Supporting Information

Total Synthesis of (±)-Hedychilactone B: Stepwise Allenolate Diene Cycloaddition to Prepare Trimethyldecalin Systems

Michael E. Jung and Masayuki Murakami

*Department of Chemistry and Biochemistry, University of California, Los Angeles, CA
90095-1569, USA*

Experimental Details.....	2
Proton and Carbon NMR's of 9	10
Proton and Carbon NMR's of 13	12
Proton and Carbon NMR's of 14	14
Proton and Carbon NMR's of 15	16
Proton and Carbon NMR's of 1	18

Supporting Information

Total Synthesis of (±)-Hedychilactone B: Stepwise Allenoate Diene Cycloaddition to Prepare Trimethyldecalin Systems

Michael E. Jung and Masayuki Murakami

*Department of Chemistry and Biochemistry, University of California, Los Angeles, CA
90095-1569, USA*

General Experimental: All ^1H NMR and ^{13}C NMR spectra were obtained on a Bruker ARX-400 spectrometer operating at 400.132 MHz and at 100.625 MHz, respectively. All ^1H and ^{13}C 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 plate 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. A microwave apparatus, Discover LabMate (CEM), was used in the standard configuration as delivered, including proprietary software

(±) Ethyl (1*R*,8*aS*) 4-(1,1-dimethylethyl)dimethylsilyloxy-5,5,8*a*-trimethyl-2-methylene-1,2,3,5,6,7,8,8*a*-octahydro-1-naphthalenecarboxylate (**4x**), (±) Ethyl (1*R*,8*aS*) 4-(1,1-dimethylethyl)dimethylsilyloxy-5,5,8*a*-trimethyl-2-methylene-1,2,3,5,6,7,8,8*a*-octahydro-1-naphthalenecarboxylate (**4x**), and (±) Ethyl 2-[3-(1,1-dimethylethyl)dimethylsilyloxy-3-(2,6,6-trimethyl-1-cyclohexenyl)cyclobutylidene]-acetate (**5**). The diene **2** (1.45 g, 5.17 mmol), the allenolate **3** (1.45 g, 2.5 eq), and hydroquinone (5 mg) were added to a tube. The tube was sealed and 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 the starting diene **2** (462 mg, 30.8%), 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. Separate thermolysis of **5** in a sealed tube gave a quantitative yield of a 2:1 mixture of **4x** and **4n**. 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 small amount of mixture of **4x** and **4n**.

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.

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.

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). ^{13}C -NMR (100 MHz, CDCl_3) 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.

Transformation of **5** to **4x** and **4n**

5 (12 mg, 0.03 mmol) and hydroquinone (small portion) in deuterio-toluene (0.6 mL) were added to a sealed process microwave tube. The tube was heated in a CEM microwave apparatus at 200 °C for 2 hours. After evaporation of the solvent, 2:1 mixture of **4x** and **4n** (12 mg, 100%) was obtained as a colorless oil.

($\pm$) (**1R,8aR**) 4-(1,1-Dimethylethyl)dimethylsilyloxy-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenemethanol (**6n**) and ($\pm$) (**1R,8aS**)-4-(1,1-Dimethylethyl)dimethylsilyloxy-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenemethanol (**6x**). A solution of 1 *M* diisobutylaluminum hydride (DIBAL) in hexane (6.0 mL, 3.3 eq) was added dropwise to a solution of the 2:1 mixture of **4x** and **4n** (710 mg, 1.8 mmol) in dry dichloromethane (14 mL) under Dry Ice acetone cooling. The reaction mixture was stirred for 1 h and 50% sodium hydroxide solution (1.0 mL) was added dropwise. The mixture was allowed to warm to 22 °C. After the mixture stirred 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 the alcohols **6n** (207 mg, 32.7%) and **6x** (404 mg, 63.7%) both as colorless needles.

6n: ^1H -NMR (400 MHz, CDCl_3) 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). ^{13}C -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.

6x: 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.

($\pm$) (1R,8aS)-4-(1,1-Dimethylethyl)dimethylsilyloxy-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydro-1-naphthalenecarboxaldehyde (7). Dess-Martin periodinane (1.27 g, 1.5 eq) was added to a solution of the alcohol **6x** (700 mg, 2.0 mmol) in dry dichloromethane (28 mL) at 22 °C. The reaction mixture was stirred for 1 h under water bath cooling (~ 25 °C). To the reaction mixture was added ether (28 mL), a 1 M solution of sodium thiosulfate (14 mL), and saturated sodium bicarbonate solution (14 mL). The layers were separated and the organic layer was washed with saturated sodium bicarbonate solution (14 mL) and water (14 mL), successively. The organic layer was dried over anhydrous sodium sulfate, filtered, and then evaporated in vacuo. The residue was chromatographed through silica gel (60 g, pentane/ether = 40/1) to afford the aldehyde **7** (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.

