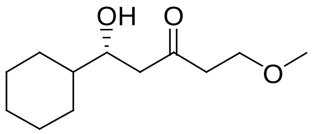
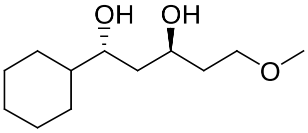
General Experimental

Solvents were purified according to the guidelines in Purification of Common Laboratory Chemicals (Perrin, Armarego, and Perrin, Pergamon: Oxford, 1966). Reagent grade dimethoxypropane, methanol, and acetone were purchased and used without further purification. Samarium metal was purchased from Strem Chemical Company; all other reagents were purchased from Aldrich and used without further purification. The titer of *n*BuLi was determined by the method of Eastham and Watson. Samarium diiodide was freshly prepared using the method of Kagan.[i] Yields were calculated for material judged homogeneous by thin layer chromatography and NMR. Thin layer chromatography was performed on Merck Kieselgel 60 F_{254} plates eluting with the solvents indicated, visualized by a 254 nm UV lamp, and stained with either an ethanolic solution of 12-molybdophosphoric acid, p-anisaldehyde, or a solution of ammonium molybdate/ceric ammonium sulfate. Flash column chromatography was performed with Davisil 62 silica gel, slurry packed with 4% EtOAc/ hexanes in glass columns, and flushed with hexanes prior to use or slurry packed with 1% MeOH/$CHCl_3$ in glass columns, and flushed with chloroform. Preparative chromatography was also carried out using a Chromatotron using glass plates coated with silica gel (P. F. 254 60) of 2 and 4 mm thickness (RPLC). Nuclear magnetic resonance spectra were acquired at 500 MHz for ^1H and 125 MHz for ^{13}C. Chemical shifts for carbon nuclear magnetic resonance (^{13}C NMR) spectra are reported in parts per million downfield relative to the center line of the $CDCl_3$ triplet at 77.0 ppm. The abbreviations s, d, t, q, br s, br t, sep and ABq stand for the resonance multiplicities singlet, doublet, triplet, quartet, broad singlet, broad triplet, septet, and AB quartet, respectively. Optical rotations were obtained (Na D line) using a micro cell with a 1 dm path length. Concentrations are reported in g/100 mL. Melting points were obtained on an Electro thermal melting point apparatus and are uncorrected. Analytical C & H combustion analyses were performed by Atlantic Microlab, Inc., Norcross, Georgia. Glassware for all reactions was oven dried at 125 °C and cooled in a desiccator prior to use. All samarium reduction reactions were carried out with pretreated flasks and stir bars (i.e. flasks and stir bars that had already been previously used for a samarium reduction) or alternatively new flasks and stir bars. High yield always resulted when the reaction vessel, containing substrate, was deoxygenated *in vacuo* and the atmosphere replaced with argon. Liquid reagents and solvents were introduced by oven dried syringes through septa sealed flasks under an argon atmosphere.

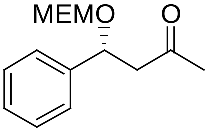
General Procedure for the Preparation of a Stock Solution of 0.10 M SmI_2/THF. To a stirring suspension of samarium chips (608 mg, 4.05 mmol) in 20 mL of THF was added diiodomethane (0.161 mL, 2.02 mmol) dropwise *via* syringe. The resulting mixture was stirred for 2 h before being used.



Analytical Data for (1RS)-1-cyclohexyl-1-hydroxy-5-methoxypentan-3-one (1). Isolated as a colorless solid: mp 26-28 °C; R_f 0.16 (30% acetone/hexane); 500 MHz ^{1}H NMR ($CDCl_3$) δ 3.81 (ddd, *J* = 8.8, 5.9, 2.9 Hz, 1H), 3.64 (ddd, *J* = 15.6, 9.8, 5.9 Hz, 1H), 3.61 (ddd, *J* = 15.6, 9.3, 5.9 Hz, 1H), 3.31 (s, 3H), 3.02 (d, *J* = 3.4 Hz, 1H), 2.68 (q, *J* = 5.9 Hz, 2H), 2.61 (dd, *J* = 17.1, 2.9 Hz, 1H), 2.55 (dd, *J* = 17.1, 9.2 Hz, 1H), 1.83 (br s, *J* = 12.5 Hz, 1H), 1.75-1.72 (m, 2H), 1.66-1.62 (m, 2H), 1.33 (dddd, *J* = 15.1, 11.7, 6.4, 3.4 Hz, 1H), 1.27-1.16 (m, 1H), 1.13 (dddd, *J* = 12.7, 12.7, 3.4, 3.4 Hz, 2H), 1.00 (dddd, *J* = 28.3, 12.2, 12.2, 3.4 Hz, 2H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 210.7, 71.5, 67.3, 58.7, 47.0, 43.4, 42.9, 28.7, 28.1, 26.3, 26.0, 25.9; IR (neat) 1707 cm^{-1}; Anal. Calcd for $C_{12}H_{22}O_3$: C, 67.26; H, 10.35. Found: C, 66.99; H, 10.17.

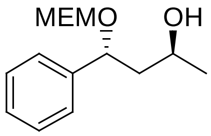


Analytical Data for (3RS, 1SR)-1-cyclohexyl-5-methoxypentane-1,3-diol (2). Isolated in 97% yield as mixture of diastereomers and as a colorless solid: mp(mixture) 48-50 °C; R$_f$ 0.11 (30% acetone/hexane); 500 MHz ^{1}H NMR (CDCl$_3$) δ 4.12 (dddd, *J* = 6.5, 6.5, 3.5, 3.5 Hz, 3H), 4.03-3.99 (m, 1H)(minor), 3.62 (ddd, *J* = 18.5, 9.0, 3.0 Hz, 3H), 3.58 (ddd, *J* = 18.0, 9.0, 4.0 Hz, 1H)(minor), 3.35 (s, 13H), 1.89-1.83 (m, 7H), 1.76-1.69 (m, 12H), 1.67-1.53 (m, 21H), 1.38-1.32 (m, 5H), 1.27-1.10 (m, 14H), 1.04-0.96 (m, 9H); 125 MHz ^{13}C NMR (CDCl$_3$) δ major: 72.9, 71.8, 69.3, 58.9, 43.6, 39.6, 36.2, 28.9, 28.4, 26.5, 26.2, 26.1; minor: 72.5, 71.1, 69.3, 58.9, 44.1, 39.7, 37.0, 28.7, 28.0, 26.5, 26.2, 26.1; IR (CHCl$_3$) 3531 cm^{-1}; Anal. Calcd for $C_{12}H_{24}O_3$: C, 66.63; H, 11.18. Found: C, 66.76; H, 11.23. The ratio of diastereomers was determined to be 26:74 (NaBH$_4$ 31:69) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 10% *i*PrOH/hexane and a flow rate of 0.7 mL/min, which gave retention times for the minor and major diastereomers of 13.6 and 17.0 min, respectively.

