Olefin Cleavage under Osmylation Conditions

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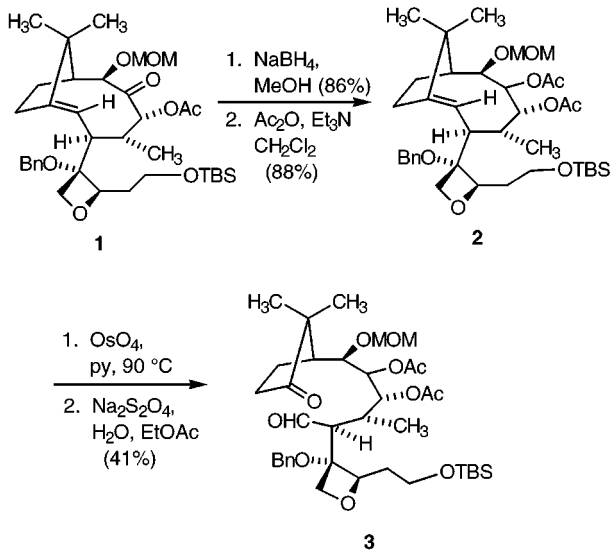
The reliability of osmium tetraoxide for accomplishing the cis-dihydroxylation of alkenes is unrivaled.[1−3] Furthermore, the ability to accomplish cyclic osmate(VI) ester formation in a diastereoselective[4,5] and enantioselective manner[6] has provided considerable impetus to the widespread application of this reagent in synthesis. If desired, higher level oxidation to give carbonyl products is customarily accomplished by the coaddition of oxidants such as sodium periodate[7,8] or hydrogen peroxide,[9] but not otherwise.

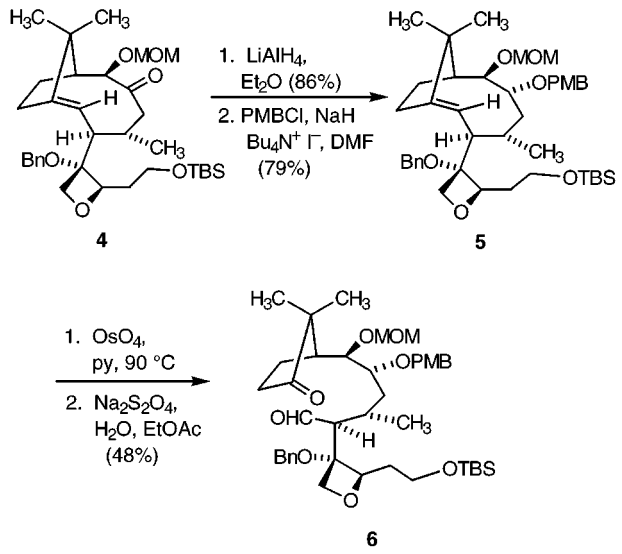
During the course of a study designed to explore the reversibility of anionic oxy-Cope rearrangements in the context of taxoid synthesis, we developed routes to the bridgehead olefinic ketones **1** and **4**.[10] In pursuit of certain objectives, subsequent occasion arose to bring their derivatives **2** and **5** into reaction with osmium tetraoxide. Both systems react sluggishly with this reagent, thereby requiring somewhat more forcing conditions than is customary. We report herein that both reactions lead to the respective keto aldehyde following appropriate reduction. To our knowledge, no observation of comparable cleavage capability with OsO_4 has previously been detailed.

The ready reduction of **1** with sodium borohydride in methanol is shown in Scheme 1. Diol production in this manner proceeded with excellent stereoselectivity to produce a single diastereomer. Neither its high-field ^{1}H NMR spectrum nor that of the derived diacetate **2** provided unequivocal confirmation of the configuration set at the new stereogenic center, and this issue was therefore not pursued. This difficulty stems in part from complexities arising from slow atropisomeric equilibration on the NMR time scale near room temperature. For example, **2** exists as a 1:1 mixture of two conformational isomers under conventional operating conditions. Saponification of **2** with aqueous sodium hydroxide in methanol returned uniquely the original diol.