($\pm$) (2R,4aS) 4-(1,1-Dimethylethyl)dimethylsilyloxy-1-[(E)-2-methoxyethenyl]-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydronaphthalene and ($\pm$) (2R,4aS) 4-

(1,1-Dimethylethyl)dimethylsilyloxy-1-[(Z)-2-methoxyethenyl]-5,5,8a-trimethyl-2-methylene-1,2,3,5,6,7,8,8a-octahydronaphthalene (9). A solution of 2 *M* *n*-butyllithium in pentane (1.38 mL, 2 eq) was added to a slurry of methoxymethyltriphenylphosphonium iodide⁵ (**8**, 1.32 g, 2.2 eq.) in dry THF (10 mL) under Dry Ice acetone cooling. The resulting slurry was stirred for 1 h under Dry Ice acetone cooling. A solution of the aldehyde **7** (480 mg, 1.38 mmol) in dry THF (8 mL) was added dropwise to the reaction mixture. The resulting slurry was stirred for 1 h under Dry Ice acetone cooling. Water (18 mL) and ether (20 mL) were added to reaction mixture and the mixture was allowed to warm to 22 °C. The layers were separated and the aqueous layer was extracted with ether (20 mL x 2). The organic layers were combined, washed with saturated sodium chloride solution, dried over anhydrous sodium sulfate, filtered, and then evaporated in vacuo. The residue was chromatographed through silica gel (50 g, deactivated by triethylamine, pentane) to afford the mixture of enol ethers **9** (321 mg, 61.9%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 6.20 (1H, d, *J* = 12.6 Hz), 6.03 (1H, dd, *J* = 6.5, 0.6 Hz), 4.70-4.88 (5H, m), 4.39 (1H, dd, *J* = 10.2, 6.5 Hz), 3.57 (3H, s), 3.56 (3H, s), 3.02-3.18 (3H, m), 2.81-2.92 (2H, m), 2.44 (1H, m), 1.53-1.71 (8H, m), 1.10-1.40 (4H, m), 1.25 (6H, s), 1.22 (6H, s), 0.91 (3H, s), 0.89 (3H, s), 0.214 (3H, s), 0.209 (3H, s), 0.204 (3H, s), 0.199 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 148.2, 147.1, 146.8, 145.7, 142.4, 142.3, 126.8, 126.6, 107.9, 107.4, 104.6, 100.7, 59.5, 56.2, 52.7, 48.0, 40.7, 40.6, 39.3, 39.0, 38.6, 38.3, 36.0, 34.8, 34.0, 33.9, 30.0, 29.9, 29.6, 29.4, 26.5, 22.5, 22.1, 18.69, 18.67, 17.1, 17.0, -2.2, -2.3, -2.49, -2.52.

(±) (1aR,2R,4aS) Octahydro-4-[(E)-2-methoxyethenyl]-4a,8,8-trimethyl-3-methylene-1(2H)-naphthalenone and (±) (1aR,2R,4aS) Octahydro-4-[(Z)-2-methoxyethenyl]-4a,8,8-trimethyl-3-methylene-1(2H)-naphthalenone (13). HF-pyridine (0.5 mL) was added to a solution of the silyl enol ether **9** (301 mg, 799 μmol) at -10 °C and then more HF pyridine (0.5 mL) was added to the mixture at -20 °C. The mixture was stirred for 4 h

at -20 °C. Water (30 mL) and ether (50 mL) were added to the reaction mixture and the layers were separated. The aqueous layer was extracted with ether (50 mL). The organic layers were combined and washed with water (30 mL), saturated sodium bicarbonate solution (30 mL), and saturated sodium chloride solution. The organic layer was dried over Na₂SO₄, filtered, and evaporated in vacuo. The residue was chromatographed through silica gel (30 g, pentane/ether = 25/1 to 20/1) to afford the non-conjugated enone **13** (137 mg, 65.3%) as colorless crystals. ¹H-NMR (400 MHz, CDCl₃) δ 6.31 (1H, d, *J* = 12.4 Hz), 6.10 (1H, dd, *J* = 6.4, 0.6 Hz), 4.79 (1H, m), 4.73-4.76 (3H, m), 4.71 (1H, dd, *J* = 12.6, 2.5 Hz), 4.37 (1H, dd, *J* = 9.9, 6.4 Hz), 3.60 (3H, s), 3.58 (3H, s), 3.39 (1H, br d, *J* = 10.8 Hz), 3.11-3.23 (2H, m), 3.05 (2H, dd, *J* = 15.0, 8.1 Hz), 2.64 (1H, d-like, *J* = 10.0 Hz), 2.25 (1H, s), 2.15 (1H, s), 1.04-1.70 (12H, m), 1.22 (3H, s), 1.21 (3H, s), 0.72 (3H, s), 0.69 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 208.4, 208.0, 149.1, 147.9, 145.8, 144.6, 110.7, 110.0, 102.8, 99.1, 65.9, 65.8, 59.7, 56.4, 56.2, 53.9, 53.8, 51.4, 43.0, 42.9, 40.8, 40.7, 40.4, 38.9, 32.9, 32.8, 32.7, 32.6, 21.7, 18.70, 18.67, 16.0, 15.5.