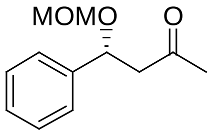


Analytical Data for (4RS)-4-[(2-methoxyethoxy)methoxy]-4-phenylbutan-2-one (5f). Isolated as a colorless oil: R$_f$ 0.37 (50% EtOAc/hexane); 500

MHz ^{1}H NMR ($CDCl_3$) δ 7.38-7.22 (m, 5H), 5.13 (dd, *J* = 9.3, 3.9 Hz, 1H), 4.58 (ABq, J_{ab} = 6.8 Hz, Δν = 37.5 Hz, 2H), 3.78-3.75 (m, 1H), 3.54-3.41 (m, 3H), 3.35 (s, 3H), 3.03 (dd, *J* = 16.1, 9.3 Hz, 1H), 2.62 (dd, *J* = 15.6, 3.9 Hz, 1H), 2.16 (s, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 206.3, 140.8, 128.5, 128.0, 126.8, 93.2, 74.0, 71.6, 67.1, 59.0, 51.5, 31.0; IR (neat) 1718 cm^{-1}; Anal. Calcd for $C_{14}H_{20}O_4$: C, 66.66; H, 7.99. Found: C, 66.49; H, 8.00.

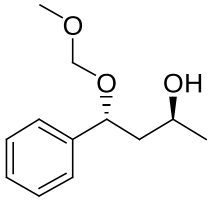


Analytical Data for (2RS, 4SR)-4-[(2-Methoxyethoxy)methoxy]-4-phenylbutan-2-ol (6f). Isolated in 87% yield as a colorless oil: R_f 0.22 (50% EtOAc/hexane); 500 MHz ^{1}H NMR ($CDCl_3$) δ 7.39-7.23 (m, 5H), 4.97 (dd, *J* = 10.3, 2.9 Hz, 1H), 4.60 (ABq, J_{ab} = 6.8 Hz, Δν = 18.9 Hz, 2H), 4.12 (ddq, *J* = 10.3, 6.4, 4.4 Hz, 1H), 3.99-3.94 (m, 1H), 3.61-3.55 (m, 3H), 3.42 (s, 3H), 2.05 (dd, *J* = 6.3, 0.97 Hz, 1H), 1.87 (ddd, *J* = 10.3, 1.9, 0.97 Hz, 1H), 1.67 (ddd, *J* = 13.2, 10.3, 2.4 Hz, 1H), 1.21 (d, *J* = 6.4 Hz, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 142.0, 128.4, 127.5, 129.6, 92.4, 74.5, 71.7, 66.9, 63.4, 59.0, 47.5, 23.3; IR (neat) 3240(br) cm^{-1}; Anal. Calcd for $C_{14}H_{22}O_4$: C, 66.12; H, 8.72. Found: C, 65.96; H, 8.69. The ratio of diastereomers was determined to be 9:91 (44:56 $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 10% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 15.7 and 17.7 min, respectively.

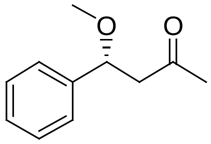


Analytical Data for (4RS)-4-(Methoxymethoxy)-4-phenylbutan-2-one (5d). Isolated as a colorless oil: R_f 0.58 (50% EtOAc/hexane); 500 MHz ^{1}H NMR

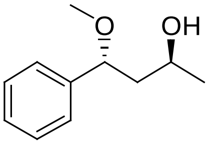
(CDCl$_3$) δ 7.39-7.22 (m, 5H), 5.10 (dd, *J* = 9.5, 4.0 Hz, 1H), 4.58 (ABq, J_{ab} = 6.8 Hz, Δν = 10.8 Hz, 2H), 3.31 (s, 3H), 3.04 (dd, *J* = 16.0, 9.5 Hz, 1H), 2.62 (dd, *J* = 16.5, 4.0 Hz, 1H), 2.18 (s, 3H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 206.3, 140.8, 128.6, 128.0, 126.8, 94.2, 74.0, 55.8, 51.6, 31.0; IR (neat) 1716 cm^{-1}. Anal. Calcd for $C_{12}H_{16}O_3$: C, 69.21; H, 7.74. Found: C, 69.46; H, 7.80.



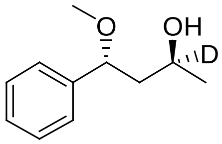
Analytical Data for (2RS, 4SR)-4-(Methoxymethoxy)-4-phenylbutan-2-ol (6d). Isolated in 71% yield as colorless oil: R$_f$ 0.38 (50% EtOAc/hexane); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.39-7.20 (m, 5H), 4.88 (dd, *J* = 9.3, 3.4 Hz, 1H), 4.53 (ABq, J_{ab} = 6.3 Hz, Δν = 9.4 Hz, 2H), 4.05 (ddq, *J* = 9.3, 6.4, 2.4 Hz, 1H), 3.38 (s, 3H), 2.55 (br s, 1H), 1.89 (ddd, *J* = 14.2, 9.3, 2.4 Hz, 1H), 1.74 (ddd, *J* = 14.2, 9.3, 3.4 Hz, 1H), 1.19 (d, *J* = 5.9 Hz, 3H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 141.7, 128.5, 127.6, 126.5, 96.6, 75.7, 64.4, 55.9, 46.6, 23.3; IR 3435(br), 2938 cm^{-1}; Anal. Calcd for $C_{12}H_{18}O_3$: C, 68.54; H, 8.63. Found: C, 68.35; H, 8.69. The ratio of diastereomers was determined to be 6:94 (60:40, NaBH$_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 3% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 22.9 and 23.6 min, respectively.



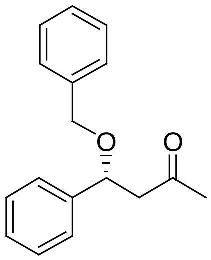
(4RS)-4-methoxy-4-phenylbutan-2-one (5b):[ii] To a stirring solution of acetone (2.0 mL, 1.6 g, 28 mmol) in 300 mL of CH_2Cl_2 at –78 °C was added down the side of the flask a solution of $TiCl_4$ (30 mL, 30 mmol, 1.0 M in CH_2Cl_2) followed by the dropwise addition of DIPEA (6.3 mL, 4.7 g, 36 mmol). After 15 min a solution of benzaldehyde dimethyl acetal (3.8 mL, 3.8 g, 25 mmol, in 50 mL of CH_2Cl_2) was added *via* cannula down the side of the flask. The reaction was stirred 1 h before being quenched cold with a saturated aqueous $NaHCO_3$ solution. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were dried with $MgSO_4$, filtered, and concentrated under reduced pressure. Purification of this material was accomplished by RPLC, using a 4 mm plate, eluting with a gradient of 200 mL of each of 20%, 30%, 40% EtOAc/hexanes, collecting 8 mL fractions. The product containing fractions were collected and concentrated to give material that required further purification. Analytically pure material was obtained by preparative HPLC eluting with 15% EtOAc/hexanes at 20 mL/min, collecting 8 mL fractions. The product containing fractions were collected and concentrated to give (4RS)-4-methoxy-4-phenylbutan-2-one (0.96 g, 22% yield) as colorless oil: R_f 0.50 (50% EtOAc/hexane); 500 MHz ^{1}H NMR ($CDCl_3$) δ 7.37-7.27 (m, 5H), 4.62 (dd, *J* = 8.8, 3.9 Hz, 1H), 3.17 (s, 3H), 2.95 (ddd, *J* = 16.1, 9.3, 1.4 Hz, 1H), 2.56 (ddd, *J* = 16.1, 4.4, 1.4 Hz, 1H), 2.12 (s, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 206.5, 140.9, 128.5, 127.9, 126.5, 79.5, 56.7, 51.8, 30.9; IR 1718 cm^{-1}; Anal. Calcd for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: C, 73.83; H, 7.87.