Scheme 1



Scheme 2



When preliminary attempts to dihydroxylate the bridgehead olefinic bond in **2** with osmium tetraoxide under catalytic conditions gave little evidence of reaction, recourse was eventually made to somewhat greater than stoichiometric quantities of the reagent in pyridine solution at 90 °C. When this measure was taken, **2** was completely consumed within 2 h. However, subsequent decomposition of the osmate complex with sodium dithionite or hydrogen sulfide produced not the expected diol but keto aldehyde **3** instead. The diagnostic spectral features of **3** include, but are hardly limited to, an aldehyde proton signal at δ 9.74 (d, $J = 2.6$ Hz) and two carbonyl carbons at 222.3 and 201.9 ppm.

Comparable behavior was exhibited by **5** (Scheme 2). This aspect of the investigation was made possible by the smooth conversion of **4**[10] into the α-alcohol by reduction with lithium aluminum hydride in ether and subsequent formation of the *p*-methoxybenzyl ether. In this instance,

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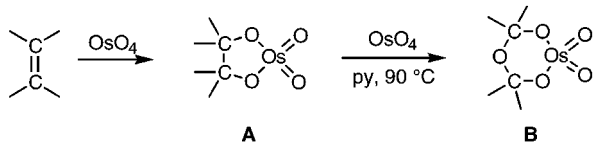
stereochemistry was directly revealed by NOE difference experiments. Suggested by this stereochemical outcome is the preferred adoption by **4** of a "carbonyl-down" conformation, with the hydride reagent then attacking from the less hindered π-surface.

When **5** was subjected to analogous osmylating conditions, comparable conversion to **6** was similarly noted. On this basis, it would seem possible that the elevated temperatures utilized promote reaction beyond the usual formation of **A**. Cleavage of the C−C bond with insertion



A **B**

of an oxygen atom as depicted in **B** would set the stage for direct keto aldehyde production during reductive workup. Our attempts to obtain crystalline samples of the osmate intermediates for X-ray crystallographic analysis were to no avail. Notwithstanding, these results provide unambiguous evidence that olefinic cleavage can indeed occur during osmylation.

Experimental Section

(1*S*,2*R*,4*R*,5*R*,6*S*,7*E*)-6-[(2*R*,3*S*)-3-(Benzyloxy)-2-[2-(*tert*-butyldimethylsiloxy)ethyl]-3-oxetanyl]-2-(methoxymethoxy)-5,11,11-trimethylbicyclo[6.2.1]undec-7-ene-3,4-diol Diacetate (2). To a solution of **1**[10] (60 mg, 0.10 mmol) in methanol (2 mL) at 0 °C was added sodium borohydride (19 mg, 0.50 mmol). The mixture was allowed to warm slowly to room temperature, stirred for 2 h, quenched with saturated $NaHCO_3$ solution, and diluted with ether. The separated aqueous layer was extracted further with ether (×2). The combined organic layers were dried, filtered, and evaporated, followed by chromatography on silica gel (elution with 1:1 hexanes−ethyl acetate). There was isolated 50 mg (83%) of the diol as a colorless oil: IR (film, cm^{-1}) 3483; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48−7.22 (m, 5 H), 5.83 (d, J = 11.3 Hz, 1 H), 5.04−4.95 (m, 3 H), 4.76−4.63 (m, 4 H), 4.50 (s, 1 H), 3.88 (t, J = 3.0 Hz, 1 H), 3.81 (d, J = 5.0 Hz, 1 H), 3.75−3.58 (m, 2 H), 3.52 (dd, J = 9.5, 3.0 Hz, 1 H), 3.47 (s, 3 H), 3.07 (d, J = 9.6 Hz, 1 H), 2.92 (dd, J = 11.5, 7.5 Hz, 1 H), 2.49−2.40 (m, 2 H), 2.24−1.90 (m, 6 H), 1.29 (s, 3 H), 1.07 (s, 3 H), 1.05 (d, J = 7.4 Hz, 3 H), 0.86 (s, 9 H), −0.01 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 140.6, 139.7, 128.3 (2 C), 127.0, 126.7 (2 C), 121.4, 98.2, 96.7, 87.4, 86.8, 83.5, 77.0, 70.1, 65.6, 59.1, 56.2, 52.0, 48.2, 45.4, 38.3, 35.1, 27.5, 25.9 (3 C), 24.6, 22.9, 21.6, 20.4, 18.3, −5.32, −5.34; FAB MS m/z (M^+ + H) calcd 605.21, obsd 605.26; $[\alpha]^{21}_D$ −103 (c 0.84, $CHCl_3$).

