

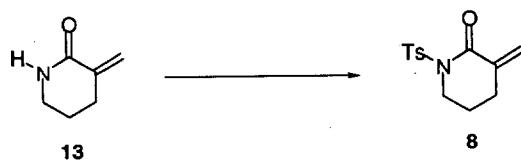
**General Procedures.** All non-aqueous reactions were carried out under nitrogen atmosphere in oven-dried (120°C) glassware. Tetrahydrofuran (THF) and diethyl ether (Et<sub>2</sub>O) were distilled immediately prior to use from sodium metal/benzophenone ketyl. Methylene chloride (CH<sub>2</sub>Cl<sub>2</sub>, EM Science) and benzene (EM Science) were distilled from calcium hydride prior to use. Methanol (MeOH, EM Science) was distilled from magnesium methoxide. Dimethyl sulfoxide (DMSO) and N, N-dimethylformide (DMF) were distilled from calcium hydride and stored over 4Å molecular sieves. Triethylamine (Et<sub>3</sub>N, EM Science), 2,6-lutidine (Acros) and pyridine (py., EM Science) were distilled from calcium hydride immediately prior to use. The molarities indicated for organolithium reagents were established by titration with 2,6-di-tert-butyl-4-methylphenol and 1,10-phenanthroline as indicator. Dibutylboron triflate was purchased from Fluka and used as received. All other commercially obtained reagents were used as received. Rochelle's salt solution refers to 2M aqueous sodium potassium tartrate.



**Diene 7.** In a 250 mL round bottom flask was added THF (130 mL) and 2.0M sodium(bistrimethylsilyl)amide (NaHMDS) in THF (14.7 mL, 29.4 mmol). The solution was cooled to -78°C and then ketone **19** (3.27 g, 26.8 mmol) in THF (10 mL) was added over 30 min via syringe pump. After addition was complete, the solution was stirred for an additional 10 min and then TBSOTf (7.4 mL, 32.1 mmol) was added dropwise. The reaction was stirred for 30 min and quenched with pH 7 buffer (20 mL) at -78°C. After warming to ambient temperature, the aqueous layer was extracted with ether (3 X 150 mL), washed with brine, and the combined

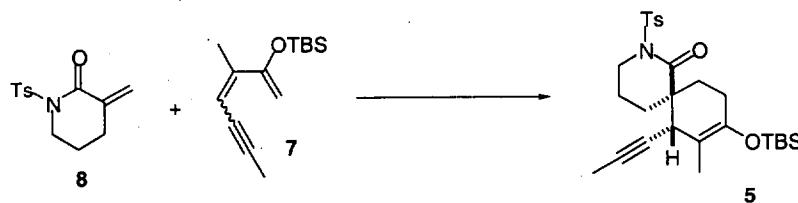
organics were dried over  $\text{Na}_2\text{SO}_4$ .  $^1\text{H}$  NMR analysis of the crude reaction mixture indicated that no olefin isomerization had occurred during the reaction. On smaller scale, rapid purification by passing through a silica gel plug with hexane as eluent could minimize isomerization. However, in this particular run, purification gave 5.22 g (82%) of diene 7 as a slightly yellow oil as a 2:1 (Z/E) ratio by  $^1\text{H}$  NMR.  $R_f = 0.71$  (1:5-EtOAc:hexanes); IR (thin film) 2957, 2930, 2858, 2223, 1579, 1251  $\text{cm}^{-1}$ ; HRMS (FAB) Calcd for  $\text{C}_{14}\text{H}_{25}\text{OSi} [\text{M}+\text{H}]$ : 237.1675. Found: 237.1656. (Z)-isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  5.41-5.36 (m, 1H), 4.88 (d,  $J = 1.2$  Hz, 1H), 4.46 (d,  $J = 0.9$  Hz, 1H), 1.95 (app dd,  $J = 0.9$  Hz, 2.4 Hz, 3H), 1.84 (app d,  $J = 0.6$  Hz, 3H), 0.93 (s, 9H), 0.17 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  155.3, 143.5, 107.2, 95.3, 90.3, 78.3, 25.7, 21.4, 18.3, 4.6, -4.6.