(±) (1*S*,2*R*,4*aS*,8*aR*)-Decahydro-4-[(*E*)-2-methoxyethenyl]-4*a*,8,8-trimethyl-3-methylene-1-naphthalenol and (±) (1*S*,2*R*,4*aS*,8*aR*)-Decahydro-4-[(*Z*)-2-methoxyethenyl]-4*a*,8,8-trimethyl-3-methylene-1-naphthalenol (14**).** A solution of 1 *M* diisobutylaluminum hydride (DIBAL) in hexane (2.8 mL, 6 eq) was added dropwise to a solution of the enone **13** (124 mg, 473 μmol) in dry THF (5 mL) under Dry Ice acetone cooling. The reaction mixture was stirred for 1 h and saturated potassium sodium tartrate solution (10 mL) was added dropwise. The mixture was allowed to warm to 22 °C. After the reaction stirred for 30 min, ethyl acetate (50 mL) and water (10 mL) was added. The layers were separated and the organic layer was washed with water (10 mL) and saturated sodium chloride solution (10 mL). The crude product obtained by evaporation of the organic layer was chromatographed through silica gel (5 g, dichloromethane) to afford the alcohol **14** (118 mg, 94.4%) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 6.22 (1H, d,

$J = 12.5$ Hz), 6.02 (1H, dd, $J = 6.5, 0.7$ Hz), 4.91 (1H, m), 4.88 (1H, m), 4.84 (2H, m), 4.81 (1H, dd, $J = 12.5, 2.2$ Hz), 4.45 (1H, dd, $J = 10.2, 6.6$ Hz), 4.38 (2H, m), 3.57 (3H, s), 3.56 (3H, s), 2.97 (1H, br d, $J = 10.2$ Hz), 2.34-2.48 (4H, m), 2.23 (1H, br d, $J = 10.4$ Hz), 1.53-1.64 (4H, m), 1.33-1.49 (4H, m), 1.224 (3H, s), 1.216 (3H, s), 0.94-1.12 (4H, m), 1.07 (3H, s), 1.04 (3H, s), 1.010 (3H, s), 1.007 (3H, s). ^{13}C -NMR (100 MHz, CDCl_3) δ 148.2, 147.0, 146.7, 145.6, 111.5, 111.0, 103.6, 99.7, 69.2, 69.1, 59.5, 57.00, 56.99, 56.8, 56.2, 52.0, 46.5, 44.11, 44.10, 43.7, 42.7, 40.1, 39.9, 34.5, 34.4, 33.63, 33.62, 19.3, 17.7, 17.3.

(±) (1*S*,2*R*,4*aS*,8*aR*)-Decahydro-4-hydroxy-5,5,8*a*-trimethyl-2-methylene-1-naphthaleneacetaldehyde (15). To a solution of the alcohol **14** (13 mg, 49.2 μmol) was added 2.2 N aqueous HCl solution (0.44 mL) and the mixture was stirred overnight. The solvent was evaporated and saturated sodium bicarbonate solution (1 mL) and dichloromethane (2 mL) were added. The layers were separated and the aqueous layer was extracted with dichloromethane (2 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 (0.5 g, dichloromethane) to afford the aldehyde **15** (8 mg, 65.0%) as a colorless oil. ^1H -NMR (400 MHz, CDCl_3) δ 9.68 (1H, dd, $J = 3.1, 1.0$ Hz), 4.99 (1H, br s), 4.67 (1H, br s), 4.43 (1H, m), 2.59 (1H, ddd, $J = 16.9, 10.9, 3.1$ Hz), 2.35-2.52 (5H, m), 1.47-1.70 (3H, m), 1.40 (1H, m), 1.10-1.27 (2H, m), 1.22 (3H, s), 1.03 (3H, s), 1.02 (3H, s). ^{13}C -NMR (100 MHz, CDCl_3) δ 202.9, 144.3, 111.6, 69.1, 57.2, 51.4, 47.1, 43.8, 42.3, 40.0, 39.8, 34.4, 33.6, 23.7, 19.4, 17.4.