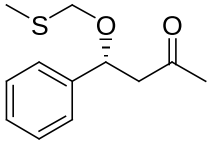
Preparation of (2RS, 4SR)-4-Methoxy-4-phenylbutan-2-ol (6b). Isolated in 99% yield as a colorless oil; R_f 0.27 (50% EtOAc/hexane); 500 MHz ^{1}H NMR ($CDCl_3$) δ 7.36-7.27 (m, 5H), 4.48 (dd, *J* = 8.4, 3.9 Hz, 1H), 4.03 (ddq, *J* = 14.7, 6.4, 2.9 Hz, 1H), 3.24 (s, 3H), 1.87 (ddd, *J* = 14.7, 8.3, 2.9 Hz, 1H), 1.76 (ddd, *J* = 14.7, 8.3, 3.4 Hz, 1H), 1.19 (d, *J* = 6.4 Hz, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 141.1, 128.5, 127.5, 126.4, 81.3, 64.7, 56.7, 46.2, 23.3; IR (neat) 3457(br) cm^{-1}; Anal. Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.17; H, 9.06. The ratio of diastereomers was determined to be 1:99 (55:45, $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 3 % *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 18.3 and 19.5 min, respectively.



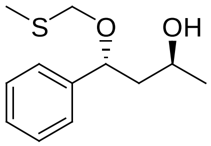
Analytical Data for (2RS, 4SR)-4-methoxy-4-phenylbutan-(2-^{2}H)-2-ol (9). Isolated in 89% yield as a colorless oil: R$_f$ 0.43 (40% EtOAc/hexane); 500 MHz ^{1}H NMR ($CDCl_3$) δ 7.39-7.22 (m, 5H), 4.49 (dd, *J* = 8.5, 3.5 Hz, 1H), 3.25 (s, 3H), 2.77 (s, 1H), 1.89 (dd, *J* = 14.5, 8.5 Hz, 1H), 1.77 (dd, *J* = 14.5, 3.5 Hz, 1H), 1.20 (s, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 141.6, 128.5, 127.6, 126.4, 81.4, 64.3 (t, *J* = 21.9 Hz), 56.7, 46.1, 23.2; Anal. Calcd for $C_{11}H_{15}DO_2$: C, 72.89; H+D, 8.89. Found: C, 72.64; H+D, 8.77. The ratio of diastereomers was determined to be 1:99 (55:45, $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 3% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 19.1 and 20.5 min, respectively.



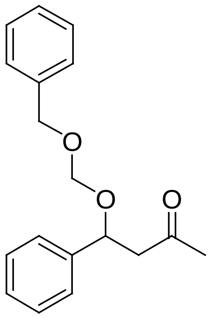
Analytical Data for (4RS)-4-Phenyl-4-(phenylmethoxy)butan-2-one (5h). Isolated as a colorless oil: R$_f$ 0.61 (50% EtOAc/hexane); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.37-7.27 (m, 10H), 4.85 (dd, *J* = 8.8, 4.4 Hz, 1H), 4.31 (ABq, J_{ab} = 11.2 Hz, Δν = 53.0 Hz, 2H), 3.02 (dd, *J* = 16.1, 8.8 Hz, 1H), 2.60 (dd, *J* = 16.1, 4.4 Hz, 1H), 2.12 (s, 3H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 206.6, 141.2, 138.0, 128.7, 128.3, 128.0, 127.9, 127.6, 126.7, 77.6, 70.8, 52.0, 31.0; IR (neat) 1718 cm^{-1}.



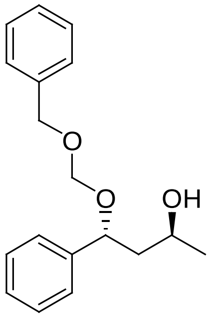
Analytical Data for (4RS)-4-(methylthiomethoxy)-4-phenylbutan-2-one (5e). Isolated as a colorless oil: R$_f$ 0.51 (30% EtOAc/hexanes); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.35-7.32 (m, 5H), 5.21 (dd, *J* =9.3, 3.7 Hz, 1H), 4.59 (d, *J* = 11.7 Hz, 1H), 4.27 (d, *J* =11.7 Hz, 1H), 3.02 (dd, *J* = 16.1, 9.3 Hz, 1H), 2.64 (dd, *J* =15.6, 3.9 Hz, 1H) 2.19 (s, 3H), 2.12 (s, 3H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 206.1, 140.0, 128.6, 128.1, 126.9, 74.2, 72.3, 51.5, 30.8, 14.0; IR (neat) 2916, 1723 cm^{-1}; Anal. Calcd for $C_{12}H_{16}O_2S$: C, 64.25; H, 7.19; S, 14.29. Found: C, 64.36; H, 7.11; S, 14.06.



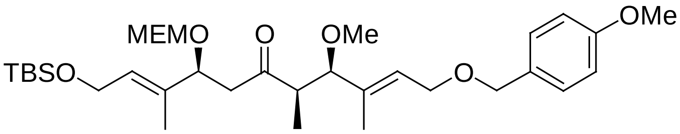
Analytical Data for (2SR, 4RS)-4-(methylthiomethoxy)-4-phenylbutan-2-ol (6e). Isolated in 92% yield as a colorless oil: R$_f$ 0.41 (30% EtOAc/hexanes); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.36-7.25 (m, 5H), 4.93 (dd, *J* = 10.3, 3.4 Hz, 1H), 4.69 (d, *J* = 11.7 Hz, 1H), 4.33 (d, *J* = 11.7 Hz, 1H), 4.07 (ddq, *J* = 8.8, 6.4, 2.4 Hz, 1H), 2.75 (br s, 1H), 2.22 (s, 3H), 1.92 (ddd, *J* = 14.7, 10.3, 2.4 Hz, 1H), 1.71 (ddd, *J* = 14.7, 9.3, 3.4 Hz, 1H), 1.21 (d, *J* = 6.3 Hz, 3H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 141.1, 128.6, 127.8, 126.7, 75.7, 73.3, 64.5, 46.7, 23.0, 14.6; IR (neat) 3452 (br), 2917 cm^{-1}; Anal. Calcd for $C_{12}H_{18}O_2S$: C, 68.54; H, 8.63. Found: C, 68.35; H, 8.69. The ratio of diastereomers was determined to be 4:96 (42:58, $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 4% *i*PrOH/hexane and a flow rate of 0.3 mL/min, which gave retention times for the minor and major diastereomers of 20.4 and 19.7 min, respectively.



