Vinylketones to Allenes: Preparation of 1,3-Dien-2-yl Triflates and Their Application in Pd-Catalyzed Reaction with Soft Nucleophiles

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Experimental Section.

General. All anaerobic and/or moisture sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen or with glovebox techniques under prepurified argon. ^1H NMR (at 400, 396, or 300 MHz) and ^{13}C NMR (at 101, 100, or 75 MHz) chemical shifts are reported in ppm downfield of internal tetramethylsilane. Tetrahydrofuran and Et_2O were distilled from benzophenone-ketyl under nitrogen prior to use. Dichloromethane was distilled from CaH_2 under nitrogen prior to use. $\text{Pd}(\text{PPh}_3)_4$,¹ *N*-(5-chloro-2-pyridyl)triflimide **4**,² $[\text{PdCl}(\pi\text{-allyl})]_2$,³ *dpbp*,⁴ $\text{Pd}(\text{dba})_2$,⁵ and (*R*)-*tms*-BINAP⁶ were prepared according to the reported methods. All the other chemicals were obtained from commercial sources.

Preparation of Vinylketones 1a-f.⁷ A typical procedure is given for the preparation of **1a**. To a THF suspension of PhCH_2ZnCl , which was prepared from PhCH_2MgCl (60 mmol) in THF (50 mL) and dry ZnCl_2 (8.9 g, 65 mmol), was added a solution of acryloyl chloride (5.8 g, 65 mmol) and $\text{Pd}(\text{PPh}_3)_4$ (3.5 g, 3.0 mmol) in THF (40 mL) at 0 °C. The mixture was stirred for 30 min at 0 °C, then quenched with a small amount of water. The mixture was diluted with hexane and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by silica gel chromatography (hexane/ EtOAc = 9/1) followed by vacuum transfer. The vinylketone **1a** was obtained as colorless liquid (6.1 g, 70%).

4-Phenyl-1-buten-3-one (1a).⁸ Yield: 70%. ^1H NMR (CDCl_3): δ 3.87 (s, 2H), 5.82 (dd, J = 1.3 and 10.2 Hz, 1H), 6.30 (dd, J = 1.3 and 17.6 Hz, 1H), 6.40 (dd, J = 10.2 and 17.6 Hz, 1H), 7.20-7.35 (m, 5H). ^{13}C NMR (CDCl_3): δ 47.2, 127.0, 128.7, 129.0, 129.5, 134.0, 135.6, 197.7.

4-Ethyl-1-hexen-3-one (1b).⁹ Yield: 40%. ^1H NMR (CDCl_3): δ 0.85 (t, J = 7.3 Hz, 6H), 1.46-1.55 (m, 2H), 1.60-1.70 (m, 2H), 2.59-2.65 (m, 1H), 5.77 (dd, J = 0.9 and 10.5 Hz, 1H), 6.26 (dd, J = 0.9 and 17.5 Hz, 1H), 6.45 (dd, J = 10.5 and 17.5 Hz, 1H). ^{13}C NMR (CDCl_3): δ 11.9, 24.4, 52.4, 128.0, 136.1, 204.5.

6-Methyl-1-hepten-3-one (1c).¹⁰ Yield: 58%. ^1H NMR (CDCl_3): δ 0.90 (d, J = 6.5 Hz, 6H), 1.48-1.60 (m, 3H), 2.58 (t, J = 7.6 Hz, 2H), 5.81 (dd, J = 1.1 and 10.3 Hz, 1H), 6.24 (dd, J = 1.1 and 17.5 Hz, 1H), 6.35 (dd, J = 10.3 and 17.5 Hz, 1H). ^{13}C NMR (CDCl_3): δ 22.4, 27.8, 32.9, 37.7, 127.9, 136.6, 201.3.

4-Phenyl-1-penten-3-one (1d).¹¹ Yield: 41%. ^1H NMR (CDCl_3): δ 1.43 (d, J = 6.9 Hz, 3H), 3.97 (q, J = 6.9 Hz, 1H), 5.64 (dd, J = 2.0 and 10.1 Hz, 1H), 6.26 (dd, J = 2.0 and 17.4 Hz, 1H), 6.35 (dd, J = 10.1 and 17.4 Hz, 1H), 7.20-7.27 (m, 3H), 7.31-7.34 (m, 2H). ^{13}C NMR (CDCl_3): δ 17.7, 51.0, 127.2, 128.1, 128.3, 129.0, 134.7, 140.4, 199.7.

2-Methyl-4-phenyl-1-penten-3-one (1e).¹² Yield: 42%. ^1H NMR (CDCl_3): δ 1.41 (d, J = 6.9 Hz, 3H), 1.83 (s, 3H), 4.43 (q, J = 6.9 Hz, 1H), 5.70 (s, 1H), 5.97 (s, 1H), 7.19-7.21 (m, 3H), 7.28-7.31 (m, 2H). ^{13}C NMR (CDCl_3): δ 18.3, 19.4, 46.9, 125.3, 126.9, 127.6, 128.9, 141.9, 143.8, 202.2.

5-Methyl-5-phenyl-1-hexen-3-one (1f).¹³ Yield: 73%. ^1H NMR (CDCl_3): δ 1.45 (s, 6H), 2.87 (s, 2H), 5.57 (dd, J = 2.0 and 9.8 Hz, 1H), 6.02 (dd, J = 2.0 and 17.5 Hz, 1H), 6.10 (dd, J = 9.8 and 17.5 Hz, 1H), 7.16-7.20 (m, 1H), 7.28-7.32 (m, 2H), 7.35-7.37 (m, 2H). ^{13}C NMR (CDCl_3): δ 28.9, 37.5, 53.3, 125.5, 125.9, 127.5, 128.2, 137.2, 148.4, 199.4.

