

Supporting Materials

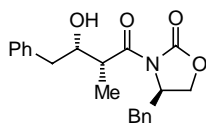
Enantiospecific Synthesis of *N*-Boc-Adda: a Linear Approach

Clay Pearson, Kenneth L. Rinehart*, Michihiro Sugano, and Jennifer R. Costerison

Roger Adams Laboratory, University of Illinois, Urbana, Illinois 61801

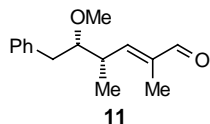
Experimental Section

General Methods. ^1H and ^{13}C NMR spectra were recorded at either 400 MHz on a Varian XL-400 instrument or at 500 MHz on a General Electric QN-500 spectrometer, with tetramethylsilane as an internal standard. Coupling constants, J_{HH} , values are given in Hz. NMR data are reported in ppm from a tetramethylsilane standard for ^1H NMR spectra and from the solvent for ^{13}C NMR. NMR data are reported as follows: chemical shift, multiplicity (br = broad, ovrlp = overlapping, s = singlet, d = doublet, t = triplet, q = quartet, qu = quintet), coupling constant, and integration results. Fast atom bombardment (FAB) mass spectra were recorded on a VG ZAB-SE mass spectrometer operating in the FAB mode. Samples were concentrated on a Buchi (Rotavapor) rotary evaporator. Melting points were taken on a Thomas-Hoover capillary melting apparatus and are uncorrected. Normal phase silica gel columns were loaded with Merck-kieselgel silica gel (230-400 mesh). All reagents and solvents were purchased from Aldrich, Sigma, or Fisher. THF was dried by distilling from Na / benzophenone. Methylene chloride and Et_3N were distilled from CaH_2 prior to use. All reactions were conducted in an inert atmosphere in oven- or flame-dried glassware. All reactions were closely monitored by TLC to ensure high yields. Solution pH measurements were made with Whatman type CF pH 0 - 14 indicator strips.



8

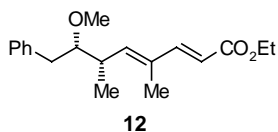
(2'R,3'S,4R)-4-Benzyl-3-[3'-hydroxy-2'-methyl-4'-phenylbutanoyl]-oxazolin-2-one (8). Acylated oxazolidinone **7** (9.9 g, 42.5 mmol) was added to 100 mL of CH₂Cl₂ and the solution was cooled to 0 °C. Freshly distilled dibutylboron triflate (12.4 mL, 48.9 mmol) was added dropwise at a rate to maintain a temperature of < 3 °C. The solution was then stirred for 5 min and Et₃N (7.4 mL, 53.1 mmol) was added dropwise during 10 min at < 3 °C then the solution was stirred at 0 °C for 45 min. The resulting solution was cooled to -78 °C, then freshly distilled phenylacetaldehyde (6.0 mL, 51.0 mmol) was added. After 1 h, the solution was allowed to warm to 0 °C and stirred for an additional 1 h prior to quenching with 100 mL of pH 7 phosphate buffer (T < 10 °C). To this solution 100 mL of 2:1 MeOH / 30% H₂O₂ was added at such a rate to keep the internal temperature < 10 °C. The resulting solution was stirred for 1 h and concentrated then extracted 4 x with 100 mL of CH₂Cl₂. The combined CH₂Cl₂ extracts were washed 2 x with 100 mL of 5% NaHCO₃ and 100 mL of brine, dried over anhydrous MgSO₄, and concentrated to an oil. The product was crystallized from 100 mL of 20% ethyl acetate in hexanes to give 11.45 g of **8** as white needles. Chromatography of the mother liquor (30% ethyl acetate in hexanes) provided an additional 3.3 g (99% yield): R_f 0.35 (6:4 hexanes:EtOAc); mp 112-112.5 °C; ¹H NMR (400 MHz, CDCl₃) δ 1.37 (d, J = 6.8 Hz, 3H), 2.81 (ovrlp, 11 lines, 4 H), 3.24 (dd, J = 13.5, 3.3 Hz, 1H), 3.82 (qd, J = 7.0, 3.4 Hz, 1H), 4.19 (ovrlp, 9 lines, 3H), 4.66 (ddt, J = 14.2, 6.6, 3.3 Hz, 1H), 7.28 (m, 10H); ¹³C NMR (100 MHz, CDCl₃) δ 11.15, 37.97, 40.70, 42.00, 55.28, 66.34, 72.93, 126.76, 127.65, 128.74, 129.19, 129.51, 129.64, 135.21, 138.49, 153.10, 177.24; MS *m/z* calcd for (M+H)⁺: 354.1705. Found 354.1709 (HRFABMS). Anal. Calcd for C₂₁H₂₃NO₄: C, 71.37; H, 6.56; N, 3.96. Found: C, 71.38; H, 6.53; N, 4.01.