Analytical Data for 4RS - 4-phenyl-4-[(phenylmethoxy)methoxy]butan-2-one (5g). Isolated as a colorless oil: R_f 0.51 (40% EtOAc/hexanes); 500 MHz ^{1}H NMR ($CDCl_3$) δ 7.34–7.23 (m, 10H), 5.17 (dd, *J* = 9.3, 4.4 Hz, 1H), 4.65 (Abq, J_{ab} = 6.8 Hz, Δν = 53.3 Hz, 2H), 4.63 (d, *J* = 11.7 Hz, 1H), 4.39 (d, *J* = 11.2 Hz, 1H), 3.02 (dd, *J* = 16.1, 9.3 Hz, 1H), 2.62 (dd, *J* = 16.1, 3.9 Hz, 1H), 1.0 (s, 3H), 125 MHz ^{13}C NMR ($CDCl_3$) δ 206.1, 140.8, 137.7, 128.5, 128.1, 128.0, 127.6, 126.8, 92.4, 74.2, 69.8, 51.5, 31.0; IR (neat) 3170, 1718 cm^{-1} Anal. Calcd for $C_{18}H_{20}O_3$: C, 76.03; H, 7.09. Found: C, 76.12; H, 7.16.



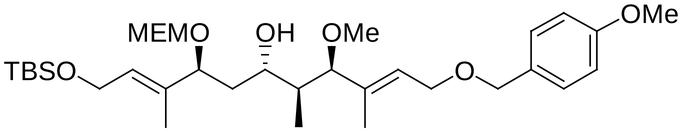
Analytical Data for (2SR,4RS)-4-phenyl-4-[(phenylmethoxy)methoxy]butan-2-ol (6g). Isolated in 41% yield as a colorless oil: R$_f$ 0.38 (40% EtOAc/hexanes); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.34–7.23 (m, 10H), 4.96 (dd, J = 9.3, 3.9 Hz, 1H), 4.65 (Abq, J_{ab} = 6.8 Hz, Δν = 56.2 Hz, 2H), 4.71 (d, J = 11.5 Hz, 1H), 4.51 (d, J = 11.7 Hz, 1H), 4.07 (brs, 1H), 2.45 (brs, 1H), 1.92 (ddd, J = 14.2, 8.8, 2.4 Hz, 1H), 1.76 (ddd, J = 14.6, 9.3, 3.9 Hz, 1H), 1.0 (d, J = 6.4 Hz, 3H) 125 MHz ^{13}C NMR (CDCl$_3$) δ 141.6, 137.6, 128.5, 128.4, 127.9, 127.8, 127.6, 126.6, 92.0, 75.9, 70.0, 64.4, 46.6, 23.5; IR (neat) 3428 (br) cm^{-1}; Anal. Calcd for $C_{18}H_{22}O_3$: C, 75.50; H, 7.74. Found: C, 75.63; H, 7.83. The ratio of diastereomers was determined to be 86:14 (40:60 NaBH$_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 1% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 29.33 and 32.39 min, respectively.



Analytical Data for (2E, 9E)(8S, 4R, 5R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-one (7c). Isolated as a colorless oil: R$_f$ 0.55 (30% acetone/hexane); $[\alpha]^{23}_D$ = - 36.6 (c 2.3, CHCl$_3$); 500 MHz ^{1}H NMR (CDCl$_3$) δ 7.26 (d, J = 8.5 Hz, 2H), 6.87 (d, J = 8.5 Hz, 2H), 5.59-

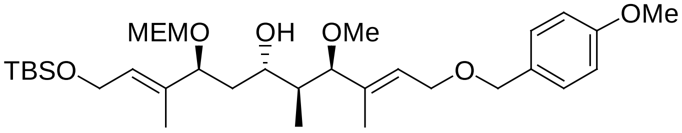
5.75 (m, 2H), 4.57 (ABq, J_{ab} = 6.7 Hz, $\Delta\nu$ = 31.0 Hz, 2H), 4.51 (dd, *J* = 9.3, 3.3 Hz, 1H), 4.41 (s, 2H), 4.20 (dd, *J* = 13.5, 5.8 Hz, 1H), 4.17 (dd, *J* = 13.8, 6.6 Hz, 1H), 4.06 (dd, *J* = 12.6, 6.9 Hz, 1H), 3.99 (dd, *J* = 12.4, 6.1 Hz, 1H), 3.76 (s, 3H), 3.73-3.69 (m, 1H), 3.68 (d, *J* = 7.7 Hz, 1H), 3.61-3.56 (m, 1H), 3.53-3.49 (m, 2H), 3.38 (s, 3H), 3.19 (s, 3H), 2.87 (dd, *J* = 16.5, 9.3 Hz, 1H), 2.76 (dq, *J* = 6.9, 6.9 Hz, 1H), 2.35 (dd, *J* = 16.5, 3.3 Hz, 1H), 1.55 (s, 6H), 1.08 (d, *J* = 7.1 Hz, 3H), 0.88 (s, 9H), 0.043 (s, 3H), 0.041 (s, 3H);(m, 5H), 4.97 (dd, *J* = 10.3, 2.9 Hz, 1H), 4.60 (ABq, J_{ab} = 6.8 Hz, $\Delta\nu$ = 18.9 Hz, 2H), 4.12 (ddq, *J* = 10.3, 6.4, 4.4 Hz, 1H), 3.99-3.94 (m, 1H), 3.61-3.55 (m, 3H), 3.42 (s, 3H), 1.87 (ddd, *J* = 10.3, 1.9, 0.97 Hz, 1H), 1.67 (ddd, *J* = 13.2, 10.3, 2.4 Hz, 1H), 1.21 (d, *J* = 6.4 Hz, 3H); 125 MHz ^{13}C NMR (CDCl$_3$) δ 209.7, 159.4, 135.8, 134.1, 130.5, 129.6, 129.2, 126.5, 114.0, 92.7, 86.7, 76.3, 72.0, 72.0, 67.1, 66.0, 59.9, 59.2, 56.8, 55.4, 50.7, 46.6, 26.1, 18.5, 12.4, 12.1, 11.9, -5.0, -5.0; IR (CHCl$_3$) 3012, 1713 cm^{-1}; Anal. Calcd for C$_{33}$H$_{56}$O$_8$Si: C, 65.10; H, 9.27. Found: C, 64.85; H, 9.30.