Direct Conversion of Vinylketone 1a into Triflate (E)-2a.² To a solution of $\text{Li}[\text{N}(\text{SiMe}_3)_2]$ (2.20 g, 13.2 mmol) in THF (36 mL) was added a THF (25 mL) solution of **1a** (1.75 g, 12.0 mmol) dropwise at -78 °C. The reaction mixture was stirred for 2 h at this temperature, then a THF (25 mL) solution of **4** (5.03 g, 12.8 mmol) was added to this mixture by cannula transfer. The mixture was stirred for further 2 h at this temperature. After removal of the solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane/ Et_2O = 8/1) to afford (*E*)-**2a** (1.52 g, 46%) as a colorless oil. The product was obtained as a single isomer and its geometry was determined by ^1H NMR nOe experiments.

(E)-1-Phenyl-1,3-butadien-2-yl Triflate ((E)-2a). ^1H NMR (CDCl_3): δ 5.47 (dd, J = 1.6 and 11.3 Hz, 1H), 5.77 (d, J = 17.0 Hz, 1H), 6.70 (s, 1H), 6.71 (dd, J = 11.3 and 17.0 Hz, 1H), 7.25-7.41 (m, 5H). ^{13}C NMR

(CDCl₃): δ 119.0 (q, $J_{\text{C-F}} = 320$ Hz), 119.9, 124.0, 127.0, 129.15, 129.19, 129.7, 132.6, 147.1. EI-HRMS Calcd for C₁₁H₉F₃O₃S: 278.0224, Found: 278.0224.

Reaction of Vinylketone 1b and LHMDs. The vinylketone **1b** was treated with Li[N(SiMe₃)₂] as described above and aqueous work-up gave a complex mixture. Dimer **5** and trimer **6** were separated from the mixture by silica gel chromatography. **Dimer 5:** ¹H NMR (CDCl₃): δ 0.74 (t, $J = 7.6$ Hz, 6H), 0.81 (t, $J = 7.6$ Hz, 6H), 1.37-1.47 (m, 2H), 1.51-1.67 (m, 6H), 1.81-1.85 (m, 2H), 2.16-2.20 (m, 2H), 2.25-2.30 (m, 1H), 5.63 (d, $J = 10.5$ Hz, 1H), 6.35 (d, $J = 16.9$ Hz, 1H), 6.82 (dd, $J = 10.5$ and 16.9 Hz, 1H). ¹³C NMR (CDCl₃): δ 8.0, 11.9, 24.4, 24.9, 26.3, 37.0, 52.6, 55.7, 128.5, 131.0, 204.0, 214.4. EI-HRMS Calcd for C₁₆H₂₈O₂: 252.2089, Found: 252.2079. **Trimer 6:** ¹H NMR (CDCl₃): δ 0.77-0.92 (m, 18H), 1.26-1.88 (m, 16H), 2.31-2.37 (m, 1H), 2.48-2.60 (m, 2H), 3.08 (dd, $J = 3.7$ and 13.3 Hz, 1H), 4.54 (s, 1H), 5.13 (d, $J = 10.8$ Hz, 1H), 5.34 (d, $J = 17.2$ Hz, 1H), 5.86 (dd, $J = 10.8$ and 17.2 Hz, 1H). ¹³C NMR (CDCl₃): δ 8.6, 9.9, 11.3, 11.6, 12.1, 12.2, 21.9, 22.4, 24.2, 24.4, 25.0, 26.0, 26.2, 30.4, 42.8, 45.1, 50.3, 53.3, 55.7, 77.4, 115.9, 140.1, 216.1, 219.1. EI-HRMS Calcd for C₂₄H₄₂O₃: 378.3134, Found: 378.3131.

Preparation of Trimethylsilyl Dienol Ethers 7a-f.¹⁴ A typical procedure is given for the preparation of **7a**. A solution of NaI (6.16 g, 41.1 mmol) in acetonitrile (40 mL) was added to an acetonitrile (40 mL) solution of **1a** (4.00 g, 27.4 mmol) at 0 °C under nitrogen. After successive addition of Et₃N (5.80 mL, 41.1 mmol) and Me₃SiCl (5.20 mL, 41.1 mmol), the reaction mixture was warmed to 80 °C and stirred for 12 h. The mixture was quenched with water, then extracted with ether three times. The combined organic solution was washed with saturated aqueous NaHCO₃, water, and brine, then dried over Na₂SO₄. After removal of the solvent under reduced pressure, the residue was purified by vacuum distillation to give (*Z*)-**7a** (3.36 g, 56%) as a colorless oil.

(Z)-Trimethylsilyl 1-Phenyl-1,3-butadien-2-yl Ether ((Z)-7a). ¹H NMR (CDCl₃): δ 0.10 (s, 9H), 5.14 (d, $J = 11.0$ Hz, 1H), 5.47 (d, $J = 17.4$ Hz, 1H), 6.33 (dd, $J = 11.0$ and 17.4 Hz, 1H), 7.14-7.17 (m, 1H), 7.25-7.30 (m, 1H), 7.52-7.53 (m, 2H). ¹³C NMR (CDCl₃): δ 0.8, 114.3, 114.8, 126.4, 128.1, 129.0, 136.3, 136.3, 149.8. EI-HRMS Calcd for C₁₃H₁₈OSi: 218.1127, Found: 218.1119.