A solution of this diol (50 mg, 0.083 mmol) in CH_2Cl_2 (2 mL) was treated with triethylamine (0.092 mL, 0.66 mmol), acetic anhydride (0.039 mL, 0.41 mmol), and a catalytic amount of 4-(*N*,*N*-dimethylamino)pyridine. After being stirred at room temperature for 3 h, the mixture was quenched with saturated $NaHCO_3$ solution and diluted with ether. The separated aqueous layer was further extracted with ether (×2), and the combined organic extracts were dried and filtered. Removal of the solvents from the filtrate in vacuo followed by column chromatography on silica gel (elution with 3:1 hexanes−ethyl acetate) gave an inseparable pair of conformers of **2** (50 mg, 88%) as a colorless oil: IR (film, cm^{-1}) 1733; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39−7.25 (m, 10 H), 5.68 (d, J = 10.6 Hz, 1 H), 5.37 (d, J = 10.8 Hz, 1 H), 5.15 (t, J = 3.0 Hz, 1 H), 5.10−4.89 (m, 10 H), 4.80−4.67 (m, 4 H), 4.48 (s, 2 H), 4.46 (s, 2 H), 3.76−3.58 (m, 6 H), 3.31 (s, 3 H), 3.30 (s, 3 H), 3.02 (dd, J = 11.4, 6.6 Hz, 1 H), 2.93 (t, J = 10.5 Hz, 1 H), 2.85−2.55 (m, 2 H), 2.30−2.09 (m, 5 H), 2.07 (s, 3 H), 2.03−2.06 (m, 2 H), 2.02 (s, 3 H), 2.01−1.89 (m, 4 H), 1.81 (s, 3 H), 1.67 (s, 3 H), 1.65−1.58 (m, 2 H), 1.35 (s, 3 H), 1.31 (s, 3 H), 1.09 (s, 3 H), 1.07 (s, 3 H), 1.00 (d, J = 7.4 Hz, 3 H), 0.88 (s, 9 H), 0.87 (s, 9 H), 0.63 (d, J = 7.1 Hz, 3 H), 0.04 (s, 6 H), 0.03 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 171.1, 170.2, 170.0, 169.7, 144.8, 143.3, 139.2, 128.4 (2 C), 128.3 (2 C), 127.5 (4 C), 127.3, 126.9 (2 C), 120.1, 118.2, 99.9, 95.6, 95.3, 86.9, 86.5, 85.8, 83.5, 83.2, 81.2, 79.0, 77.8, 75.4, 74.9, 73.8, 71.9, 65.32, 65.27, 58.9 (2 C), 55.7, 55.4, 52.9, 51.4, 48.5, 47.1, 45.6, 44.4, 38.4, 34.9, 34.6, 34.2, 27.3, 27.1, 25.9 (6 C), 25.2, 24.9, 24.5, 22.2, 21.7, 21.3, 21.21, 21.17, 20.6 (2 C), 20.4, 18.3, 13.8, −5.19, −5.26 (2 C), −5.31; FAB MS m/z (M^+ + H) calcd 689.41, obsd 689.33.