General Experimental for Procedure A



Preparation of (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3, 5, 9-trimethyl-11-(1, 1, 2, 2-tetramethyl-1-silapropoxy)undeca-2, 9-dien-6-ol (8c). To a stirring solution of (2E, 9E)(8S, 4R, 5R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-one (0.10 g, 0.16 mmol) in 1.6 mL of THF at rt was added methanol (0.14 mL, 0.11 g, 3.3 mmol), followed by the dropwise addition of a solution of SmI$_2$ (4.8 mL, 0.48 mmol, 0.10 M in THF). The resulting mixture was stirred for 24 h before the septum was removed and stirring was continued until the solution color changed from blue to yellow. The solution was then quenched with a saturated aqueous solution of $Na_2S_2O_3$ (10 mL) and diluted with EtOAc (50 mL). The layers were separated and the organic layer was washed with a saturated aqueous $Na_2S_2O_3$ (2 × 10 mL). The combined aqueous layers were then extracted with EtOAc (3 × 20 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification of this material was accomplished by flash chromatography on a 1.0 × 8.0 cm column, eluting with a gradient of 50 mL each of 20%, 30%, 40%, and 60% EtOAc/hexane, collecting 4 mL fractions. The product containing fractions (15-30) were collected and concentrated under reduced pressure to give (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-ol (77 mg, 77% yield) as a colorless oil and as a 9:91 mixture of isomers, separation of syn:anti diastereomers was accomplished by preparative HPLC. Analytical data for the anti diastereomer: R_f 0.42 (30% acetone/hexane); $[\alpha]^{23}_{D}$ = -52.40 (c 2.03, $CHCl_3$); 500 MHz ^1H NMR ($CDCl_3$) δ 7.24 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.59-5.55 (m, 2H), 4.62 (d, *J* = 7.1 Hz, 1H), 4.50 (d, *J* = 6.9 Hz, 1H), 4.43 (s, 2H), 4.28 (dd, *J* = 9.9, 2.2 Hz, 1H), 4.20 (dd, *J* = 5.8, 1.4 Hz, 2H), 4.07 (d, *J* = 6.9 Hz, 2H), 3.87 (ddd, *J* = 9.3, 7.7, 7.7 Hz, 1H), 3.77 (s, 3H), 3.74 (br d, *J* = 4.1 Hz, 1H), 3.64 (br d, *J* = 5.5 Hz, 1H), 3.56-3.49 (m, 3H), 3.37 (s, 3H), 3.23 (s, 3H), 1.73 (ddd, *J* = 13.7, 10.2, 2.2 Hz, 1H), 1.67 (ddq, *J* = 6.9, 6.9, 4.1 Hz, 1H), 1.55 (s, 6H), 1.42 (ddd, *J* = 13.5, 9.4, 2.7 Hz, 1H), 0.86 (s, 9H), 0.81 (d, *J* = 7.1 Hz, 3H), 0.029 (s, 6H); 125 MHz ^{13}C NMR ($CDCl_3$) δ 159.3, 136.9, 135.4, 130.7, 129.5, 127.8, 123.3, 113.9, 92.4, 86.0, 77.7, 77.5, 72.0, 71.7, 68.9, 67.1, 66.1, 60.0, 59.2, 57.1, 55.3, 41.4, 38.8, 26.0, 13.7, 12.1, 10.4, -5.0; IR ($CHCl_3$) 3493, 3062 cm^{-1}: Anal. Calcd for $C_{33}H_{58}O_8Si$: C, 64.88; H, 9.57. Found: C, 65.02; H, 9.54. The ratio of diastereomers was determined to be 9:91 (64:36, $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 7% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 16.3 and 17.1 min, respectively.

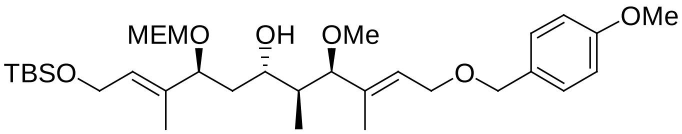
General Experimental for Procedure B



Preparation of (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3, 5, 9-trimethyl-11-(1, 1, 2, 2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-ol (8c). To stirring samarium metal (36 mg, 0.24 mmol) was added a freshly prepared solution of samarium diiodide (7.2 mL, 0.72 mmol, 0.10 M in THF) followed by a solution of (2E, 9E)(8S, 4R, 5R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-one (146 mg, 0.24 mmol) and methanol (205 mL, 154 mg, 4.8 mmol) in 2.4 mL of THF *via* cannula, the flask was then washed with THF (2 × 0.80 mL). The resulting mixture was stirred for 17 h before the septum was removed and stirring was continued until the solution color changed from blue to yellow. The solution was then quenched with a saturated aqueous solution of $Na_2S_2O_3$ (10 mL) and diluted with EtOAc (70 mL). The layers were separated and the organic layer was washed with a saturated aqueous solution of $Na_2S_2O_3$ (2 × 10 mL). The combined aqueous layers were then extracted with EtOAc (2 × 30 mL). The combined organic layers were dried over

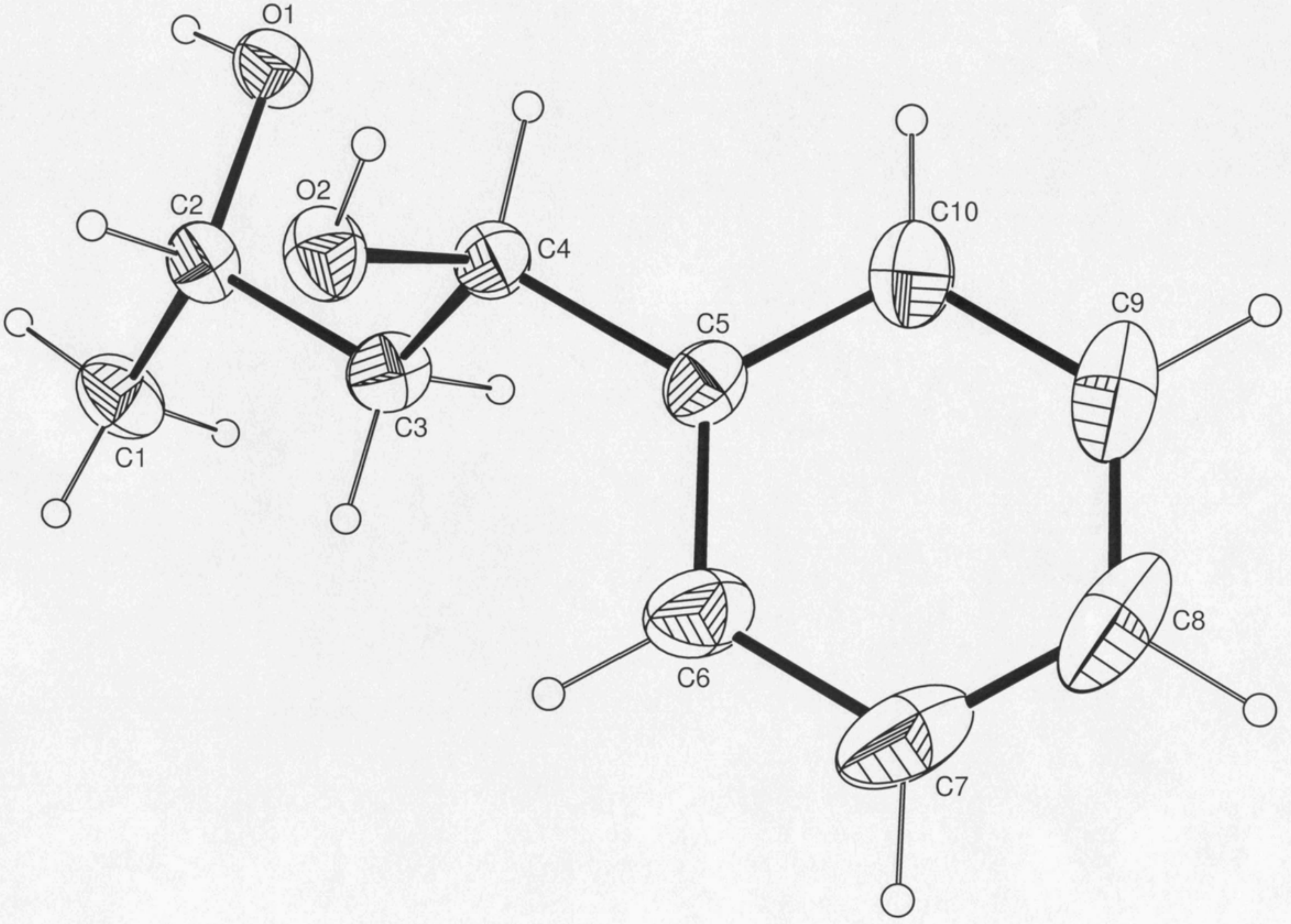
$MgSO_4$, filtered, and concentrated *in vacuo*. Purification of this material was accomplished by flash chromatography on a 1.0 × 8.0 cm column, eluting with a gradient of 50 mL each of 20%, 30%, 40%, and 60% EtOAc/hexane, collecting 4 mL fractions. The product containing fractions (15-40) were collected and concentrated under reduced pressure to give (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-ol (124 mg, 85% yield) as a colorless oil and as a 9:91 mixture of isomers, separation of syn:anti diastereomers was accomplished by preparative HPLC. Analytical data for the anti diastereomer is identical to that obtained above.