Trimethylsilyl 4-Ethyl-1,3-hexadien-3-yl Ether (7b). ¹H NMR (CDCl₃): δ 0.19 (s, 9H), 0.99 (t, $J = 7.8$ Hz, 6H), 2.09-2.20 (m, 4H), 5.00 (dd, $J = 1.8$ and 10.9 Hz, 1H), 5.29 (dd, $J = 1.8$ and 16.9 Hz, 1H), 6.61 (dd, $J = 10.9$ and 16.9 Hz, 1H). ¹³C NMR (CDCl₃): δ 1.0, 12.7, 14.4, 23.0, 23.2, 112.8, 129.0, 131.3, 143.1. EI-HRMS Calcd for C₁₁H₂₂OSi: 198.1440, Found: 198.1434.

(E)- & (Z)-Trimethylsilyl 6-Methyl-1,3-heptadien-3-yl Ether ((E)- and (Z)-7c). **(E)-isomer:** ¹H NMR (CDCl₃): δ 0.20 (s, 9H), 0.90 (d, $J = 6.6$ Hz, 6H), 1.57-1.66 (m, 1H), 1.96-2.01 (m, 2H), 4.86-4.90 (m, 1H), 5.06-5.10 (m, 1H), 5.42-5.47 (m, 1H), 6.49-6.58 (m, 1H). ¹³C NMR (CDCl₃): δ 0.0, 22.1, 29.2, 35.1, 112.4, 113.8, 129.4, 147.9. **(Z)-isomer:** ¹H NMR (CDCl₃): δ 0.21 (s, 9H), 0.91 (d, $J = 6.6$ Hz, 6H), 1.57-1.66 (m, 1H), 1.96-2.01 (m, 2H), 4.81-4.86 (m, 1H), 4.93-4.96 (m, 1H), 5.22-5.27 (m, 1H), 6.14-6.23 (m, 1H). ¹³C NMR (CDCl₃): δ 0.5, 22.3, 29.2, 35.0, 111.3, 115.0, 135.6, 148.9. EI-HRMS Calcd for C₁₁H₂₃OSi (M+H): 199.1518, Found: 199.1520.

(E)- & (Z)-Trimethylsilyl 4-Phenyl-1,3-pentadien-3-yl Ether ((E)- and (Z)-7d). **(E)-isomer:** ¹H NMR (CDCl₃): δ 0.62 (s, 9H), 2.39 (s, 3H), 5.26 (d, $J = 10.8$ Hz, 1H), 5.67 (d, $J = 17.2$ Hz, 1H), 6.67 (dd, $J = 10.8$ and 17.2 Hz, 1H), 7.51-7.67 (m, 5H). ¹³C NMR (CDCl₃): δ 0.6, 22.5, 112.8, 122.8, 126.4, 127.9, 129.0, 132.4, 141.9, 145.5. **(Z)-isomer:** ¹H NMR (CDCl₃): δ 0.15 (s, 9H), 2.43 (s, 3H), 5.51 (d, $J = 10.8$ Hz, 1H), 5.82 (d, $J = 16.7$ Hz, 1H), 7.13 (dd, $J = 10.8$ and 16.7 Hz, 1H), 7.51-7.67 (m, 5H). ¹³C NMR (CDCl₃): δ 0.0, 18.4, 114.3, 119.9, 126.1, 127.7, 129.1, 131.2, 142.1, 144.1. EI-HRMS Calcd for C₁₄H₂₀OSi: 232.1283, Found: 232.1287.

(E)- & (Z)-Trimethylsilyl 2-Methyl-4-phenyl-1,3-pentadien-3-yl Ether ((E)- and (Z)-7e). **(E)-isomer:** ¹H NMR (CDCl₃): δ 0.22 (s, 9H), 1.70 (s, 3H), 1.99 (s, 3H), 4.76 (br, 1H), 4.83 (br, 1H), 7.15-7.39 (m, 5H). **(Z)-isomer:** ¹H NMR (CDCl₃): δ -0.09 (s, 9H), 1.92 (s, 3H), 2.00 (s, 3H), 4.99 (br, 1H), 5.17 (br, 1H), 7.15-7.39 (m, 5H). **(E)- and (Z)-isomers:** ¹³C NMR (CDCl₃): 0.6, 0.9, 19.3, 20.0, 21.7, 22.1, 115.9, 116.8, 118.6, 118.9, 126.2,

126.3, 128.1, 128.3, 129.1, 129.3, 142.4, 142.5, 142.7, 143.6, 147.5, 148.3. EI-HRMS Calcd for $C_{15}H_{22}OSi$: 246.1440, Found: 246.1427.

Preparation of Dienyl Triflates 2a-f (from 7a-f).² A typical procedure is given for the synthesis of (*E*)- and (*Z*)-**2a**. To a solution of (*Z*)-**7a** (1.00 g, 4.58 mmol) in THF (20 mL) was added a MeLi solution (0.98 M in Et_2O ; 5.4 mL, 5.13 mmol) dropwise at 0 °C. The solution was warmed to room temperature and stirred for 30 min. The reaction mixture was cooled to –78 °C, and to this was added a solution of **4** (2.16 g, 5.50 mmol) in THF (20 mL) by cannula transfer. The reaction mixture was slowly warmed to room temperature and stirred for 2 h. After removal of the solvent under reduced pressure, the residue was purified by silica gel chromatography (with hexane) to afford **2a** (0.69 g, 54%) as a colorless oil.

(Z)-1-Phenyl-1,3-butadien-2-yl Triflate ((Z)-2a). 1H NMR ($CDCl_3$): δ 5.40 (d, J = 11.4 Hz, 1H), 5.67 (d, J = 17.4 Hz, 1H), 6.39 (dd, J = 11.4 and 17.4 Hz, 1H), 6.44 (s, 1H), 7.31-7.40 (m, 3H), 7.45-7.47 (m, 2H). ^{13}C NMR ($CDCl_3$): δ 117.5, 118.3 (q, J_{C-F} = 320 Hz), 123.2, 128.7, 129.2, 129.5, 130.6, 131.9, 145.1. EI-HRMS Calcd for $C_{11}H_9F_3O_3S$: 278.0224, Found: 278.0224.