(±)-Hedychilactone B (1). A solution of the aldehyde **15** (14 mg, 55.9 μmol), dihydro-3-(triphenylphosphoranylidene)-2(3*H*)-furanone⁸ **21** (77 mg, 4 eq) in dry dichloromethane (0.5 mL) was stirred for 6.5 h. The reaction mixture was chromatographed through silica gel (2 g, pentane/ether = 5/1 to 3/1) to afford **1** (14 mg, 78.6%) as colorless crystals and a

mixture of **15**, **1**, and the *Z*-isomer of **1** (0.2:1:0.3, 4 mg) as a colorless oil. ¹H-NMR (400 MHz, CDCl₃) δ 6.72 (1H, m), 5.00 (1H, br s), 4.67 (1H, br s), 4.40 (1H, m), 4.39 (2H, t-like, *J* = 7.4 Hz), 2.88 (2H, m), 2.22-2.44 (4H, m), 1.92 (1H, br d, *J* = 11.1 Hz), 1.71 (1H, m), 1.47-1.68 (2H, m), 1.39 (1H, m), 1.07-1.27 (3H, m), 1.22 (3H, s), 1.04 (3H, s), 1.01 (3H, s). ¹³C-NMR (100 MHz, CDCl₃) δ 171.3, 143.8, 141.6, 124.9, 111.2, 69.1, 65.3, 57.3, 56.8, 47.3, 43.8, 42.2, 40.6, 34.4, 33.6, 25.4, 25.3, 23.7, 19.5, 17.1. The spectral data matches that reported for **1**.^{1b}

Current Data Parameters
NAME Sep01-2006
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060901
Time 9.04
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCI3
NS 16
DS 0
SWH 8064.516 Hz
FIDRES 0.123055 Hz
AQ 4.0632820 sec
RG 2048
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
D1 2.0000000 sec
P1 7.50 usec
SFO1 400.1324008 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 65536
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME Sep01-2006
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060901
Time 10.55
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCI3
NS 256
DS 0
SWH 27777.777 Hz
FIDRES 0.423855 Hz
AQ 1.1796980 sec
RG 32768
DW 18.000 usec
DE 25.71 usec
TE 300.0 K
D12 0.0000200 sec
DL5 23.50 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.0000000 sec
P1 8.25 usec
SFO1 100.6248445 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127710 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME Sep14-2006
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060914
Time 19.47
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDCI3
NS 8
DS 0
SWH 8064.516 Hz
FIDRES 0.123055 Hz
AQ 4.0632820 sec
RG 512
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
D1 2.00000000 sec
P1 7.50 usec
SFO1 400.1324008 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 65536
SF 400.1300173 MHz
WDW no
SSB 0
LB 0
GB 0
PC 1.00

Current Data Parameters
NAME Sep14-2006
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060914
Time 20.15
INSTRUM airx400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 0
SWH 27777.777 Hz
FIDRES 0.423855 Hz
AQ 1.1796980 sec
RG 32768
DW 18.000 usec
DE 25.71 usec
TE 300.0 K
D12 0.000200 sec
DL5 23.50 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.0000000 sec
P1 8.25 usec
SFO1 100.6248445 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127710 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME Sep19-2006
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060919
Time 15.24
INSTRUM airx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8064.516 Hz
FIDRES 0.123055 Hz
AQ 4.0632820 sec
RG 715
DW 62.000 usec
DE 88.57 usec
TE 300.0 K
D1 2.00000000 sec
P1 7.50 usec
SFO1 400.1324008 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 65536
SF 400.1300173 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Current Data Parameters
NAME Sep19-2006
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060919
Time 15:38
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 27777.777 Hz
FIDRES 0.423855 Hz
AQ 1.1796980 sec
RG 32768
DW 18.000 usec
DE 25.71 usec
TE 300.0 K
D12 0.000200 sec
DL5 23.50 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.0000000 sec
P1 8.25 usec
SFO1 100.6248445 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127710 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

Current Data Parameters
 NAME Sep20-2006
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060920
 Time 19:23
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCI3
 NS 16
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 715
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 65536
 SF 400.1300173 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
NAME Sep20-2006
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060920
Time 19.51
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 27777.777 Hz
FIDRES 0.423855 Hz
AQ 1.1796980 sec
RG 32768
DW 18.000 usec
DE 25.71 usec
TE 300.0 K
D12 0.0000200 sec
DL5 23.50 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 8.25 usec
SFO1 100.6248445 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127710 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

Current Data Parameters
 NAME Sep22-2006
 EXPNO 70
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060922
 Time 19.16
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 715
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 65536
 SF 400.1300173 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
NAME Sep22-2006
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060922
Time 19:54
INSTRUM arx400
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 27777.777 Hz
FIDRES 0.423855 Hz
AQ 1.1796980 sec
RG 16384
DW 18.000 usec
DE 25.71 usec
TE 300.0 K
D12 0.000200 sec
DL5 23.50 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.0000000 sec
P1 8.25 usec
SFO1 100.6248445 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127710 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40