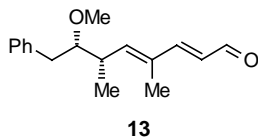
(2E,4S,5S)-5-Methoxy-2,4-dimethyl-6-phenyl-2-hexenal (11). Ester **10** (3.47 g, 12.6 mmol) was dissolved into 30 mL of dry THF and cooled to $-78\text{ }^{\circ}\text{C}$ then DIBAL-H (31.4 mL, 31.4 mmol) was added dropwise during 1 h. The solution was stirred for an additional 1 h prior to quenching with 10 mL of MeOH. A 1 M solution of tartaric acid (31.4 mL) was added at $-78\text{ }^{\circ}\text{C}$ and the solution was warmed to room temperature and stirred for 30 min. The resulting suspension was filtered through Celite and the Celite pad was washed with a copious amount of Et₂O. The organic layer was separated and washed with 100 mL of deionized water. The combined aqueous material was then extracted 4 x with 100 mL of Et₂O. The combined ether extracts were dried over anhydrous MgSO₄, concentrated, and purified by silica gel chromatography (30% ethyl acetate in hexanes) to provide 2.87 g (98%) of the alcohol (**10**) as a colorless oil: R_f 0.25 (7:3 hexanes:EtOAc); ¹H NMR (500 MHz, CDCl₃) δ 1.02 (d, $J = 6.7\text{ Hz}$, 3H), 1.29 (br t, $J = 5.8\text{ Hz}$, 1H), 1.57 (d, $J = 1.3\text{ Hz}$, 3H), 2.55 (dqu, $J = 9.8, 6.6\text{ Hz}$, 1H), 2.71 (dd, $J = 13.9, 7.5\text{ Hz}$, 1H), 2.80 (dd, $J = 13.8, 4.8\text{ Hz}$, 1H), 3.20 (ddd, $J = 7.6, 6.3, 4.8\text{ Hz}$, 1H), 3.24 (s, 3H), 3.99 (d, $J = 4.8\text{ Hz}$, 2H), 5.32 (dq, $J = 9.8, 1.3\text{ Hz}$, 1H), 7.19 (m, 3H), 7.27 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 14.01, 16.58, 36.18, 38.24, 58.67, 68.92, 87.21, 126.14, 128.37, 128.69, 129.58, 134.99, 139.77; Anal. Calcd for C₁₅H₂₂O₂: C, 76.88; H, 9.31; N, 0.0. Found: C, 76.81; H, 9.35; N, 0.0.