4-Ethyl-1,3-hexadien-3-yl Triflate (2b). 1H NMR ($CDCl_3$): δ 1.08 (t, J = 7.6 Hz, 3H), 1.08 (t, J = 7.6 Hz, 3H), 2.25 (q, J = 7.6 Hz, 2H), 2.29 (q, J = 7.6 Hz, 2H), 5.29 (d, J = 11.5 Hz, 1H), 5.52 (d, J = 17.0 Hz, 1H), 6.52 (dd, J = 11.5 and 17.0 Hz, 1H). ^{13}C NMR ($CDCl_3$): 11.7, 12.9, 23.4, 23.5, 116.3, 118.5 (q, J_{C-F} = 320 Hz), 126.4, 139.7, 140.7. EI-HRMS Calcd for $C_9H_{13}F_3O_3S$: 258.0537, Found: 258.0545.

(E)- & (Z)-6-Methyl-1,3-heptadien-3-yl Triflate ((E)- and (Z)-2c). (*E*)- and (*Z*)-isomers: 1H NMR ($CDCl_3$): δ 0.94 (d, J = 6.4 Hz, 6H of both isomers), 1.69-1.78 (m, 1H of both isomers), 2.08-2.19 (m, 2H of both isomers), 5.23 (d, J = 11.2 Hz, 1H of (*Z*)-isomer), 5.36 (m, 1H of (*E*)-isomer), 5.46-5.69 (m, 2H of both isomers), 6.19-6.28 (m, 1H of (*Z*)-isomer), 6.46-6.56 (m, 1H of (*E*)-isomer). (*E*)-isomer: ^{13}C NMR ($CDCl_3$): δ 22.2, 28.2, 35.1, 115.7, 118.5 (q, J_{C-F} = 318 Hz), 123.5, 125.2, 146.4. (*Z*)-isomer: ^{13}C NMR ($CDCl_3$): δ 22.0, 28.6, 35.3, 117.4, 118.6 (q, J_{C-F} = 319 Hz), 124.8, 129.7, 146.0. EI-HRMS Calcd for $C_9H_{13}F_3O_3S$: 258.0537, Found: 258.0547.

(E)-4-Phenyl-1,3-pentadien-3-yl Triflate ((E)-2d). 1H NMR ($CDCl_3$): δ 2.22 (s, 3H), 5.17 (d, J = 11.5 Hz, 1H), 5.52 (d, J = 17.2 Hz, 1H), 6.27 (dd, J = 11.5 and 17.2 Hz, 1H), 7.24-7.26 (m, 2H), 7.34-7.41 (m, 3H). ^{13}C NMR ($CDCl_3$): δ 20.1, 116.0, 118.5 (q, J_{C-F} = 319 Hz), 124.9, 127.8, 128.3, 128.6, 133.9, 138.6, 143.1. EI-HRMS Calcd for $C_{12}H_{11}F_3O_3S$: 292.0381, Found: 292.0385.

(Z)-4-Phenyl-1,3-pentadien-3-yl Triflate ((Z)-2d). 1H NMR ($CDCl_3$): δ 2.23 (s, 3H), 5.45 (d, J = 11.4 Hz, 1H), 5.70 (d, J = 17.0 Hz, 1H), 6.73 (dd, J = 11.4 and 17.0 Hz, 1H), 7.26-7.29 (m, 2H), 7.32-7.38 (m, 3H). ^{13}C NMR ($CDCl_3$): δ 20.2, 77.2, 117.88, 117.91 (q, J_{C-F} = 320 Hz), 127.0, 128.3, 128.4, 132.0, 137.9, 141.3. EI-HRMS Calcd for $C_{12}H_{11}F_3O_3S$: 292.0381, Found: 292.0385.

(E)-2-Methyl-4-phenyl-1,3-pentadien-3-yl Triflate ((E)-2e). 1H NMR ($CDCl_3$): δ 1.68 (s, 3H), 2.17 (s, 3H), 5.10 (br, 1H), 5.12 (br, 1H), 7.19-7.21 (m, 2H), 7.26-7.34 (m, 3H). ^{13}C NMR ($CDCl_3$): δ 19.8, 21.1, 118.5 (q, J_{C-F} = 320 Hz), 122.6, 127.8, 128.0, 128.5, 131.1, 137.0, 139.2, 145.2. EI-HRMS Calcd for $C_{13}H_{12}F_3O_3S$: 306.0537, Found: 306.0535.

(Z)-2-Methyl-4-phenyl-1,3-pentadien-3-yl Triflate ((Z)-2e). 1H NMR ($CDCl_3$): δ 2.00 (s, 3H), 2.13 (s, 3H), 5.35 (br, 1H), 5.47 (br, 1H), 7.27-7.33 (m, 3H), 7.35-7.39 (m, 2H). ^{13}C NMR ($CDCl_3$): δ 20.7, 21.2, 118.1 (q, J_{C-F} = 321 Hz), 122.4, 127.9, 128.0, 128.4, 130.7, 136.2, 137.9, 143.5. EI-HRMS Calcd for $C_{13}H_{12}F_3O_3S$: 306.0537, Found: 306.0535.

(E)-5-Methyl-5-phenyl-1,3-hexadien-3-yl Triflate ((E)-2f). 1H NMR ($CDCl_3$): δ 1.53 (s, 6H), 5.08 (d, J = 11.4 Hz, 1H), 5.44 (d, J = 17.2 Hz, 1H), 5.73 (dd, J = 11.4 and 17.2 Hz, 1H), 6.00 (s, 1H), 7.22-7.26 (m, 1H), 7.31-7.36 (m, 4H). ^{13}C NMR ($CDCl_3$): δ 31.4, 39.3, 117.8, 118.6 (q, J_{C-F} = 320 Hz), 126.2, 126.5, 126.6, 128.7, 134.4, 146.4, 147.9. EI-HRMS Calcd for $C_{14}H_{15}F_3O_3S$: 320.0694, Found: 320.0703.