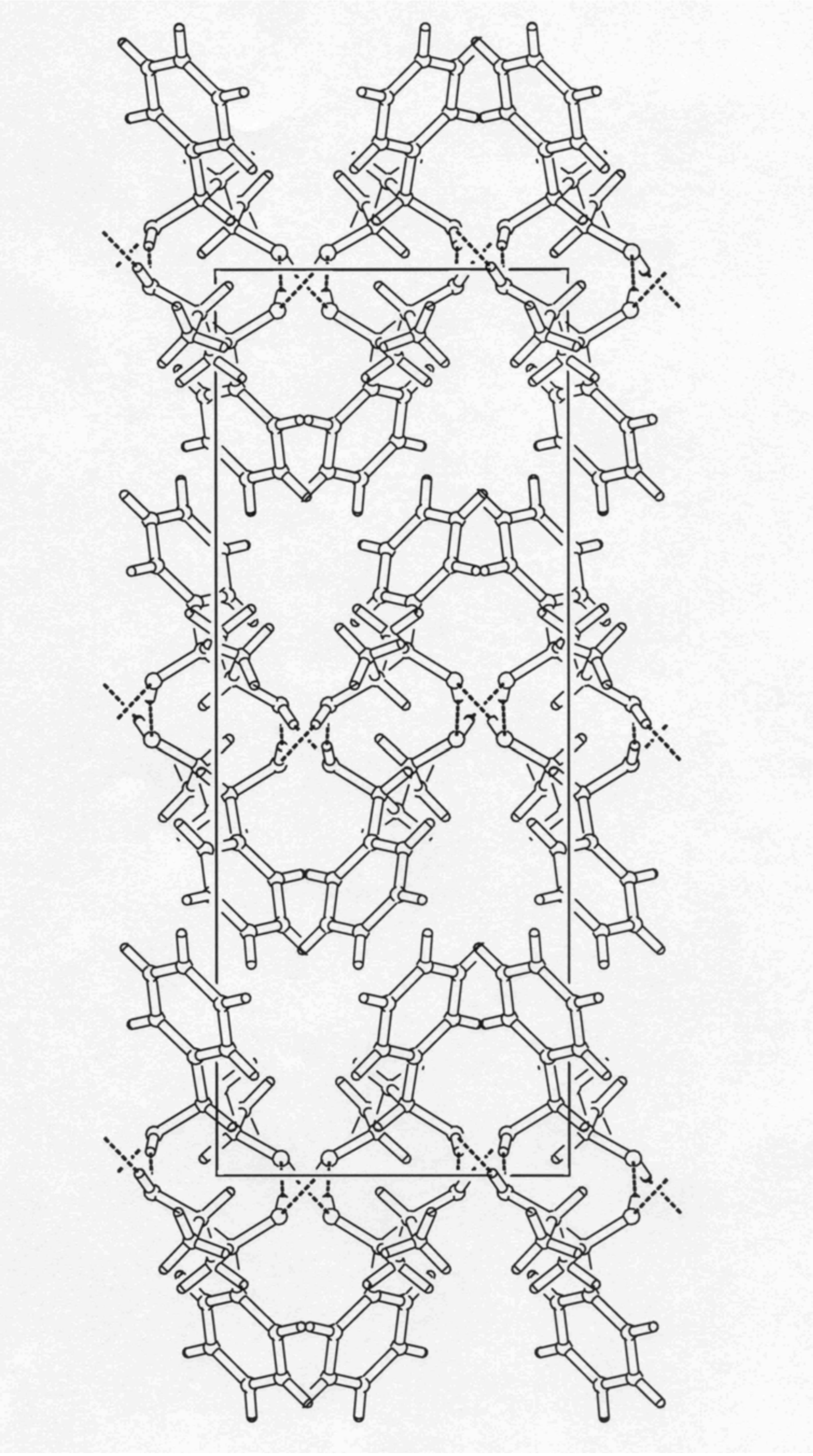
General Experimental for Procedure C



Preparation of (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-ol (8c). To a stirring suspension of samarium chips (1.90 g, 12.6 mmol) in 12.6 mL of THF was added diiodomethane (1.69 mL, 6.30 mmol) dropwise *via* syringe. The resulting mixture was stirred for 2 h before being cooled to 0 °C and then a solution of (2E, 9E)(8S, 4R, 5R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-one (1.28 g, 2.10 mmol) and methanol (1.80 mL, 1.34 g, 42.0 mmol) in 2.2 mL of THF was added *via* cannula, and the flask was washed with THF (2 × 1.0 mL). The resulting mixture was allowed to warm to rt over 4 h. This blue solution was transfered to a separatory funnel leaving samarium chips behind. The color changed from blue to yellow at which time a saturated aqueous solution of $Na_2S_2O_3$ (500 mL) was added along Et_2O (400 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2 × 400 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification of this material was accomplished by flash chromatography on a 3.5 × 24 cm column, eluting with a gradient of 200 mL each of 10%, 20%, and 30% acetone/hexane, collecting 8 mL fractions. The product containing fractions (33-55) were collected and concentrated under reduced pressure to give (2E, 9E)(5S, 6S, 8S, 4R)-4-methoxy-8-[(2-methoxyethoxy)methoxy]-1-[(4-methoxyphenyl)methoxy]-3,5,9-trimethyl-11-(1,1,2,2-tetramethyl-1-silapropoxy)undeca-2,9-dien-6-ol (1.22 g, 87% yield) as a colorless oil and as a 12:88 mixture of isomers, separation of syn:anti diastereomers was accomplished by preparative HPLC. Analytical data for the anti diastereomer is identical to that obtained above. The ratio of diastereomers was determined to be 12:88 (64:36, $NaBH_4$) by HPLC analysis using a Microsorb Si 80-125-C5 silica column, using a mobile phase of 7% *i*PrOH/hexane and a flow rate of 0.5 mL/min, which gave retention times for the minor and major diastereomers of 16.2 and 17.1 min, respectively.

