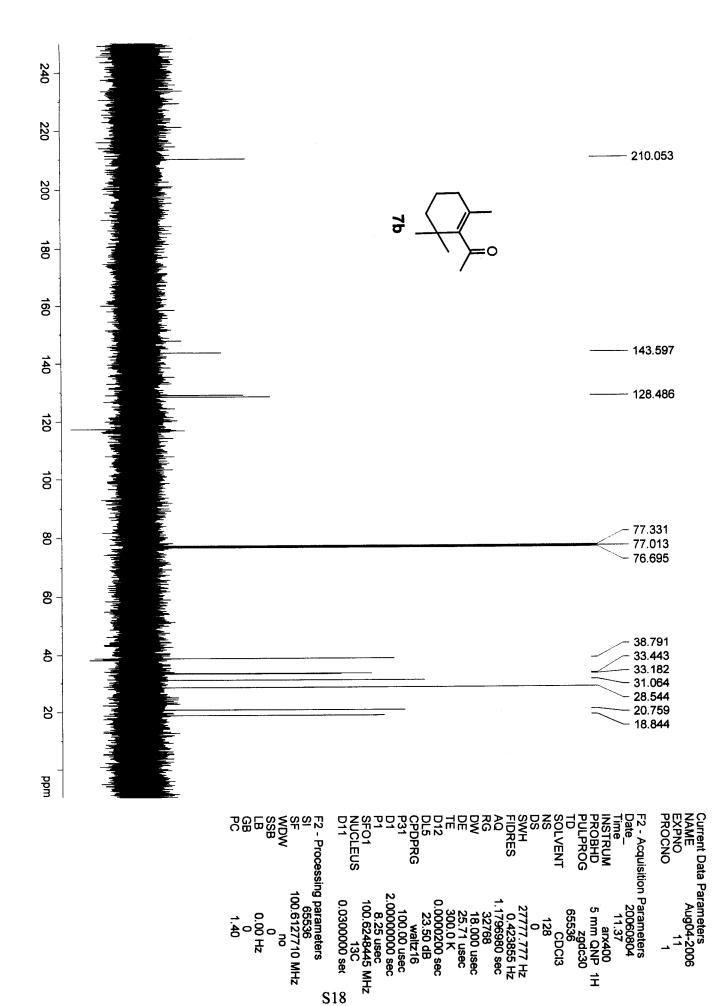


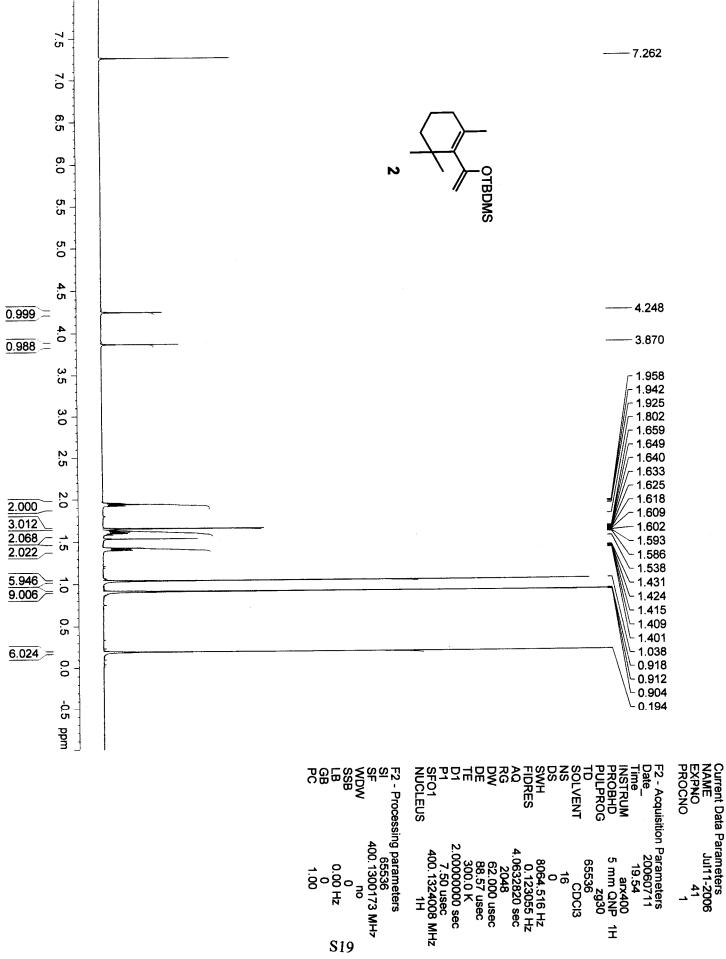


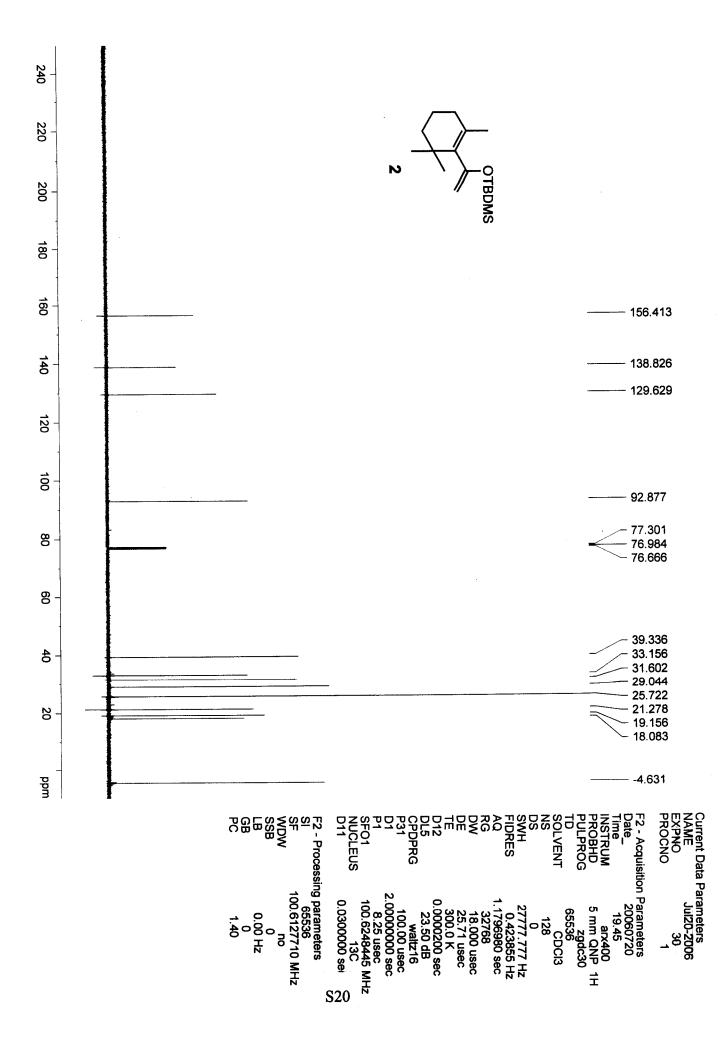


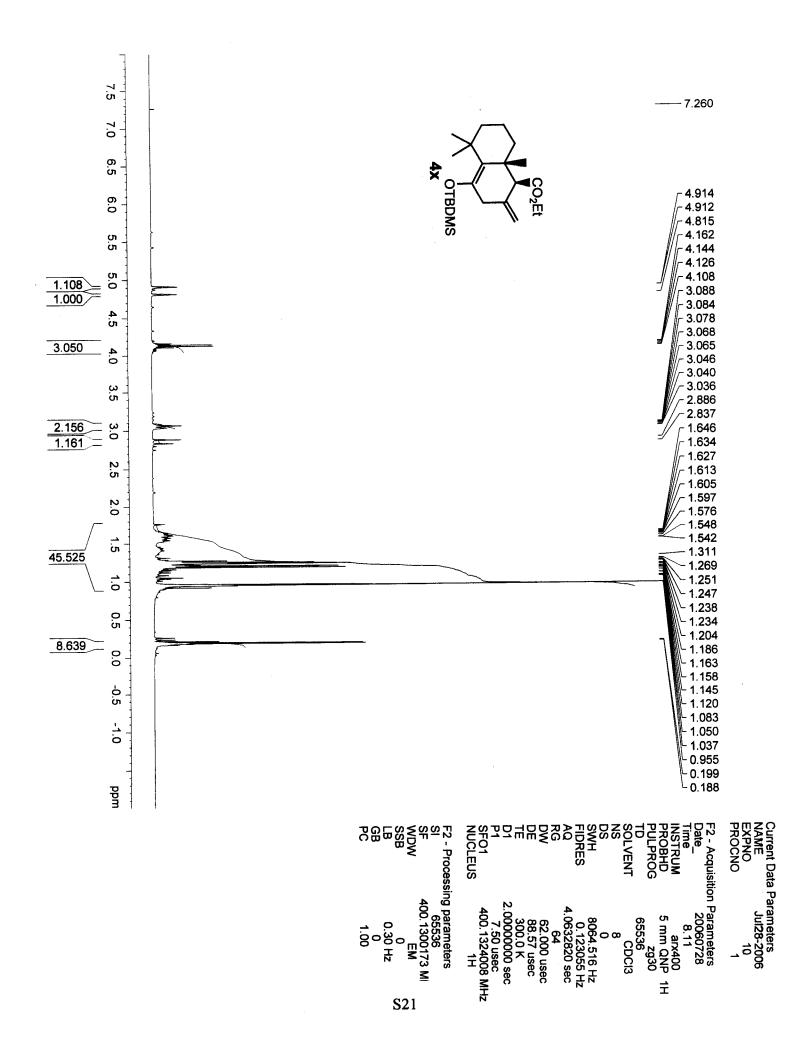