(Z)-5-Methyl-5-phenyl-1,3-hexadien-3-yl Triflate ((Z)-2f). 1H NMR ($CDCl_3$): δ 1.58 (s, 6H), 5.26 (d, J = 11.3 Hz, 1H), 5.53 (d, J = 17.2 Hz, 1H), 5.74 (s, 1H), 6.18 (dd, J = 11.3 and 17.2 Hz, 1H), 7.20-7.23 (m, 1H),

7.29-7.35 (m, 4H). ^{13}C NMR (CDCl_3): δ 29.7, 40.4, 117.7, 118.3 (q, $J_{\text{C-F}} = 320$ Hz), 126.0, 126.5, 128.5, 130.6, 132.3, 143.9, 147.7. EI-HRMS Calcd for $\text{C}_{14}\text{H}_{15}\text{F}_3\text{O}_3\text{S}$: 320.0694, Found: 320.0703.

Palladium-Catalyzed Synthesis of Allenes 9. The reactions were carried out according to a reported procedure.¹⁵ The reaction conditions and results are summarized in Table 2. A typical procedure is given for the synthesis of **9am**. A mixture of $[\text{PdCl}(\eta^3\text{-C}_3\text{H}_5)]_2$ (1.5 mg, 8.2 μmol /Pd), dpbp (4.7 mg, 9.0 μmol), and **8m** (80.1 mg, 476 μmol) was dissolved in THF (5 mL) at room temperature. To this mixture was added (*E*)-**2a** (119 mg, 428 μmol) by means of syringe. The reaction mixture was stirred at this temperature for 12 h, then filtered through a short pad of SiO_2 to remove precipitated inorganic salts. The silica gel pad was washed three times with a small amount of Et_2O and the combined solution was evaporated to dryness under reduced pressure. The residue was purified by chromatography on silical gel (hexane/ Et_2O = 4/1) to afford the allene **9am** (114.4 mg, 97%) as a colorless oil.

The allene **9am** was characterized by comparison of its spectroscopic data with those reported previously.¹⁵ The characterization data of the other allenes are listed below.

Dimethyl 2-(4-Ethyl-2,3-hexadienyl)-2-methylpropane-1,3-dioate (9bm). ^1H NMR (CDCl_3): δ 0.98 (t, $J = 7.4$ Hz, 6H), 1.46 (s, 3H), 1.90-1.96 (m, 4H), 2.58 (d, $J = 7.5$, 2H), 3.72 (s, 6H), 4.96-5.00 (m, 1H). ^{13}C NMR (CDCl_3): δ 12.2, 19.8, 25.4, 36.6, 52.3, 53.7, 86.8, 107.9, 172.3, 202.2. EI-HRMS Calcd for $\text{C}_{14}\text{H}_{22}\text{O}_4$: 254.1518, Found: 254.1514.

Diethyl 2-Acetamido-2-(4-ethyl-2,3-hexadienyl)propane-1,3-dioate (9bn). ^1H NMR (CDCl_3): δ 0.96 (t, $J = 7.6$ Hz, 6H), 1.26 (t, $J = 6.8$ Hz, 6H), 1.90-1.93 (m, 4H), 2.03 (s, 3H), 3.03 (d, $J = 7.6$ Hz, 2H), 4.19-4.29 (m, 4H), 4.86-4.89 (m, 1H), 6.79 (s, 1H). ^{13}C NMR (CDCl_3): δ 12.3, 14.0, 23.1, 25.5, 33.5, 62.6, 66.5, 85.7, 108.3, 167.9, 168.8, 202.4. EI-HRMS Calcd for $\text{C}_{17}\text{H}_{27}\text{NO}_5$: 325.1889, Found: 325.1893.

Di-tert-butyl N-(4-Ethyl-2,3-hexadienyl)iminodicarboxylate (9bo). ^1H NMR (CDCl_3): δ 0.99 (t, $J = 7.6$ Hz, 6H), 1.50 (s, 18H), 1.90-1.99 (m, 4H), 4.14 (d, $J = 5.5$ Hz, 2H), 5.24-5.27 (m, 1H). ^{13}C NMR (CDCl_3): δ 12.3, 25.6, 28.2, 45.5, 82.1, 90.0, 110.4, 152.5, 200.5. EI-HRMS Calcd for $\text{C}_{18}\text{H}_{32}\text{NO}_4$ (M+H): 326.2331, Found: 326.2321.

Dimethyl 2-Methyl-2-(6-methyl-2,3-heptadienyl)propane-1,3-dioate (9cm). ^1H NMR (CDCl_3): δ 0.90 (d, $J = 6.6$ Hz, 6H), 1.44 (s, 3H), 1.59-1.68 (m, 1H), 1.83-1.88 (m, 2H), 2.54-2.59 (m, 2H), 3.72 (s, 6H), 4.90-4.93 (m, 1H), 5.02-5.05 (m, 1H). ^{13}C NMR (CDCl_3): δ 19.7, 22.1, 22.2, 28.4, 36.0, 38.2, 52.4, 52.5, 53.9, 84.3, 89.4, 172.2, 172.3, 206.5. EI-HRMS Calcd for $\text{C}_{14}\text{H}_{22}\text{O}_4$: 254.1518, Found: 254.1516. Anal Calcd for $\text{C}_{14}\text{H}_{22}\text{O}_4$: C, 66.12; H, 8.72, Found: C, 66.00; H, 8.68.