ORTEP


O1
C2
O2
C10
C4
C9
C5
C1
C3
C8
C6
C7



Crystal Structure Report

Experimental:

A colorless prism shaped crystal 0.33 x 0.28 x 0.25 mm in size was mounted on a glass fiber with traces of viscous oil and then transferred to a Nonius KappaCCD diffractometer equipped with Mo $K\alpha$ radiation (λ = 0.71073 Å). Ten frames of data were collected at 200(0.1)K with an oscillation range of *1* deg/frame and an exposure time of *20* sec/frame. [REF1] Indexing and unit cell refinement based on 682 observed reflections from those ten frames, indicated a orthorhombic *P* lattice. A total of 5648 reflections (Θ_{max} = 32.6°) were indexed, integrated and corrected for Lorentz, polarization and absorption effects using DENZO-SMN and SCALEPAC. [REF 2] Post refinement of the unit cell gave *a* = 8.8295(3) Å , b = 9.0227(3) Å, c = 22.9237(8) Å, and V = 1826.24(11) Å^3. Axial photographs and systematic absences were consistent with the compound having crystallized in the orthorhombic space group *P b c a.*

The structure was solved by a direct methods using SIR 97. [REF 3]
All of the non-hydrogen atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were located and refined isotropically using SHELXL97. [REF 4] The weighting scheme employed was $w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.2392P]$ where $P = (F_o^2 + 2F_c^2)/3$. The refinement converged to R1 = 0.0505, wR2 = 0.1225, and S = 1.027 for 2294 reflections with 1> $2\sigma(I)$, and R1 = 0.0761, wR2 = 0.1375, and S = 1.027 for 3219 unique reflections and 166 parameters. [REF 5] The maximum Δ/σ in the final cycle of the least-squares was 0.001, and the residual peaks on the final difference-Fourier map ranged from -0.190 to 0.331 e/Å^3. Scattering factors were taken from the International Tables for Crystallography, Volume C. [REF 6, REF 7]

REF 1 COLLECT Data Collection Software. Nonius B.V. 1998.

REF 2 Otwinowski, Z.; Minor, W., “Processing of X-ray Diffraction Data Collected in Oscillation Mode”, Methods Enzymol. 1997, 276, 307-326.

REF 3 SIR97 (Release 1.02) - A program for automatic solution and refinement of crystal structure. A. Altomare, M.C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A.G. G. Moliteni, G. Polidori, and R. Spagna.

REF 4 SHELX97 [Includes SHELXS97, SHELXL97, CIFTAB] - Sheldrick, G. M. (1997). Programs for Crystal Structure Analysis (Release 97-2). University of Göttingen, Germany.

REF 5 $R1 = \Sigma(|| F_o | - |F_c ||) / \Sigma |F_o|$, $wR2 = [\Sigma(w(F_o^2 - F_c^2)2) / \Sigma(F_o^2)^2]^{1/2}$, and S = Goodness-of-fit on $F^2 = [\Sigma(w(F_o^2 - F_c^2)^2 / (n-p)]^{1/2}$, where n is the number of reflections and p is the number of parameters refined.

REF 6 Maslen, E. N.; Fox, A. G.; O’Keefe, M. A., International Tables for Crystallography: Mathemetical, Physical and Chemical Tables, Vol. C, Chapter 6, Wilson, A. J. C., Ed.; Kluwer, Dordrecht, The Netherlands, 1992; pp. 476-516.

REF 7 Creagh, D. C.; McdAuley, W. J., International Tables for Crystallography: mathematical, Physical and Chemical tables, Vol. C, Chapter 4 Wilson, A. J. C., Ed.; Kluwer, Dordrecht, The Netherlands, 1992; pp. 206-222.

REF8 ORTEP3 for Windows - L. J. Farrugia, *J. Appl. Crystallogr*. **1997**, *30*, 565.

REF9 WinGX A Windows Program for Crystal Structure Analysis. L. J. Farrugia, University of Glasgow, Glasgow, 1998.

Table 1. Crystal data and structure refinement for shelxl.

Identification code	shelxl	
Empirical formula	C10 H14 O2	
Formula weight	166.21	
Temperature	200(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*P b c a*	
Unit cell dimensions	a = 8.8295(3) Å	α= 90°.
	b = 9.0227(3) Å	β= 90°.
	c = 22.9237(8) Å	γ = 90°.
Volume	1826.24(11) Å^3	
Z	8	
Density (calculated)	1.209 g/cm^3	
Absorption coefficient	0.083 mm^{-1}	
F(000)	720	
Crystal size	0.33 x 0.28 x 0.25 mm^3	
Theta range for data collection	3.69 to 32.60°.	
Index ranges	-13<=h<=13, -12<=k<=12, -34<=l<=34	
Reflections collected	5648	
Independent reflections	3219 [R(int) = 0.0388]	
Completeness to theta = 32.60°	96.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9796 and 0.9732	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3219 / 0 / 166	
Goodness-of-fit on F^2	1.027	
Final R indices [I>2sigma(I)]	R1 = 0.0505, wR2 = 0.1225	
R indices (all data)	R1 = 0.0761, wR2 = 0.1375	
Extinction coefficient	0.006(5)	
Largest diff. peak and hole	0.331 and -0.190 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for shelxl. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6812(1)	-2312(1)	5187(1)	28(1)
O(2)	3243(1)	-329(1)	5456(1)	27(1)
C(1)	5728(1)	-4392(1)	5724(1)	33(1)
C(2)	5472(1)	-2845(1)	5481(1)	25(1)
C(3)	5091(1)	-1777(1)	5971(1)	27(1)
C(4)	4623(1)	-216(1)	5790(1)	23(1)
C(5)	4405(1)	767(1)	6319(1)	25(1)
C(6)	3313(2)	422(1)	6733(1)	37(1)
C(7)	3160(2)	1293(2)	7232(1)	50(1)
C(8)	4091(2)	2513(2)	7314(1)	52(1)
C(9)	5156(2)	2876(2)	6899(1)	46(1)
C(10)	5312(1)	2009(1)	6400(1)	33(1)

Table 3. Bond lengths [Å] and angles [°] for shelxl.