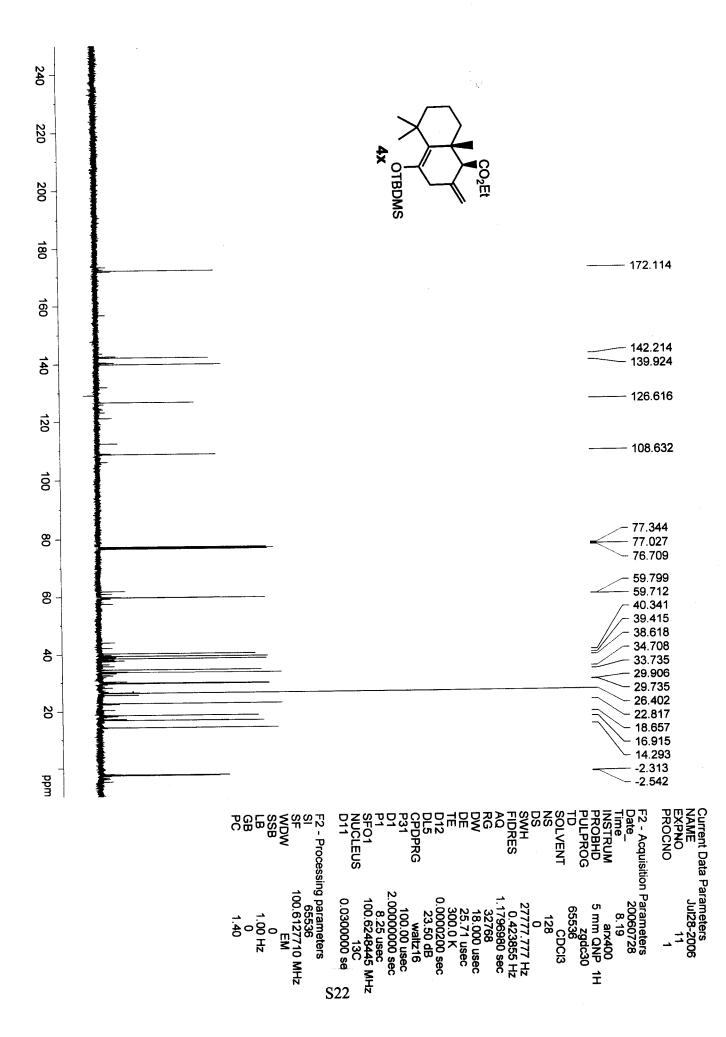


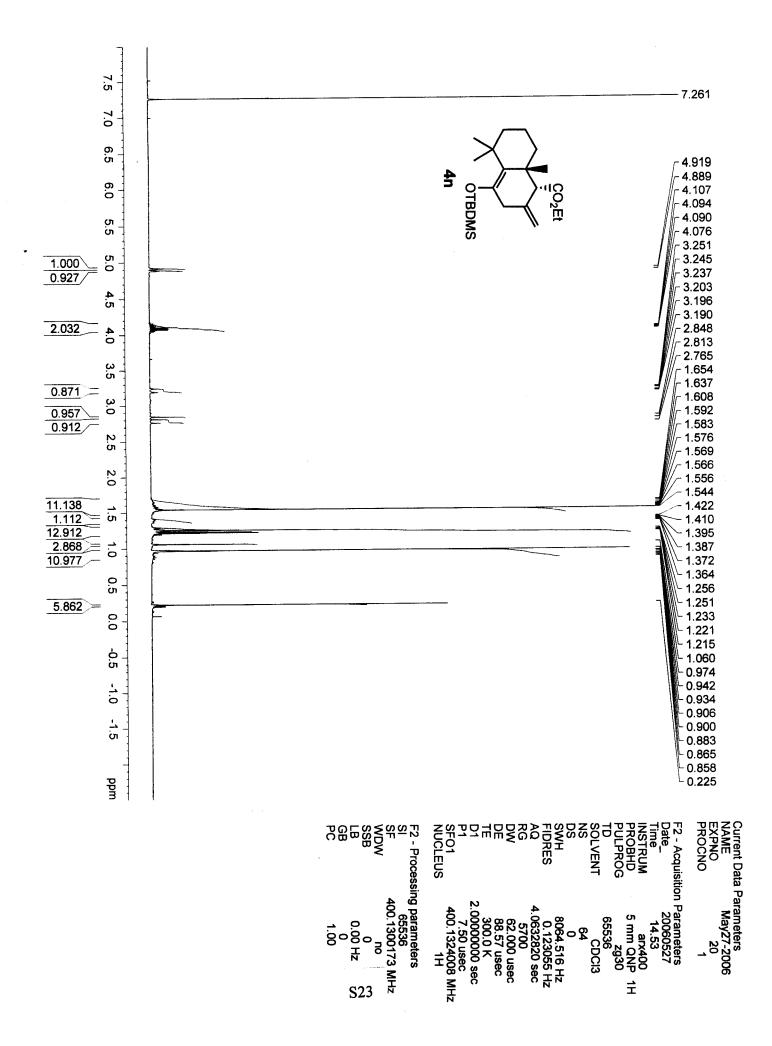


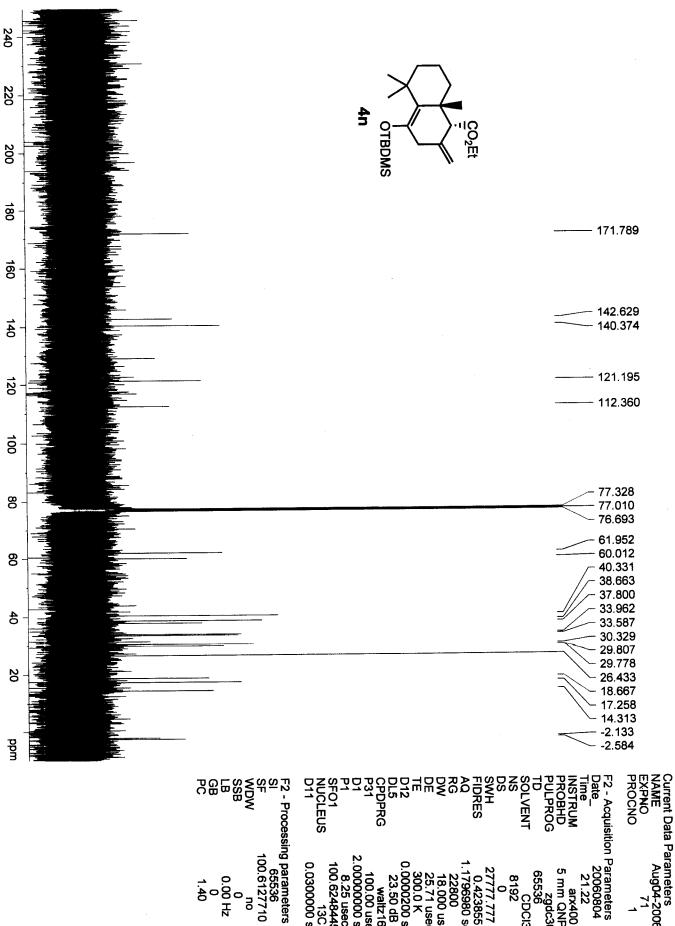












18.000 usec 25.71 usec 300.0 K 0.0000200 sec 23.50 dB walt216 100.00 usec 8.25 usec 100.6248445 M 13C 100.6127710 MHz 0.0300000 sec .1796980 sec 27777.777 Hz 0.423855 Hz 0.00 Hz 1.40 22800 Ы

zgdc30 65536

8192

CDCI3

5 mm QNP

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arx400

Aug04-2006