Dimethyl 2-Methyl-2-(4-phenyl-2,3-pentadienyl)propane-1,3-dioate (9dm). ^1H NMR (CDCl_3): δ 1.50 (s, 3H), 2.06 (d, $J = 2.8$ Hz, 3H), 2.69 (br, 1H), 2.70 (br, 1H), 3.68 (s, 3H), 3.69 (s, 3H), 5.32-5.34 (m, 1H), 7.17-7.20 (m, 1H), 7.30-7.32 (m, 2H), 7.36-7.37 (m, 2H). ^{13}C NMR (CDCl_3): δ 17.2, 20.0, 35.9, 52.6 (2C), 53.9, 87.4, 100.8, 125.9, 126.7, 128.4, 137.0, 172.3 (2C), 206.1. EI-HRMS Calcd for $\text{C}_{17}\text{H}_{20}\text{O}_4$: 288.1361, Found: 288.1360.

Dimethyl 2-Methyl-2-(2-methyl-4-phenyl-2,3-pentadienyl)propane-1,3-dioate (9em). ^1H NMR (CDCl_3): δ 1.47 (s, 3H), 1.78 (s, 3H), 2.02 (s, 3H), 2.62 (d, $J = 15.6$ Hz, 1H), 2.83 (d, $J = 15.6$ Hz, 1H), 3.48 (s, 3H), 3.67 (s, 3H), 7.15-7.19 (m, 1H), 7.27-7.34 (m, 4H). ^{13}C NMR (CDCl_3): δ 17.1, 19.8, 20.4, 39.7, 52.4, 52.5, 53.3, 96.4, 100.7, 125.8, 126.4, 128.1, 137.7, 172.1, 172.5, 202.3. EI-HRMS Calcd for $\text{C}_{18}\text{H}_{22}\text{O}_4$: 302.1513, Found: 302.1518.

Diethyl 2-Acetamido-2-(2-methyl-4-phenyl-2,3-pentadienyl)propane-1,3-dioate (9en). ^1H NMR (CDCl_3): δ 1.18 (t, $J = 7.1$ Hz, 3H), 1.23 (t, $J = 7.1$ Hz, 3H), 1.65 (s, 3H), 1.78 (s, 3H), 2.00 (s, 3H), 3.14 (d, $J = 16.0$ Hz, 1H), 3.22 (d, $J = 16.0$ Hz, 1H), 4.08-4.23 (m, 4H), 6.63 (br, 1H), 7.17-7.21 (m, 1H), 7.28-7.34 (m, 4H). ^{13}C NMR (CDCl_3): δ 14.26, 14.34, 17.6, 20.5, 23.0, 36.9, 62.87, 62.93, 66.2, 96.3, 101.5, 126.1, 127.0, 128.7, 138.1, 167.9, 168.0, 169.3, 202.7. EI-HRMS Calcd for $\text{C}_{21}\text{H}_{27}\text{NO}_5$: 373.1889, Found: 373.1895.

Di-tert-butyl N-(2-Methyl-4-phenyl-2,3-pentadienyl)iminodicarboxylate (9eo). ^1H NMR (CDCl_3): δ 1.39 (s, 18H), 1.79 (s, 3H), 2.04 (s, 3H), 4.12 (d, $J = 16.0$ Hz, 1H), 4.23 (d, $J = 16.0$ Hz, 1H), 7.14-7.16 (m, 1H), 7.24-

7.28 (m, 2H), 7.35-7.37 (m, 2H). ¹³C NMR (CDCl₃): δ 16.8, 17.3, 28.0, 48.1, 82.2, 99.5, 103.3, 126.0, 126.4, 128.1, 137.8, 152.5, 200.3. EI-HRMS Calcd for C₂₂H₃₁NO₄: 373.2253, Found: 373.2261.

Dimethyl 2-Methyl-2-(5-methyl-5-phenyl-2,3-hexadienyl)propane-1,3-dioate (9fm). ¹H NMR (CDCl₃): δ 1.40 (s, 3H), 1.42 (s, 3H), 1.44 (s, 3H), 2.59-2.71 (m, 2H), 3.71 (s, 3H), 3.73 (s, 3H), 5.08-5.16 (m, 1H), 5.33-5.35 (m, 1H), 7.16-7.20 (m, 1H), 7.24-7.31 (m, 2H), 7.37-7.39 (m, 2H). ¹³C NMR (CDCl₃): δ 19.8, 29.3, 29.4, 36.1, 38.9, 52.4, 52.5, 53.7, 87.8, 102.1, 125.8 (2C), 128.0, 148.9, 172.1, 172.2, 203.7. EI-HRMS Calcd for C₁₉H₂₄O₄: 316.1674, Found: 316.1662.

Palladium-Catalyzed Asymmetric Synthesis of Axially Chiral Allenes 9. The reactions were performed according to a reported procedure.^{16,17} The reaction conditions and the results are summarized in Table 3. A typical procedure is given for the preparation of (*R*)-**9ao**. A mixture of Pd(dba)₂ (28.8 mg, 50.0 μmol), (*R*)-tms-BINAP (43.0 mg, 56.0 μmol), and **8o** (550 μmol; prepared in situ from HN(boc)₂ and KH) was dissolved in THF (5 mL). After stirring the solution at room temperature for 15 min, (*Z*)-**2a** (139 mg, 500 μmol) was added by means of syringe. The mixture was stirred at this temperature for 24 h, then filtered through a short pad of Al₂O₃ to remove precipitated inorganic salts. The alumina pad was washed with a small amount of Et₂O three times and the combined solution was evaporated to dryness under reduced pressure. The residue was purified by chromatography on Al₂O₃ (hexane/Et₂O = 4/1) to afford the allene (*R*)-**9ao** (124 mg, 72%) as a colorless oil.