Fifty mL of CH₂Cl₂ was added to the alcohol (1.82 g, 7.8 mmol) and the resulting solution was cooled to $0\text{ }^{\circ}\text{C}$. Activated MnO₂ (18.2 g) was then added in several small quantities during 1 h. The mixture was stirred vigorously for 2 h at $0\text{ }^{\circ}\text{C}$ then allowed to sit in a freezer ($T = -15\text{ }^{\circ}\text{C}$) for 24 h. Aldehyde **11** was isolated by filtering the mixture through Celite and washing with copious amounts of CH₂Cl₂. The combined organic material was concentrated to an oil then subjected to silica gel chromatography (30% ethyl acetate in hexanes) which provided 1.79 g (99%) of **11** as a colorless oil that was used immediately in the next step. R_f 0.54 (7:3 hexanes:EtOAc); ¹H NMR (400 MHz, CDCl₃) δ 1.13 (d, $J = 6.8\text{ Hz}$, 3H), 1.62 (d, $J = 1.4\text{ Hz}$, 1H), 2.80 (ovrlp,

3H), 3.29 (s, 3H), 3.33 (m, 1H), 6.41 (dq, $J = 10.0, 1.3$ Hz, 1H), 7.20 (m, 3H), 7.28 (m, 2H), 9.38 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 9.51, 14.91, 37.21, 38.06, 58.78, 85.86, 126.54, 128.59, 129.52, 138.66, 138.91, 156.78, 195.60; MS m/z calcd for $(\text{M}+\text{H})^+$: 233.1544. Found 233.1542 (HRFABMS).



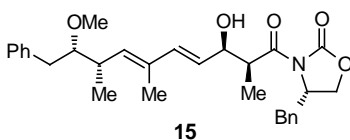
Ethyl (2E,4E,6S,7S)-7-methoxy-4,6-dimethyl-8-phenyl-2,4-octadienoate (12). (Carbethoxymethylene)-triphenylphosphorane (12.91 g, 37.1 mmol) was added to 100 mL of toluene and the mixture was heated to reflux in a round-bottomed flask equipped with a Dean-Stark trap. After 15 min, aldehyde **11** was added in 10 mL toluene and the resulting solution was stirred at reflux for 2.5 h then concentrated to an oily solid. The oily solid was dissolved in a small amount of methylene chloride and filtered through a short silica gel column. The filtrate, which contained product (by TLC), was concentrated to an oil and purified by silica gel chromatography (10% ethyl acetate in hexanes) to give 5.39 g (96%) of **12** and 170 mg (3%) of the *Z* product isomer: R_f 0.25 (9:1 hexanes:EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 1.06 (d, $J = 6.9$ Hz, 3H), 1.30 (t, $J = 7.3$ Hz, 3H), 1.66 (d, $J = 1.4$ Hz, 3H), 2.63 (dqu, $J = 10.1, 6.6$ Hz, 1H), 2.70 (dd, $J = 14.1, 7.1$ Hz, 1H), 2.80 (dd, $J = 13.9, 4.67$ Hz, 1H), 3.23 (m, 10 lines, 1H), 3.25 (s, 3H), 4.21 (q, $J = 7.1$ Hz, 2H), 5.80 (dd, $J = 15.6, 0.5$ Hz, 1H), 5.82 (d, $J = 9.9$ Hz, 1H), 7.19 (m, 3H), 7.27 (m, 2H), 7.32 (dd, $J = 15.6, 0.7$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 12.57, 14.54, 15.86, 37.28, 38.36, 58.89, 60.41, 86.67, 116.39, 126.31, 128.45, 129.61, 132.62, 139.17, 144.48, 149.74, 167.68; MS m/z calcd for $(\text{M}+\text{H})^+$: 303.1960. Found 303.1960 (HRFABMS).