(E)-isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.00-5.95 (m, 1H), 4.53 (d,  $J = 1.2$  Hz, 1H), 4.35 (br s, 1H), 2.04 (d,  $J = 2.4$  Hz, 3H), 2.00 (br s, 3H),  $\delta$  0.96 (s, 9H), 0.17 (s, 6H).



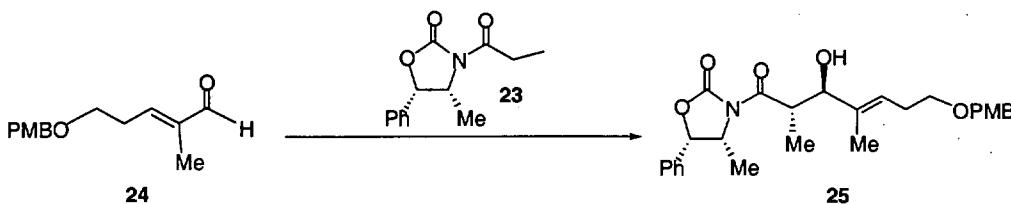
**Dienophile 8.** Lactam 13 (0.504 g, 5.44 mmol) was dissolved in THF (60 mL) in a 100 mL round bottom flask. The solution was cooled to  $-78$   $^{\circ}\text{C}$  ( $\text{CO}_2$ /acetone) and 1.0 M sodium(bistrimethylsilyl)amide (NaHMDS) in THF (5.98 mL, 5.98 mmol) was added dropwise via syringe. After 10 min, *p*-toluenesulfonyl chloride (TsCl) (1.14 g, 5.98 mmol) was added in one portion and the resulting solution was stirred for 30 min. The reaction was warmed to ambient temperature (23  $^{\circ}\text{C}$ ) and quenched with sat. aqueous sodium bicarbonate, washed with water, brine, and concentrated under reduced pressure. Flash column chromatography on silica gel (50-60%  $\text{CH}_2\text{Cl}_2$ /EtOAc) afforded 1.37 g (95%) of dienophile 8 as a colorless, crystalline solid. Large scale purification was performed by recrystallization from ethyl acetate: m.p. =

119.5-120.5 °C (ether);  $R_f$  = 0.12 (1:4-EtOAc:hexanes);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) 7.93 (d,  $J$  = 8.4 Hz, 2H), 7.32 (dd,  $J$  = 0.3, 9.0 Hz, 2H), 6.26 (app q,  $J$  = 1.5 Hz, 1H), 5.42 (app q,  $J$  = 1.8 Hz, 1H) 4.01 (t,  $J$  = 5.8 Hz, 2H), 2.56 (tt,  $J$  = 1.80, 6.30 Hz, 2H), 2.43 (s, 3H), 2.01-1.92 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  163.6, 144.6, 136.9, 136.2, 129.3, 128.5, 125.8, 47.3, 29.1, 23.2, 21.6. HRMS (FAB) Calcd for  $\text{C}_{13}\text{H}_{16}\text{NO}_3\text{S}[\text{M}+\text{H}]$ : 266.0851. Found 266.0862. Anal Calcd for  $\text{C}_{13}\text{H}_{14}\text{NO}_3\text{S}$ : C, 58.85; H, 5.70. Found: C, 58.61; H, 5.72.