(α*S*,2*R*,3*S*)-3-(Benzyloxy)-2-[2-(*tert*-butyldimethylsiloxy)ethyl]-α-[1*R*,2*R*,4*R*)-4-[(*S*)-2,2-dimethyl-3-oxocyclopentyl]-2,3-dihydroxy-4-(methoxymethoxy)-1-methylbutyl]-3-oxetaneacetaldehyde Diacetate (3). To a solution of **2** (10 mg, 0.017 mmol) in pyridine (0.10 mL) was added osmium tetraoxide (6.5 mg, 0.026 mmol) in pyridine (0.10 mL). The mixture was heated at 90 °C for 2 h, and the solvent was removed in vacuo. Column chromatography of the residue on silica gel (elution with 2:3 hexanes−ethyl acetate) gave a dipyridylosmium complex as a brown colored oil: IR (film, cm^{-1}) 1739; ^1H NMR (300 MHz, $CDCl_3$) δ 8.75−8.73 (m, 4 H), 7.85−7.70 (m, 2 H), 7.45−7.15 (m, 9 H), 5.67 (d, J = 3.5 Hz, 1H), 5.28 (dd, J = 12.2, 3.3 Hz, 1 H), 5.07 (d, J = 8.5 Hz, 1 H), 4.93 (d, J = 12.3 Hz, 1 H), 4.86 (d, J = 12.3 Hz, 1 H), 4.79 (d, J = 6.7 Hz, 1 H), 4.70 (d, J = 8.3 Hz, 1 H), 4.64−4.59 (m, 2 H), 4.48 (dd, J = 9.9, 2.9 Hz, 1 H), 4.05 (d, J = 8.5 Hz, 1 H), 3.70−3.63 (m, 1 H), 3.44 (s, 3 H), 2.80−2.67 (m, 3 H), 2.44−2.21 (m, 2 H), 2.17−1.90 (m, 4 H), 2.05 (s, 3 H), 2.01 (s, 3 H), 1.41 (s, 3 H), 1.39 (s, 3 H), 0.89 (d, J = 7.3 Hz, 3 H), 0.82 (s, 9 H), −0.026 (s, 3 H), −0.030 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 170.9, 169.6, 149.6 (4 C), 140.3, 139.5 (2 C), 128.1 (4 C), 126.6 (2 C), 126.4, 124.6 (2 C), 100.0, 96.1, 92.9, 90.0, 86.5, 82.9, 75.5, 73.8, 71.6, 63.4, 60.2, 55.5, 54.8, 50.0, 42.6, 36.6, 35.8, 30.6, 30.1, 26.1 (3 C), 23.4, 21.5, 21.0, 20.7, 18.5, 11.4, −5.09, −5.12.