(2E,4E,6S,7S)-7-Methoxy-4,6-dimethyl-8-phenyl-2,4-octadienal (13). Ester **12** (5.0 g, 16.6 mmol) was dissolved into 100 mL of dry THF and the solution was cooled to $-78\text{ }^{\circ}\text{C}$, then DIBAL-H (57.9 mL, 57.9 mmol) was added during 1 h. The solution was stirred an additional 1 h then quenched with 5 mL of MeOH. A 1 M solution of tartaric acid (57.9 mL) was added at $-78\text{ }^{\circ}\text{C}$ and then the solution was warmed to room temperature and stirred for an additional 1 h. The resulting suspension was filtered through Celite and the Celite pad was washed with a copious amount of Et₂O. The organic layer was separated and washed with 25 mL of deionized water. The combined aqueous material was then extracted 3 x with 70 mL of Et₂O. The combined ether extracts were dried over anhydrous MgSO₄, concentrated, and purified by silica gel chromatography (30% ethyl acetate in hexanes) to provide 4.27 g (99%) of the alcohol as a colorless oil: R_f 0.32 (7:3 hexanes:EtOAc); ¹H NMR (400 MHz, CDCl₃) δ 1.04 (d, J = 6.6 Hz, 3H), 1.60 (s, 1H), 1.66 (d, J = 1.7 Hz, 3H), 2.61 (dqu, J = 9.9, 6.7 Hz, 1H), 2.68 (dd, J = 14.0, 7.2 Hz, 1H), 2.81 (dd, J = 13.9, 4.6 Hz, 1H), 3.19 (ddd, J = 7.5, 6.3, 4.6 Hz, 1H), 3.24 (s, 3H), 4.21 (d, J = 6.0 Hz, 2H), 5.42 (d, J = 10.0 Hz, 1H), 5.74 (dtd, 15.6, 6.1, 0.5, 1H), 6.27 (dq, J = 7.8, 0.8, 1 H), 7.19 (m, 3H), 7.27 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 12.86, 16.37, 36.82, 38.39, 58.82, 63.97, 87.15, 126.01, 126.15, 128.35, 129.60, 132.87, 136.13, 136.78, 139.51; MS *m/z* calcd for (M+H)⁺: 261.1855. Found 261.1854 (HRFABMS).

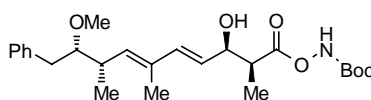
Fifty mL of CH₂Cl₂ was added to the alcohol (1.20 g, 4.62 mmol) and the resulting solution was cooled to 0 °C. Activated MnO₂ (12.0 g) was then added in several small quantities during 1 h. The mixture was vigorously stirred for 2 h at 0 °C then allowed to sit in a freezer at $-15\text{ }^{\circ}\text{C}$ for 24 Hr. Aldehyde **13** was isolated by filtering the mixture through Celite and washing with copious amounts of CH₂Cl₂. The combined organic material was concentrated to an oil then subjected to silica gel chromatography (30% ethyl acetate in hexanes), which provided 1.14 g (96%) of **13** as a colorless oil that was used immediately in the next step. R_f 0.55 (7:3

hexanes:EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 1.09 (d, $J = 6.6$ Hz, 3H), 1.70 (d, $J = 1.3$ Hz, 3H), 2.69 (m, 16 lines, 1H), 2.75 (dd, $J = 14.0, 6.9$ Hz, 1H), 2.80 (dd, $J = 14.4, 5.9$ Hz, 1H), 3.28 (m, 1H), 3.28 (s, 3H), 5.95 (dq, $J = 10.0, 0.55$ Hz, 1H), 6.11 (ddd, 15.6, 7.8, 0.5, 1H), 7.11 (dd, $J = 15.5, 0.5$, 1 H), 7.20 (m, 3H), 7.28 (m, 2H), 9.57 (d, $J = 7.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 12.57, 15.45, 37.16, 38.07, 58.69, 86.30, 126.30, 127.32, 128.41, 129.43, 133.08, 138.84, 146.74, 157.90; MS m/z calcd for $(\text{M}+\text{H})^+$: 259.1697. Found 259.1698 (HRFABMS).