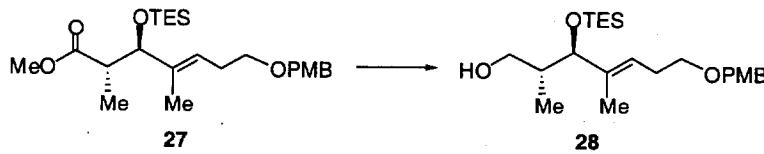
**Spirocycle 5.** In a 250 mL round bottom flask, dienophile **8** (3.29 g, 12.4 mmol) was dried azeotropically with benzene and then diluted in 147 mL  $\text{CH}_2\text{Cl}_2$ . The resulting solution was cooled to -78°C, 1.8 M  $\text{Et}_2\text{AlCl}$  in toluene (6.89 mL, 12.4 mmol) was added dropwise, and the solution was stirred for 20 min. Diene **7** (3.08 g, 13.0 mmol) was then added dropwise and the reaction was maintained at -30°C for 3 h. The reaction was quenched at -78°C with sat.  $\text{NaHCO}_3$  (45 mL) and warmed to ambient temperature. The aqueous layer was extracted with  $\text{CH}_2\text{Cl}_2$  (3X50 mL), washed with brine, and the organics were dried over  $\text{Na}_2\text{SO}_4$ . After concentrating under reduced pressure, the crude product was purified by silica gel chromatography (1:4-EtOAc:hexanes) to give 4.28 g (67%) spirocycle **5** as a light yellow solid. Recrystallization from ether gave colorless, clear crystals which were suitable for x-ray analysis. M.p. = 136.5-137.5°C(ether);  $R_f$  = 0.37 (1:5-EtOAc:hexanes); IR (KBr pellet) 2955, 2930, 2857, 1686, 1354, 1262, 1170, 1102  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (d,  $J$  = 8.4 Hz, 2H), 7.30 (d,  $J$  = 8.1 Hz, 2H), 4.13-4.03 (m, 1H), 3.80-3.70 (m, 1H), 3.67 (br s, 1H), 2.42 (s, 3H), 2.10-

1.70 (m, 8H), 1.67 (d,  $J$  = 2.4 Hz, 3H), 1.62 (app q,  $J$  = 1.5 Hz, 3H), 0.92 (s, 9H), 0.08 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  174.9, 144.1, 141.5, 136.2, 129.0, 128.3, 109.5, 79.2, 77.3, 47.3, 47.1, 40.2, 30.0, 26.3, 25.7, 25.6, 21.5, 20.5, 18.0, 14.5, 3.3, -3.95, -4.0; HRMS (FAB) Calcd for  $\text{C}_{27}\text{H}_{40}\text{NO}_4\text{SSI}$  [ $\text{M}+\text{H}$ ]: 502.2447. Found: 502.2425; Anal Calcd for  $\text{C}_{27}\text{H}_{39}\text{NO}_4\text{SSI}$ : C, 64.63; H, 7.83; N, 2.79. Found: C, 64.54; H, 8.02; N, 2.80. Note: To prevent desilylation, this silyl enol ether was best stored in base washed glassware and NMR spectra were recorded in buffered chloroform (containing 0.1%  $\text{d}^5$ -pyridine).