The enantiopurity of (*R*)-**9ao** and (*R*)-**9an** was determined to be 22% and 79% ee, respectively, by HPLC analysis with a chiral stationary phase column (**9ao**: Daicel Chiralpak OD-H, hexane/PrOH = 100/1, 0.2 mL/min; **9an**: Daicel Chiralcel OJ-H, hexane/PrOH = 9/1, 1.0 mL/min).

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Current Data Parameters

NAME 001
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040122
Time 21.29
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 715
DW 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1300143 MHz
WDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.45000 ppm/cm
HZCM 180.05690 Hz/cm

Current Data Parameters

NAME 001
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040122
Time 21.44
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zgpgc
TO 131072
SOLVENT CDCl3
NS 129
DS 4
SWH 33333.332 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 32768
DW 15.000 usec
DE 21.43 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 100.6254358 MHz
NUCLEUS 13C

F2 - Processing parameters

SI 65536
SF 100.6127717 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -15.000 ppm
F2 -1509.19 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04687 Hz/cm

Current Data Parameters

NAME 001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040121
Time 19.17
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 715
DW 60.000 usec
DE 95.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SF01 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.45000 ppm/cm
HZCM 180.05850 Hz/cm

Current Data Parameters

NAME 001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040121
Time 19.42
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zgpg
TO 131072
SOLVENT CDCl3
NS 257
DS 4
SWH 33333.332 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 32768
DW 15.000 usec
DE 21.43 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SF01 100.6254358 MHz
NUCLEUS 13C

F2 - Processing parameters

SI 65536
SF 100.6127672 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 215.000 ppm
F1 21631.75 Hz
F2P -15.000 ppm
F2 -1509.19 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04687 Hz/cm

Current Data Parameters

NAME 001

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20040202

Time 19.49

INSTRUM arx400

PROBHD 5 mm Multinu

PULPROG zg

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SWH 8333.333 Hz

FIDRES 0.254313 Hz

AQ 1.9661300 sec

RG 512

DW 60.000 usec

DE 85.71 usec

TE 300.0 K

D1 3.00000000 sec

P1 4.00 usec

SFO1 400.1324710 MHz

NUCLEUS 1H

F2 - Processing parameters

SI 15384

SF 400.1300148 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

F1P 8.500 ppm

F1 3401.10 Hz

F2P -0.500 ppm

F2 -200.07 Hz

PPMCM 0.45000 ppm/cm

HZCM 180.05850 Hz/cm

Current Data Parameters

NAME 001

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20040202

Time 20.11

INSTRUM arx400

PROBHD 5 mm Multinu

PULPROG zgpg

TD 131072

SOLVENT CDCl3

NS 129

DS 4

SWH 33333.332 Hz

FIDRES 0.254313 Hz

AQ 1.9661300 sec

RG 32768

DW 15.000 usec

DE 21.43 usec

TE 300.0 K

D1 1.00000000 sec

P1 4.00 usec

SFO1 100.6254358 MHz

NUCLEUS 13C

F2 - Processing parameters

SI 65536

SF 100.6127712 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

F1P 215.000 ppm

F1 21631.75 Hz

F2P -15.000 ppm

F2 -1509.19 Hz

PPMCM 11.50000 ppm/cm

HZCM 1157.04687 Hz/cm

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 COMMONS
 DATIM6 Jul 23 10:56:37 2004
 OBNUGH
 EXMOCOM
 OBFRO 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 8
 ACQTM 5.449 sec
 PD 1.551 sec
 PW1 5.2 us
 IRNUGH
 CTEMP 25.4 c
 SLVNCDCL3
 EXREF 0.00 ppm
 BF 0.01 Hz
 RGAIN 12

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 COMMONS
 DATIM6 Jul 23 11:48:16 2004
 OBNUGH
 EXMOCOM
 OBFRO 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 1000
 ACQTM 1.606 sec
 PD 1.394 sec
 PW1 4.1 us
 IRNUGH
 CTEMP 25.1 c
 SLVNCDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 24

Current Data Parameters

NAME	193-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040830
Time	19.37
INSTRUM	arx400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	8333.333 Hz
FIDRES	0.254313 Hz
AQ	1.9681300 sec
RG	5700
DW	60.000 usec
DE	85.71 usec
TE	300.0 K
D1	1.00000000 sec
P1	4.00 usec
SFO1	400.1324710 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.866 ppm
F1	3547.55 Hz
F2P	-0.618 ppm
F2	-247.09 Hz
PPMCM	0.47418 ppm/cm
HZCM	189.73213 Hz/cm

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COMDB3-2
DATIMue Aug 31 09:32:40 2004
ORNUICIC
EXMORCM
ORFRQ 75.45 MHz
ORSET 124.00 KHz
ORFIN 1840.0 Hz
POINT 131072
FREQU 20408.1 Hz
SCANS 4541
ACQTM 6.423 sec
PD 1.394 sec
PW1 4.1 us
IRNUOH
CTEMP 25.3 c
SOLVENTCL3
EXREF 77.00 ppm
RF 0.62 Hz
RGAIN 24

Current Data Parameters
NAME 219
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041011
Time 22:58
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 9661300 sec
RG 128
DM 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 7.989 ppm
F1 3196.74 Hz
F2P -0.611 ppm
F2 -244.62 Hz
PWCN 0.43003 ppm/cm
HZCN 172.06792 Hz/cm

