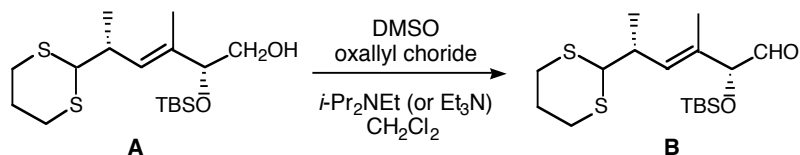


## Swern Oxidation Procedure



**Preparation of (+)-B.** A solution of oxalyl chloride (130  $\mu\text{L}$ , 1.49 mmol) in dichloromethane (5 mL) was cooled to  $-78^\circ\text{C}$ . Following the dropwise addition of a solution of dimethylsulfoxide (211  $\mu\text{L}$ , 2.98 mmol) in 1 mL dichloromethane, the reaction mixture was stirred for 5 min. A solution of alcohol **A** (448 mg, 1.24 mmol) in 1.5 mL dichloromethane was then added to the reaction mixture. After 5 min of stirring, diisopropylethylamine (1.1 mL, 6.20 mmol) was added, and the reaction mixture was warmed to ambient temperature. The reaction mixture was then diluted with diethyl ether and water and the layers were separated. The organic layer was washed with water, brine, dried over  $\text{MgSO}_4$ , filtered, and concentrated in vacuo. Flash chromatography with 10:1 hexanes:ethyl acetate as eluant gave **B** (413 mg, 92%) as a white solid: mp  $61.0\text{--}62.5^\circ\text{C}$ ;  $[\alpha]_{\text{D}}^{23} +60.9^\circ$  ( $c$  0.42,  $\text{CHCl}_3$ ); IR ( $\text{CHCl}_3$ ) 2960 (s), 2940 (s), 2860 (s), 1735 (s), 1450 (m), 1380 (m), 1250 (m), 1110 (s), 910 (m), 835 (s)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.36 (d,  $J = 1.6$  Hz, 1 H), 5.65 (d,  $J = 9.9$  Hz, 1 H), 4.31 (t,  $J = 1.4$  Hz, 1 H), 4.03 (d,  $J = 6.5$  Hz, 1 H), 2.86–2.81 (m, 5 H), 2.10–2.07 (m, 1 H), 1.83–1.80 (m, 1 H), 1.61 (d,  $J = 1.4$  Hz, 3 H), 1.15 (d,  $J = 6.8$  Hz, 3 H), 0.92 (s, 9 H), 0.08 (s, 3 H), 0.08 (s, 3H);  $^{13}\text{C}$  NMR (62.5 MHz,  $\text{CDCl}_3$ )  $\delta$  200.0, 131.5, 131.3, 82.9, 54.4, 37.4, 30.7, 30.6, 26.1, 25.7, 18.2, 12.8,  $-4.8$ ,  $-5.1$ ; TLC  $R_f = 0.50$ , **SM**  $R_f = 0.39$  (4:1 hexanes:ethyl acetate); high-resolution mass spectrum (CI,  $\text{NH}_3$ )  $m/z$  361.1685  $[(\text{M}+\text{H})^+]$ ; calcd for  $\text{C}_{17}\text{H}_{33}\text{O}_2\text{S}_2\text{Si}$ : 361.1691].

Anal. Calcd for  $\text{C}_{17}\text{H}_{32}\text{O}_2\text{S}_2\text{Si}$ : C, 56.62; H, 8.94. Found: C, 56.48; H, 9.39.

See: a) Mancuso, A.J.; Huang, S.-L.; Swern, D. *J. Org. Chem.* **1978**, *43*, 2480.  
 b) Mancuso, A.J.; Swern, D. *Synthesis* **1981**, 165.