**Anti-Aldol Product 25.** To a cooled ( $0^\circ\text{C}$ ) solution of imide **23** (4.88 g, 21.0 mmol) in 63 mL of  $\text{Et}_2\text{O}$  was added  $\text{Bu}_2\text{BOTf}$  (42 mL, 42 mmol) followed by diisopropylethylamine (4.19 mL, 24.0 mmol). The resulting yellow slurry was stirred for 45 min then cooled to  $-78^\circ\text{C}$ . Over a 30 min period was added a  $-78^\circ\text{C}$  solution of aldehyde **24** (6.12 g, 26 mmol) in 30 mL  $\text{Et}_2\text{O}$ . The solution was stirred for 6 h and then diluted with  $\text{Et}_2\text{O}$  (30 mL). The reaction was quenched at  $-78^\circ\text{C}$  with 1M tartaric acid (60 mL), warmed to  $22^\circ\text{C}$ , and then stirred for 2 h. The reaction mixture was partitioned between  $\text{Et}_2\text{O}$  and  $\text{H}_2\text{O}$ . The aqueous layer was extracted with  $\text{Et}_2\text{O}$  (2 X 50 mL), and the combined organic layers were washed with saturated  $\text{NaHCO}_3$  (2 X 50 mL). The combined organic layers were then cooled to  $0^\circ\text{C}$  and a 3:1  $\text{MeOH}/30\% \text{H}_2\text{O}_2$  solution (60 mL) was added. After 30 min at  $22^\circ\text{C}$ , the solution was washed with saturated  $\text{NaHCO}_3$  (40 mL), brine (40 mL) and dried ( $\text{Mg}_2\text{SO}_4$ ). Concentration of the organic layer provided a yellow oil. Analysis of the crude 300 MHz  $^1\text{H}$  NMR showed a mixture of three aldol products in a ratio of  $\sim 5:1:0.5$ . Purification by flash chromatography on  $\text{SiO}_2$  eluting with hexanes:  $\text{EtOAc}(7:3)$

provided alcohol **25** (5.12 g, 68%) as a viscous, colorless oil. On standing at 22°C, the aldol adduct **25** crystallized as colorless needle-like crystals:  $R_f = 0.19$  (7:3-hexane:ethyl acetate);  $[\alpha]_D^{23} +18.5^\circ$  ( $c$  1.22,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.88 (d,  $J=6.6$  Hz, 3H), 1.04 (m,  $J=2.1, 6.6$  Hz, 3H), 1.67 (d,  $J=1.2$  Hz, 3H), 2.34 (q,  $J=6.6$  Hz, 3H), 3.43 (t,  $J=6.6$  Hz, 2H), 3.78 (s, 3H); 4.11 (m, 2H), 4.41 (s, 2H), 4.76 (m,  $J=6.6$  Hz, 1H), 5.45 (dq,  $J=7.2, 1.2$  Hz, 1H), 5.64 (d,  $J=7.2$  Hz, 1H), 6.85 (dt,  $J=8.7, 2.1$  Hz, 2H), 7.20-7.40 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.0, 14.3, 14.8, 28.4, 40.6, 55.2, 69.2, 72.5, 77.2, 78.9, 81.1, 113.7, 125.5, 125.6, 128.7, 129.1, 130.5, 133.3, 136.3, 153.4, 159.1, 176; IR (thin film) 3489, 2929, 1783, 1696, 1509  $\text{cm}^{-1}$ ; HRMS (FAB) Calcd for  $\text{C}_{27}\text{H}_{33}\text{NO}_6[\text{M}+\text{Na}]$ : 490.2206. Found: 490.2227; Anal. Calcd for  $\text{C}_{27}\text{H}_{33}\text{NO}_6$ : C, 69.36; H, 7.11; N, 2.99; Found: C, 69.30; H, 7.17; N, 2.92.



**Alcohol 28.** To a cooled (-78°C) solution of ester **27** (6.57 g, 15.05 mmol) in  $\text{CH}_2\text{Cl}_2$  (150 mL) was added 1M solution of DIBAL-H in  $\text{CH}_2\text{Cl}_2$  (33.11 mmol, 33.11 mL) slowly. The reaction was stirred at -78°C for 30 min, then quenched with 1M tartaric acid (34 mL) at -78°C slowly and warmed to 22°C. The reaction mixture was separated and the organic phase was washed with  $\text{H}_2\text{O}$  (2 X 50 mL). The aqueous phase was extracted with  $\text{Et}_2\text{O}$  (3 X 30 mL). The combined organic layers were washed with saturated  $\text{NaHCO}_3$  solution (50 mL), brine (50 mL), dried over  $\text{MgSO}_4$ , and concentrated *in vacuo*. Purification by flash chromatography on  $\text{SiO}_2$  eluting with hexanes:  $\text{EtOAc}$  (9:1 → 4:1, gradient elution) gave alcohol **28** as a colorless oil (5.74 g, 93%);  $R_f = 0.45$  (7:3-hexane:ethyl acetate);  $[\alpha]_D^{23} +3.16^\circ$  ( $c$  3.99,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.62 (q,  $J=7.8$  Hz, 6H), 0.75 (d,  $J=6.9$  Hz, 3H), 0.96 (t,  $J=7.8$  Hz, 9H), 1.62 (s,