(2'S,3'R,4'E,6'E,8'S,9'S,4S)-4-Benzyl-3-[3'-hydroxy-9'-methoxy-2',6',8'-trimethyl-10'-phenyl-4,6-decadienoyl]-oxazolin-2-one (15). Acylated oxazolidinone **14** (3.4 g, 14.5 mmol) was added to 75 mL of CH_2Cl_2 which was cooled to 0 °C. Freshly distilled dibutylboron triflate (4.2 mL, 16.7 mmol) was added dropwise at a rate to maintain the temperature < 3 °C, then the solution was stirred for 10 min and Et_3N (2.6 mL, 18.3 mmol) was added dropwise over 10 min ($T < 3$ °C) and the solution was stirred at 0 °C for 45 min. The resulting solution was cooled to -78 °C and aldehyde **13** (3.4 g, 13.2 mmol) in 5 mL CH_2Cl_2 was added. After 45 min, the solution was allowed to warm to 0 °C and stirred for an additional 1 h prior to quenching with 50 mL of 1:1 MeOH : pH=7 phosphate buffer ($T < 10$ °C). To this solution, was added 50 mL of 2:1 MeOH / 30% H_2O_2 at a rate to keep the internal temperature below 10 °C, then the solution was stirred at 0 °C for 1 h. The resulting solution was concentrated then extracted 4 x with 100 mL of CH_2Cl_2 . The combined CH_2Cl_2 extracts were washed with 100 mL of 5% NaHCO_3 and 100 mL of brine, dried over anhydrous MgSO_4 , and concentrated to an oil. Silica gel chromatography (30% ethyl acetate in hexanes) of the crude oil gave 5.89 g (91%) of pure **15** as an oil; R_f 0.24 (7:3 hexanes:EtOAc); ^1H NMR (500 MHz, CDCl_3) δ 1.02 (d, $J = 6.6$ Hz, 3H), 1.28 (d, $J = 7.1$ Hz, 3H), 1.65 (d, $J = 1.1$ Hz, 3H), 2.59 (dqu, $J = 9.8, 6.7$ Hz, 1H), 2.68 (dd, $J = 13.9, 7.5$

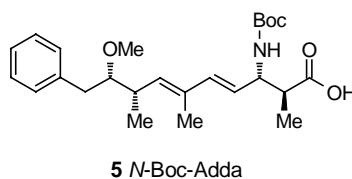
Hz, 1H), 2.80 (ovrlp, 5 lines, 3H), 3.20 (ddd, J = 7.3, 6.5, 4.5 Hz, 1H), 3.24 (s, 3H), 3.26 (dd, J = 13.8, 3.2 Hz, 1H), 3.91 (dq, J = 7.0, 3.7 Hz, 1H), 4.20 (ovrlp, 7 lines, 2H), 4.55 (br m, 1H), 4.71 (m, 8 lines, 1H), 5.43 (d, J = 9.86 Hz, 1H), 5.57 (dd, J = 15.6, 6.4 Hz, 1H), 6.32 (d, J = 15.8 Hz, 1H), 7.20 (m, 5H), 7.28 (m, 3H), 7.34 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 11.46, 12.86, 16.41, 36.88, 38.02, 38.41, 43.25, 55.36, 58.86, 66.39, 73.20, 87.11, 126.04, 126.17, 127.66, 128.39, 129.20, 129.65, 132.77, 135.24, 136.58, 136.97, 139.56, 153.35, 176.89; MS m/z calcd for $(\text{M}+\text{H})^+$: 492.2750. Found 492.2749 (HRFABMS). Anal Calcd for $\text{C}_{30}\text{H}_{37}\text{NO}_5$: C, 73.29; H, 7.59; N, 2.85. Found: C, 73.24; H, 7.60; N, 2.83.