The brown colored oil from above was dissolved in ethyl acetate (2 mL), and 20% aqueous sodium dithionite (2 mL) was added. The mixture was stirred at room temperature for 2 h, water and ether were added, and the separated aqueous layer was extracted with ether (×2), dried, and filtered. Concentration of the filtrate followed by chromatography on silica gel (elution with 2:1 hexanes−ethyl acetate) gave **3** (5.0 mg, 41%) as a colorless oil: IR (film, cm^{-1}) 1741; ^1H NMR (300 MHz, $CDCl_3$) δ 9.74 (d, J = 2.6 Hz, 1 H), 7.38−7.27 (m, 5 H), 5.37 (dd, J = 8.1, 1.6 Hz, 1 H), 5.28 (dd, J = 8.1, 1.3 Hz, 1 H), 5.18 (dd, J = 10.8, 2.3 Hz, 1 H), 5.05 (d, J = 12.1 Hz, 1 H), 4.94 (d, J = 12.1 Hz, 1 H), 4.79 (d, J = 8.3 Hz, 1 H), 4.71 (d, J = 7.1 Hz, 1 H), 4.58 (d, J = 8.5 Hz, 1 H), 4.51 (d, J = 7.1 Hz, 1 H), 3.77−3.63 (m, 2 H), 3.33 (s, 3 H), 2.87 (dd, J = 8.5, 2.6 Hz, 1 H), 2.63−2.56 (m, 1 H), 2.39−2.29 (m, 1 H), 2.08 (s, 3 H), 2.07 (s, 3 H), 2.17−1.81 (m, 7 H), 1.06 (s, 3 H), 0.93−0.86 (m, 15 H), 0.03 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 222.3, 201.9, 169.92, 169.88, 138.3, 128.7 (2 C), 127.6, 126.8 (2 C), 96.6, 85.6, 83.6, 76.2, 73.0, 72.0, 65.5, 58.2, 56.8, 56.7, 47.8, 47.3, 35.8, 35.7, 32.0, 25.93, 25.89 (4 C), 24.5, 21.0, 20.9, 18.4, 18.3, 11.7, −5.4, −5.5; FAB MS m/z (M^+ + H) calcd 721.40, obsd 721.28; $[\alpha]^{21}_D$ +84 (c 0.08, $CHCl_3$).

2-[(2*R*,3*S*)-3-(Benzyloxy)-3-[(1*E*,3*R*,4*S*,6*R*,7*R*,8*S*)-6-[(*p*-methoxybenzyl)oxy]-7-(methoxymethoxy)-4,11,11-trimethylbicyclo[6.2.1]undec-1-en-3-yl]-2-oxetanyl]ethoxy]-*tert*-butyldimethylsilane (5). To a solution of **4**[10] (61 mg, 0.10 mmol) in ether (0.5 mL) at 0 °C was added lithium aluminum hydride (12 mg, 0.32 mmol). The cooling bath was removed, and the mixture was allowed to stir at room temperature for 3 h, cooled to 0 °C, and quenched with saturated NH_4Cl solution. Water and ether were added, and the separated aqueous layer was extracted with ether (×2). The combined organic extracts were dried and filtered. Removal of solvents from the filtrate in vacuo followed by chromatography on silica gel (elution with 2:1 hexanes−ethyl acetate) gave a single diastereoisomeric alcohol (53 mg, 86%) as a colorless oil: IR (film, cm^{-1}) 3480; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41−7.28 (m, 5 H), 5.16 (d, J = 11.3 Hz, 1 H), 5.05−4.96 (m, 3 H), 4.81 and 4.77 (ABq, J = 7.8 Hz, 2 H), 4.64 and 4.61 (ABq, J = 6.7 Hz, 2 H), 3.77−3.60 (m, 2 H), 3.47 (d, J = 4.9 Hz, 1 H), 3.39 (s, 3 H), 3.33−3.29 (m, 1 H), 2.93 (d, J = 9.8 Hz, 1 H), 2.80 (dd, J = 11.6, 7.7 Hz, 1 H), 2.49−2.41 (m, 1 H), 2.36−2.23 (m, 2 H), 2.22−1.95 (m, 5 H), 1.38−1.30 (m, 1 H), 1.28 (s, 3 H), 1.12 (d, J = 7.0 Hz, 3 H), 1.05 (s, 3 H), 1.03−0.90 (m, 1 H), 0.86 (s, 9 H), 0.02 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 143.4, 139.7, 128.3 (2 C), 127.1, 126.6 (2 C), 121.8, 98.3, 95.0, 86.6, 83.8, 76.8, 68.3, 65.4, 59.0, 55.7, 52.8, 47.7, 46.0,

41.2, 35.2, 30.9, 27.3, 25.9 (3 C), 25.8, 23.0, 22.9, 21.4, 18.3, −5.32, −5.35; FAB MS *m/z* ($M^+ + H$) calcd 589.39, obsd 589.18; $[\alpha]^{23}_D$ −121 (*c* 1.29, $CHCl_3$).