3H), 1.81-1.94 (m, 1H), 2.37 (q,  $J=6.9$  Hz, 2H), 3.24 (s, 1H), 3.49 (t,  $J=6.9$  Hz, 2H), 3.64 (d,  $J=5.4$  Hz, 2H), 3.83 (s, 3H), 3.87 (d,  $J=8.4$  Hz, 1H), 4.46 (s, 2H), 5.40 (t,  $J=6.9$  Hz, 1H), 6.88 (dt,  $J=6.9, 3$  Hz, 2H), 7.28 (dt,  $J=6.9, 3$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  4.7, 6.7, 11.3, 14.1, 28.2, 38.1, 55.2, 67.5, 69.3, 72.5, 85.3, 113.7, 124.1, 129.1, 130.5, 137.6, 159.1; IR (thin film) 3466, 2957, 1612, 1509, 1092  $\text{cm}^{-1}$ ; HRMS (FAB) Calcd for  $\text{C}_{23}\text{H}_{40}\text{O}_4\text{Si}[\text{M}+\text{Na}]$ : 431.2594. Found: 431.2589; Anal. Calcd for  $\text{C}_{23}\text{H}_{40}\text{O}_4\text{Si}$ : C, 67.60; H, 9.86; Found: C, 67.51; H, 9.89.



**Methoxy Furanose 30.** To a solution of methoxy olefin **29** (846 mg, 1.95 mmol) in  $\text{CH}_3\text{OH}$  (20 mL) was added p-TsOH (37.08 mg, 0.19 mmol) in 1 mL  $\text{CH}_3\text{OH}$  at 0°C. The ice bath was removed and the reaction mixture was stirred at 22°C for 30 min. The reaction was quenched carefully by pouring it into aqueous  $\text{NaHCO}_3$  (40 mL). The pH was adjusted to neutral by the addition of solid  $\text{NaHCO}_3$ , and the mixture was extracted with  $\text{CH}_2\text{Cl}_2$  (3 X 45 mL). The combined organic layers were washed with brine (40 mL), dried ( $\text{Na}_2\text{SO}_4$ ), filtered, and concentrated in vacuo. Purification by flash chromatography on  $\text{SiO}_2$  eluting with hexanes:EtOAc (9:1) yielded 583 mg (86%) of furanose **30** as a colorless oil and as a mixture (1.5:1) of diastereomers:  $R_f = 0.35$  (4:1-hexane:ethyl acetate); IR (thin film) 2953, 1612, 1512, 1454, 1249  $\text{cm}^{-1}$ ; MS (FAB) Calcd for  $\text{C}_{19}\text{H}_{28}\text{O}_4[\text{M}+\text{Na}]$ : 343. Found: 343.

Major isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.97 (d,  $J=6.6$  Hz, 3H), 1.67 (d,  $J=1.2$  Hz, 3H), 1.61-1.71 (m, 1H), 2.09 (dd,  $J=6.6, 12.6$  Hz, 1H), 2.19-2.31 (m, 1H), 2.42 (q,  $J=6.9$  Hz, 2H), 3.39 (s, 3H), 3.50 (dt,  $J=1.2, 6.9$  Hz, 2H), 3.83 (s, 3H), 3.85 (d,  $J=8.7$  Hz, 1H), 4.48 (s, 2H), 4.94 (d,  $J=4.8$  Hz, 1H), 5.45 (dt,  $J=6.8, 1.2$  Hz, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.28 (dt,  $J=8.7, 3$  Hz,

2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  10.8, 15.7, 28.7, 34.1, 41.7, 54.5, 55.3, 69.4, 72.5, 93.6, 104.3, 113.7, 125.0, 129.2, 130.6, 135.5, 159.1.