Current Data Parameters
NAME 219
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041011
Time 23:08
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zgpg
TD 131072
SOLVENT CDCl3
NS 205
DS 4
SWH 33333.332 Hz
FIDRES 0.254313 Hz
AQ 1.966110 sec
RG 32.68
DM 15.000 usec
DE 21.43 usec
TE 300.0 K
D12 0.0000200 sec
DL5 21.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 1.00000000 sec
P1 4.00 usec
SFO1 100.6254358 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters
SI 65536
SF 100.6127250 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 235.194 ppm
F1 23663.56 Hz
F2P -7.455 ppm
F2 -750.02 Hz
PWCN 12.13245 ppm/cm
HZCN 1220.67908 Hz/cm

Current Data Parameters

NAME 189
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040730
Time 19.24
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl₃
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.961100 sec
RG 512
DW 60.000 usec
DE 95.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SF01 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1298821 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.460 ppm
F1 3385.04 Hz
F2P -0.708 ppm
F2 -283.12 Hz
PPMCM 0.45837 ppm/cm
HZCM 183.40773 Hz/cm

Current Data Parameters

NAME 200
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040909
Time 21.04
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.966130 sec
RG 115
DW 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 7.950 ppm
F1 3180.96 Hz
F2P -0.670 ppm
F2 -268.12 Hz
PPMCM 0.43099 ppm/cm
HZCM 172.45370 Hz/cm

Current Data Parameters

NAME 200
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040909
Time 22.49
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zgpg
TD 131072
SOLVENT CDCl3
NS 120
DS 4
SWH 33333.332 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 32768
DW 15.000 usec
DE 21.43 usec
TE 300.0 K
D12 0.0000200 sec
DL5 21.00 dB
CPOPRG waltz16
P31 100.00 usec
D1 1.00000000 sec
P1 4.00 usec
SFO1 100.6254358 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 - Processing parameters

SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 229.980 ppm
F1 23138.87 Hz
F2P -12.976 ppm
F2 -1305.56 Hz
PPMCM 12.14779 ppm/cm
HZCM 1222.22229 Hz/cm

Current Data Parameters

NAME 215
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040930
Time 14.40
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 128
OW 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1299909 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 7.701 ppm
F1 3081.34 Hz
F2P -0.218 ppm
F2 -87.38 Hz
PPHMC 0.39596 ppm/cm
HZCM 158.43620 Hz/cm

DFILEC:\WINNMR95\COMMON\COM005

DATIMThu Sep 30 17:12:30 2004

OBNUC

EXMOCM

OBFRQ 75.45 MHz

OBSET 124.00 KHz

OBFIN 1840.0 Hz

POINT 32768

FREQU 20408.1 Hz

SCANS 64

ACQTM 1.606 sec

PD 1.394 sec

PW1 4.1 us

IRNUQH

CTEMP 26.7 c

SLVNTDCL3

EXREF 77.00 ppm

BF 0.62 Hz

RGAIN 24

DFILEC3\WINNMR95\DATA\Geyanghui\197-H1.als
 COMND7
 DATIMon Sep 06 17:09:55 2004
 OBNUII
 EXMOCION
 OBFRO 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6013.2 Hz
 SCANS 8
 ACQTM 5.449 sec
 PD 1.551 sec
 PWI 5.2 us
 IRNUGH
 CTEMP 25.5 c
 SLVNTCDL3
 EXREF 0.00 ppm
 BF 0.20 Hz
 RGAIN 14

DFILEC3\WINNMR95\COMMON_DEFAULT.ALS
 COMND7
 DATIMon Sep 06 17:36:17 2004
 OBNUII
 EXMOCION
 OBFRO 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.0 Hz
 POINT 32768
 FREQU 20408.1 Hz
 SCANS 500
 ACQTM 1.606 sec
 PD 1.394 sec
 PWI 4.1 us
 IRNUGH
 CTEMP 26.9 c
 SLVNTCDL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 25

Current Data Parameters
NAME 207
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040915
Time 21.37
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 180
DM 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 400.1299980 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 7.800 ppm
F1 3121.09 Hz
F2P -0.376 ppm
F2 -150.51 Hz
PPMCM 0.40882 ppm/cm
HZCM 163.58026 Hz/cm

Current Data Parameters
NAME 207
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040915
Time 15.38
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zgpg
TD 131072
SOLVENT CDCl3
NS 112
DS 4
SWH 33333.332 Hz
FIDRES 0.254313 Hz
AQ 1.9661300 sec
RG 32768
DM 15.000 usec
DE 21.43 usec
TE 300.0 K
D12 0.00000000 sec
DL5 21.00 dB
CPDPRG waltz16
P31 100.00 usec
D1 1.00000000 sec
P1 4.00 usec
SFO1 100.6254358 MHz
NUCLEUS 13C
D11 0.03000000 sec

F2 - Processing parameters
SI 65536
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 233.354 ppm
F1 23478.38 Hz
F2P -11.442 ppm
F2 -1151.25 Hz
PPMCM 12.23982 ppm/cm
HZCM 1231.48132 Hz/cm

Current Data Parameters

NAME 228
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20041023
Time 16.23
INSTRUM arx400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8333.333 Hz
FIDRES 0.254313 Hz
AQ 0.01300 sec
RG 128
DW 60.000 usec
DE 85.71 usec
TE 300.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 400.1324710 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.497 ppm
F1 3399.89 Hz
F2P -0.437 ppm
F2 -174.75 Hz
PPMCM 0.44668 ppm/cm
HZCM 178.73201 Hz/cm

DFILE:WINNMR95\COMM
COMMZ8
DATE: Oct 23 18:28:35 2004
EXMOBCM
OBNUC3C
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
CLPNT 256
TODAT 1
CLFRQ 100.0 Hz
SCANS 148
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
PW2 10.0 us
PW3 10.0 us
PI1 1.000 ms
PI2 1.000 ms
PI3 1.000 ms
IRNUQH
CTEMP 23.6 c
SLVNCDC13
EXREF 77.00 ppm
CLEXR 0.00
RGAIN 24