A suspension of sodium hydride (4.0 mg, 0.16 mmol) in THF (1 mL) cooled to 0 °C was treated with a solution of the above alcohol (48 mg, 0.082 mmol) in DMF (0.5 mL). The mixture was stirred at 0 °C for 15 min whereupon a mixture of *p*-methoxybenzyl chloride (26 mg, 0.16 mmol) and a catalytic amount of tetra-*n*-butylammonium iodide in DMF (0.5 mL) were introduced. The mixture was warmed to room temperature, stirred for another 2 h, returned to 0 °C, and quenched with saturated NH_4Cl solution. Water and ether were added, and the separated aqueous layer was extracted with ether (×2). The combined organic extracts were dried and filtered. Solvent evaporation followed by column chromatography on silica gel (elution with 5:1 hexanes−ether) afforded **5** (46 mg, 79%) as a colorless oil: IR (film, cm^{-1}) 1613; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41−7.22 (m, 7 H), 6.88−6.83 (m, 2 H), 5.20 (d, J = 11.6 Hz, 1 H), 5.05−4.95 (m, 3 H), 4.78 (s, 2 H), 4.68 and 4.66 (ABq, J = 6.5 Hz, 1H), 4.49 (d, J = 10.2 Hz, 1 H), 4.19 (d, J = 10.1 Hz, 1 H), 3.91 (d, J = 4.4 Hz, 1 H), 3.79 (s, 3 H), 3.78−3.61 (m, 2 H), 3.35 (s, 3 H), 3.08 (d, J = 3.6 Hz, 1 H), 2.76 (dd, J = 11.6, 8.0 Hz, 1 H), 2.55−2.12 (m, 6 H), 2.03−1.97 (m, 2 H), 1.34 (s, 3 H), 1.34−1.24 (m, 2 H), 1.08 (s, 3 H), 1.02 (d, J = 7.0 Hz, 3 H), 0.87 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 159.1, 143.3, 139.6, 130.6, 129.5 (2 C), 128.3 (2 C), 127.1, 126.6 (2 C), 122.0, 113.7 (2 C), 95.7, 86.6, 83.7, 82.0, 77.7, 76.9, 69.8, 65.3, 59.0, 55.3, 55.2, 51.3, 48.0, 46.1, 37.6, 35.2, 31.0, 27.4, 26.0, 25.9 (3 C), 23.1, 22.7, 21.5, 18.3, −5.3, −5.4; FAB MS *m/z* ($M^+ + H$) calcd 709.45, obsd 709.15; $[\alpha]^{23}_D$ −110 (*c* 1.09, $CHCl_3$).

