

**General Procedure for Indium-Promoted Allylation.** 2,3-Butanedione (**1**) (439  $\mu$ L, 5.0 mmol) was dissolved in 65 mL of THF-water (4:1) and vigorously stirred while indium powder (1.53 g, 2.7 equiv) and then allyl bromide (1.73 mL, 4.0 equiv) were introduced. The reaction mixture was stirred for 3h during which time a milky appearance developed and the metal separated as pellets. At this point, 5 mL of 2N potassium hydrogen sulfate and 75 mL of chloroform were added. The separated aqueous phase was extracted with chloroform (2 x 75 mL) and the combined organic layers were dried and concentrated to leave a residue that was chromatographed on silica gel with 25% ether in ligroin as eluent to give 740 mg (87%) of diol **2** as a 3:2 mixture of diastereomers:<sup>4</sup> <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  6.04-5.89 (m, 1 H), 5.19-5.09 (m, 2 H), 2.54-2.42 (m, 1 H), 2.24-2.17 (m, 1 H), 2.06 (br s, 1 H), 1.19 (s, 0.35 X 3 H), 1.16 (s, 0.65 X 3 H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  134.5\*, 134.4, 118.7\*, 118.5, 76.2, 76.1\*, 41.1\*, 40.9, 21.8, 21.5\* (\* = major diastereomer).

**General Procedure for Ring Closing Metathesis.** The dienediol **2** (340 mg, 2.0 mmol) was placed in a septum-capped recovery flask under argon and dissolved in CH<sub>2</sub>Cl<sub>2</sub> (30 mL). A solution of the Grubbs catalyst in CH<sub>2</sub>Cl<sub>2</sub> (82 mg/30 mL) was transferred via cannula into the reaction mixture. A second septum was placed atop the existing one and secured with parafilm. The reaction mixture was stirred at 50 °C for 3 h, allowed to cool, and opened to the atmosphere for 2 h. After solvent evaporation, the residue was chromatographed on silica gel (elution with 40% ethyl acetate in ligroin) to furnish 201 mg (71%) of **3**; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  5.58 (s, 1 H), 2.32-2.26 (m, 1 H), 2.19-2.10 (m, 1 H), 1.93 (br s, 1 H), 1.24 (s, 0.76 X 3 H), 1.21 (s, 0.24 X 3 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  125.6\*, 125.2, 78.8, 73.6\*, 39.3, 39.2\*, 23.1, 22.8\* (\* = major diastereomer).

**General Procedure for One-Pot Ring Closing Metathesis/Oxidative Cleavage.** The first stage of the process was carried out exactly as described above. The cooled reaction mixture was treated portionwise with lead tetraacetate (892 mg, 2.0 mmol), stirred for 20 min at room temperature, and concentrated in vacuo. The residue was taken up in ether (50 mL) and the solids were removed by filtration through a short pad of silica gel. The pad was rinsed with ether (20 mL)

and the filtrate was washed with dilute  $\text{NaHCO}_3$  solution and water, dried, and concentrated. The residue was chromatographed on silica gel (elution with 40% ethyl acetate in ligroin). There was isolated 185 mg (66%) of **4**; IR (neat,  $\text{cm}^{-1}$ ) 1714;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  5.77 (t,  $J = 5.5$  Hz, 1 H), 3.15 (d,  $J = 5.5$  Hz, 2 H), 2.13 (s, 3 H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.4, 124.3, 42.0, 29.3; MS (EI)  $m/z$  [ $\text{M}^+$ ] calcd for  $\text{C}_8\text{H}_{12}\text{O}_2$  140.0837, found 140.0836.

**General Procedure for Diol Cleavage.** To a solution of lead tetraacetate (446 mg, 1.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (7 mL) was added diol **5** (44 mg, 1.0 mmol) dissolved in  $\text{CH}_2\text{Cl}_2$  (3 mL). After 30 min of stirring at 20 °C, the reaction mixture was concentrated, taken up in ether and further processed in the prescribed manner. There was obtained 114 mg (80%) of **6**<sup>10</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.45-2.40 (m, 2 H), 2.11 (s, 3 H), 1.57-1.52 (m, 2 H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  208.6, 43.4, 29.9, 23.1.