Minor isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.02 (d,  $J=6.9$  Hz, 3H), 1.49-1.58 (m, 1H), 1.64 (d,  $J=1.2$  Hz, 3H), 1.90-2.08 (m, 1H), 2.34-2.45 (m, 1H), 2.42 (q,  $J=6.9$  Hz, 2H), 3.40 (s, 3H), 3.50 (dt,  $J=1.2, 6.9$  Hz, 2H), 3.84 (s, 3H), 3.93 (d,  $J=8.7$  Hz, 1H), 4.48 (s, 2H), 5.06 (dd,  $J=3.0, 5.4$  Hz, 1H), 5.50 (dt,  $J=6.8, 1.2$  Hz, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.28 (dt,  $J=8.7, 3$  Hz, 2H).



**Trisubstituted Tetrahydrofuran 33.** To a cooled (0°C) solution of methoxy furanose **30** (171.9 mg, 0.537 mmol) and allyltrimethyl silane (256  $\mu$ l, 1.61 mmol) in 5 mL  $\text{CH}_2\text{Cl}_2$  was added  $\text{BF}_3\text{OEt}_2$  (74.7  $\mu$ l, 0.591 mmol) dropwise. After stirring for 3 h, the reaction was quenched at 0°C by addition of saturated  $\text{NaHCO}_3$  (2 mL) and then warmed to ambient temperature. The layers were separated and the aqueous layer was extracted with  $\text{Et}_2\text{O}$  (3 X 10 mL). The combined organic layers were washed with brine (10 mL), dried over  $\text{MgSO}_4$ , and concentrated *in vacuo*. Purification of the yellow oil by flash chromatography on  $\text{SiO}_2$  eluting with hexanes:EtOAc (9:1) gave 168 mg (95%) of inseparable diastereomeric tetrahydrofurans **33** (4:1;  $\alpha:\beta$ ) as a colorless oil:  $R_f$  = 0.26 (9:1-hexane:ethyl acetate); IR (thin film) 3075, 2957, 1612, 1512, 1037  $\text{cm}^{-1}$ ; HRMS (FAB) Calcd for  $\text{C}_{21}\text{H}_{30}\text{O}_3[\text{M}+\text{Na}]$ : 353.2093. Found: 353.2077.

Major isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.97 (d,  $J=6.9$  Hz, 3H), 1.67 (s, 3H), 1.60-1.73 (m, 1H), 1.81-1.89 (m, 1H), 1.96-2.44 (m, 4H), 2.40 (q,  $J=7.5$  Hz, 2H), 3.48 (dt,  $J=1.2, 7.5$  Hz, 2H), 3.69 (d,  $J=8.1$  Hz, 1H), 3.83 (s, 3H), 4.02-4.14 (m, 1H), 4.47 (s, 2H), 5.06-5.14 (m, 2H), 5.46 (t,  $J=6.9$  Hz, 1H), 5.77-5.93 (m, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.29 (dt,  $J=8.7, 3$  Hz, 2H);  $^{13}\text{C}$

<sup>13</sup>NMR (75 MHz, CDCl<sub>3</sub>) δ 11.3, 16.9, 28.5, 35.9, 39.0, 40.8, 55.2, 69.5, 72.5, 77.0, 92.3, 113.7, 116.7, 123.9, 129.2, 130.6, 134.9, 135.7, 159.1.