(α*S*,2*R*,3*S*)-3-(Benzyloxy)-2-[2-(*tert*-butyldimethylsiloxy-)ethyl]-α-[1*S*,3*R*,4*S*)-4-[(*S*)-2,2-dimethyl-3-oxocyclopentyl]-3-[(*p*-methoxybenzyl)oxy]-4-(methoxymethoxy)-1-methylbutyl]-3-oxetaneacetaldehyde Diacetate (6). A solution of **5** (20 mg, 0.028 mmol) in pyridine (0.10 mL) was treated with osmium tetroxide (0.10 mL of 0.33 M in pyridine, 0.034 mmol). The mixture was heated at 90 °C for 2 h, the solvent was evaporated, and the residue was subjected to chromatography on silica gel (elution with 1:1 hexanes−ethyl acetate) to leave a dipyridylosmium complex as a brown colored oil: IR (film, cm^{-1}) 2953, 2930, 2855, 1608, 1512, 1450, 1250, 1049, 1068, 1034, 982, 830; ^1H NMR (300 MHz, $CDCl_3$) δ 8.77−8.66 (m, 4 H), 7.77−7.22 (m, 2 H), 7.45−7.15 (m, 11 H), 6.93−6.88 (m, 2 H), 4.95−4.75 (m, 4 H), 4.72−4.70 (m, 2 H), 4.60−4.43 (m, 4 H), 4.15 (d, J = 8.5 Hz, 1 H), 3.88−3.82 (m, 1 H), 3.80 (s, 3 H), 3.66−3.56 (m, 1 H), 3.43 (s, 3 H), 3.41−3.35 (m, 1 H), 2.76 (t, J = 8.8 Hz, 1 H), 2.52 (d, J = 10.6 Hz, 1 H), 2.26−2.02 (m, 3 H), 1.87−1.54 (m, 4 H), 1.37 (s, 3 H), 1.36 (s, 3 H), 1.32−1.21 (m, 2 H), 0.94 (d, J = 7.3 Hz, 3 H), 0.83 (s, 9 H), −0.02 (s, 3 H), −0.04 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 159.2, 149.6 (4 C), 140.4, 139.4 (2 C), 130.7, 129.3 (2 C), 128.0 (4 C), 126.5 (2 C), 126.4, 124.5 (2 C), 113.9 (2 C), 100.3, 96.1, 93.4, 90.2, 86.7, 83.4, 75.9, 75.8, 70.0, 63.4, 60.1, 55.5, 55.2, 53.5, 50.1, 43.1, 38.4, 36.7, 36.0, 30.6, 26.1 (3 C), 25.7, 23.5, 21.3, 18.5, 18.4, −5.1, −5.2.

The brown colored oil was dissolved in ethyl acetate (2 mL), and 20% sodium dithionite solution (2 mL) was added. The mixture was stirred at room temperature for 2 h, water and ether were introduced, and the separated aqueous layer was extracted with ether (×2), dried, and filtered. Solvent evaporation followed by chromatography on silica gel (elution with 4:1 hexanes−ethyl acetate) furnished **6** (10 mg, 48%) as a colorless oil: IR (film, cm^{-1}) 1738, 1722; ^1H NMR (300 MHz, $CDCl_3$) δ 9.74 (d, J = 3.7 Hz, 1 H), 7.33−7.26 (m, 5 H), 7.20 (d, J = 8.6 Hz, 2 H), 6.82 (d, J = 8.6 Hz, 2 H), 5.16 (dd, J = 10.2, 3.0 Hz, 1 H), 4.99 and 4.91 (ABq, J = 11.8 Hz, 2 H), 4.81 (d, J = 8.3 Hz, 1 H), 4.80 (d, J = 6.7 Hz, 1 H), 4.71 (d, J = 11.0 Hz, 1 H), 4.55 (d, J = 8.3 Hz, 1 H), 4.50 (d, J = 6.7 Hz, 1 H), 4.32 (d, J = 11.0 Hz, 1 H), 3.79 (s, 3 H), 3.74−3.55 (m, 3 H), 3.34 (s, 3 H), 3.27 (d, J = 13.7 Hz, 1 H), 2.64 (dd, J = 5.9, 3.8 Hz, 1 H), 2.42−1.75 (m, 8 H), 1.26−1.00 (m, 2 H), 0.98 (s, 3 H), 0.93 (s, 3 H), 0.87 (s, 12 H), 0.03 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 222.5, 204.1, 159.3, 138.4, 130.4, 129.4 (2 C), 128.4 (2 C), 127.5, 127.0 (2 C), 113.8 (2 C), 97.3, 85.6, 84.0, 78.6, 78.1, 75.4, 71.9, 65.4, 59.7, 58.4, 56.3, 55.3, 47.9, 47.4, 36.0, 35.6, 34.2, 27.7, 25.9 (3 C), 24.3, 22.1, 18.6, 18.4, 18.3, −5.4, −5.5; FAB MS *m/z* ($M^+ + H$) calcd 741.44, obsd 741.38; $[\alpha]^{23}_D$ +11 (*c* 0.35, $CHCl_3$).

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Supporting Information Available: Copies of the 300 MHz NMR spectra of all new compounds (6 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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