O(1)-C(2)	1.4444(12)
O(1)-H(1)	0.860(17)
O(2)-C(4)	1.4423(12)
O(2)-H(2)	0.872(19)
C(1)-C(2)	1.5188(15)
C(1)-H(1A)	0.994(15)
C(1)-H(1B)	0.952(17)
C(1)-H(1C)	0.983(15)
C(2)-C(3)	1.5173(15)
C(2)-H(2A)	1.001(12)
C(3)-C(4)	1.5250(14)
C(3)-H(3A)	0.980(14)
C(3)-H(3B)	1.015(13)
C(4)-C(5)	1.5141(14)
C(4)-H(4)	0.978(13)
C(5)-C(6)	1.3893(16)
C(5)-C(10)	1.3897(16)
C(6)-C(7)	1.3945(19)
C(6)-H(6)	0.992(16)
C(7)-C(8)	1.387(3)
C(7)-H(7)	0.94(2)
C(8)-C(9)	1.376(2)
C(8)-H(8)	0.968(19)
C(9)-C(10)	1.3921(18)
C(9)-H(9)	1.018(19)
C(10)-H(10)	0.954(16)
C(2)-O(1)-H(1)	109.0(11)
C(4)-O(2)-H(2)	107.6(11)
C(2)-C(1)-H(1A)	110.2(8)
C(2)-C(1)-H(1B)	108.5(9)
H(1A)-C(1)-H(1B)	107.1(12)
C(2)-C(1)-H(1C)	110.7(9)
H(1A)-C(1)-H(1C)	108.7(12)
H(1B)-C(1)-H(1C)	111.5(13)

O(1)-C(2)-C(3)	108.40(8)
O(1)-C(2)-C(1)	110.80(8)
C(3)-C(2)-C(1)	110.24(9)
O(1)-C(2)-H(2A)	106.3(7)
C(3)-C(2)-H(2A)	109.3(7)
C(1)-C(2)-H(2A)	111.6(7)
C(2)-C(3)-C(4)	116.47(9)
C(2)-C(3)-H(3A)	108.1(8)
C(4)-C(3)-H(3A)	108.4(8)
C(2)-C(3)-H(3B)	109.6(7)
C(4)-C(3)-H(3B)	108.2(7)
H(3A)-C(3)-H(3B)	105.4(10)
O(2)-C(4)-C(5)	111.05(8)
O(2)-C(4)-C(3)	107.91(8)
C(5)-C(4)-C(3)	110.97(8)
O(2)-C(4)-H(4)	108.8(7)
C(5)-C(4)-H(4)	109.4(7)
C(3)-C(4)-H(4)	108.6(7)
C(6)-C(5)-C(10)	119.21(11)
C(6)-C(5)-C(4)	120.29(10)
C(10)-C(5)-C(4)	120.49(10)
C(5)-C(6)-C(7)	120.16(13)
C(5)-C(6)-H(6)	120.1(9)
C(7)-C(6)-H(6)	119.7(9)
C(8)-C(7)-C(6)	120.04(14)
C(8)-C(7)-H(7)	121.7(12)
C(6)-C(7)-H(7)	118.3(12)
C(9)-C(8)-C(7)	120.01(12)
C(9)-C(8)-H(8)	120.1(11)
C(7)-C(8)-H(8)	119.9(11)
C(8)-C(9)-C(10)	120.10(13)
C(8)-C(9)-H(9)	119.7(10)
C(10)-C(9)-H(9)	120.2(10)
C(5)-C(10)-C(9)	120.44(13)
C(5)-C(10)-H(10)	118.6(9)
C(9)-C(10)-H(10)	121.0(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for shelxl. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[$h^2a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	28(1)	22(1)	35(1)	-2(1)	7(1)	0(1)
O(2)	28(1)	24(1)	29(1)	1(1)	-4(1)	0(1)
C(1)	31(1)	23(1)	45(1)	2(1)	5(1)	0(1)
C(2)	22(1)	22(1)	31(1)	-2(1)	1(1)	1(1)
C(3)	29(1)	25(1)	27(1)	1(1)	2(1)	5(1)
C(4)	23(1)	23(1)	23(1)	-1(1)	2(1)	0(1)
C(5)	27(1)	25(1)	24(1)	-2(1)	0(1)	6(1)
C(6)	43(1)	35(1)	35(1)	1(1)	13(1)	7(1)
C(7)	69(1)	50(1)	31(1)	4(1)	17(1)	24(1)
C(8)	80(1)	48(1)	27(1)	-11(1)	-9(1)	31(1)
C(9)	57(1)	36(1)	44(1)	-14(1)	-19(1)	10(1)
C(10)	33(1)	29(1)	36(1)	-5(1)	-4(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for shelxl.

	x	y	z	U(eq)
H(1)	7182(19)	-3016(19)	4978(8)	52(5)
H(2)	3133(19)	500(20)	5263(8)	57(5)
H(1A)	6082(18)	-5067(16)	5410(7)	42(4)
H(1B)	4780(20)	-4767(17)	5860(7)	48(4)
H(1C)	6490(17)	-4375(16)	6036(6)	38(4)
H(2A)	4639(14)	-2832(14)	5185(5)	25(3)
H(3A)	4270(16)	-2211(14)	6202(6)	33(3)
H(3B)	5985(16)	-1695(14)	6247(5)	30(3)
H(4)	5415(15)	200(13)	5541(5)	24(3)
H(6)	2626(19)	-433(17)	6672(7)	47(4)
H(7)	2430(20)	1020(20)	7510(8)	67(5)
H(8)	4020(20)	3090(20)	7669(8)	69(5)
H(9)	5830(20)	3780(20)	6958(8)	63(5)
H(10)	6044(19)	2248(16)	6109(7)	45(4)

Table 6. Torsion angles [°] for shelxl.

O(1)-C(2)-C(3)-C(4)	65.44(11)
C(1)-C(2)-C(3)-C(4)	-173.12(9)
C(2)-C(3)-C(4)-O(2)	62.95(11)
C(2)-C(3)-C(4)-C(5)	-175.17(8)
O(2)-C(4)-C(5)-C(6)	58.55(13)
C(3)-C(4)-C(5)-C(6)	-61.48(13)
O(2)-C(4)-C(5)-C(10)	-122.57(10)
C(3)-C(4)-C(5)-C(10)	117.40(11)
C(10)-C(5)-C(6)-C(7)	-1.71(18)
C(4)-C(5)-C(6)-C(7)	177.18(11)
C(5)-C(6)-C(7)-C(8)	0.5(2)
C(6)-C(7)-C(8)-C(9)	0.7(2)
C(7)-C(8)-C(9)-C(10)	-0.7(2)
C(6)-C(5)-C(10)-C(9)	1.74(17)
C(4)-C(5)-C(10)-C(9)	-177.15(10)
C(8)-C(9)-C(10)-C(5)	-0.53(19)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(2)#1	0.860(17)	2.024(18)	2.8802(11)	174.0(16)
O(2)-H(2)...O(1)#2	0.872(19)	1.937(19)	2.8028(11)	172.0(16)

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y-1/2,-z+1 #2 -x+1,-y,-z+1

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[ii] Stork, G.; Kraus, G. A.; Garcia, G. A. *J. Org. Chem.* **1974,** *39*, 3459.