Minor isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.97 (d,  $J=6.9$  Hz, 3H), 1.65 (s, 3H), 1.60-1.73 (m, 1H), 1.81-1.89 (m, 1H), 1.96-2.44 (m, 4H), 2.40 (q,  $J=7.5$  Hz, 2H), 3.48 (dt,  $J=1.2, 7.5$  Hz, 2H), 3.78 (d,  $J=9.0$  Hz, 1H), 3.83 (s, 3H), 4.02-4.14 (m, 1H), 4.47 (s, 2H), 5.06-5.14 (m, 2H), 5.42 (t,  $J=6.9$  Hz, 1H), 5.77-5.93 (m, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.29 (dt,  $J=8.7, 3$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.1, 16.1, 28.5, 37.8, 40.6, 40.9, 55.2, 69.5, 72.5, 78.5, 91.5, 113.7, 116.7, 123.9, 129.2, 130.6, 135.1, 135.7, 159.1.



**TIPS Ether 4.** To a cold (-78°C) solution of alcohol **34** (33.8 mg, 0.1 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (1 mL) was added freshly distilled 2,6-lutidine (34 µl, 0.29 mmol) followed by TIPSOTf (28.7 µl, 0.11 mmol). The reaction mixture was stirred at -78°C for 40 min and then quenched by addition of saturated NaHCO<sub>3</sub> solution (1 mL). The layers were separated and the aqueous phase was extracted with Et<sub>2</sub>O (2 X 5 mL). The combined organic layers were washed with H<sub>2</sub>O (2 mL) and brine (2 mL), dried over MgSO<sub>4</sub>, and concentrated in vacuo. Purification by flash chromatography on SiO<sub>2</sub> eluting with hexanes:EtOAc (9:1) furnished a mixture (4:1; α:β) of silyl ethers **4** (48 mg, 99%) as a colorless oil: R<sub>f</sub> = 0.33 (9:1-hexane:ethyl acetate); IR (thin film) 2938, 1505, 1098, 1033 cm<sup>-1</sup>; HRMS (FAB) Calcd for C<sub>30</sub>H<sub>52</sub>O<sub>4</sub>Si[M+H]: 505.3713. Found 505.3700.

Major isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.97 (d,  $J=6.6$  Hz, 3H), 1.08 (s, 21H), 1.64 (s, 3H), 1.50-1.85 (m, 6H), 1.96-2.12 (m, 1H), 2.40 (q,  $J=7.5$  Hz, 2H), 3.48 (dt,  $J=1.2, 7.5$  Hz, 2H), 3.69

(d,  $J=8.4$  Hz, 1H), 3.73 (t,  $J=6.0$  Hz, 2H), 3.83 (s, 3H), 3.96-4.07 (m, 1H), 4.47 (s, 2H), 5.45 (t,  $J=7.2$  Hz, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.29 (dt,  $J=8.7, 3$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.3, 12.0, 17.1, 18.0, 28.5, 29.4, 32.7, 36.0, 39.8, 55.2, 63.4, 69.5, 72.5, 77.6, 92.2, 113.7, 123.7, 129.2, 130.6, 135.9, 159.1.

Minor isomer:  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  0.98 (d,  $J=6.3$  Hz, 3H), 1.08 (s, 2H), 1.64 (s, 3H), 1.50-1.85 (m, 6H), 2.12-2.24 (m, 1H), 2.40 (q,  $J=7.5$  Hz, 2H), 3.48 (dt,  $J=1.2, 7.5$  Hz, 2H), 3.80 (d,  $J=9.6$  Hz, 1H), 3.73 (t,  $J=6.0$  Hz, 2H), 3.83 (s, 3H), 3.96-4.07 (m, 1H), 4.47 (s, 2H), 5.41 (t,  $J=7.2$  Hz, 1H), 6.90 (dt,  $J=8.7, 3$  Hz, 2H), 7.29 (dt,  $J=8.7, 3$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  11.2, 12.4, 16.2, 17.7, 28.5, 29.5, 32.7, 37.8, 41.5, 55.2, 63.4, 69.5, 72.5, 79.3, 91.3, 113.7, 123.7, 129.2, 130.6, 135.9, 159.1.