16

(2S,3R,4E,6E,8S,9S)-1-O-(tert-Butoxycarbonyl)-3-hydroxy-9-methoxy-2,6,8-trimethyl-10-phenyl-4,6-decadienoate (16). To 3.82 g (28.7 mmol) of *tert*-butyl *N*-hydroxycarbamate was added 100 mL of THF and the solution was cooled to 0 °C. Sodium hydride (60% in oil, 1.07 g, 26.8 mmol) was added in small portions over 10 min. After 30 min, the mixture was cooled to -25 °C and **15** (4.7 g, 9.57 mmol) was added dropwise in 10 mL THF and the mixture was stirred for 2 h. The reaction was allowed to warm to 0 °C before being quenched with 100 mL of a 1 M solution of pH 4 phosphate buffer. The final pH was adjusted to 7.0 by adding a saturated solution of NaHCO_3 . The resulting solution was extracted 3 x with 100 mL ethyl acetate then the combined organic layers were washed with 100 mL of brine solution, dried over anhydrous MgSO_4 , and concentrated to an oil. The crude oil was purified by silica gel chromatography (30% ethyl acetate in hexanes) to give 4.10 g (96%) of **16** as an oil; R_f 0.32 (7:3 hexanes:EtOAc); ^1H NMR (400 MHz, CDCl_3) δ 1.03 (d, J = 6.9 Hz, 3H), 1.24 (d, J = 7.2 Hz, 3H), 1.49 (s, 9H), 1.64 (d, J = 1.3 Hz, 3H), 2.60 (dqu, J = 9.7, 6.6 Hz, 1H),

2.68 (dd, J = 13.9, 7.3 Hz, 1H), 2.81 (dd, J = 14.1, 4.4 Hz, 1H), 2.87 (dq, J = 7.1, 4.3 Hz, 1H), 3.19 (ddd, J = 7.4, 6.3, 4.6 Hz, 1H), 3.23 (s, 3H), 4.58 (dd, J = 6.0, 4.4 Hz, 1H), 5.43 (d, J = 9.8 Hz, 1H), 5.54 (dd, J = 15.1, 6.6 Hz, 1H), 6.32 (d, J = 15.5 Hz, 1H), 7.19 (m, 3H), 7.27 (m, 2H), 7.91 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 11.05, 12.87, 16.40, 28.21, 36.92, 38.45, 44.30, 58.90, 73.61, 84.02, 87.11, 125.21, 126.17, 128.37, 129.65, 132.72, 136.80, 137.53, 139.52, 156.26, 174.57; Anal. Calcd for C₂₅H₃₇NO₆: C, 67.09; H, 8.33; N, 3.13. Found: C, 66.96; H, 8.44; N, 3.09.



(2*S*,3*S*,4*E*,6*E*,8*S*,9*S*)-3-(*tert*-Butoxycarbonyl)amino-9-methoxy-2,6,8-trimethyl-10-phenyl-4,6-decadienoic acid (5), *N*-Boc-Adda. **A. SmI₂ Method. To 70 mg (0.16 mmol) of **16** were added 82.1 mg (0.31 mmol) of Ph₃P and 10 mL of THF. The solution was cooled to -45 °C, diethyl azodicarboxylate (46.8 μL, 0.30 mmol) was added and the resulting solution was stirred at -45 °C for 1 h, warmed to 0 °C and stirred for 1 h then recooled to -45 °C. The resulting solution was titrated with SmI₂ (0.1 M solution) until the blue color remained. Stirring was continued for 30 min before the solution was warmed to 0 °C and a second addition of SmI₂ was continued until the blue color remained. Water (10 mL) was added and the reaction mixture was concentrated. The pH was adjusted to 2.5 with 1 M NaHSO₄. The solution was extracted 3 x with 30 mL of ethyl acetate. The combined ethyl acetate extracts were dried over anhydrous MgSO₄ and concentrated to an oil which was purified by silica gel chromatography (4% methanol in CH₂Cl₂) to give 43 mg (64%) of **5** as an oil; R_f 0.09 (96:4 CH₂Cl₂:MeOH); ¹H NMR (400 MHz, CDCl₃) δ 1.02 (d, J = 6.6 Hz, 3H), 1.25 (d, J = 6.4 Hz, 3H), 1.45 (s, 9 H), 1.61 (s, 3H), 2.58 (m, 8 lines, 1H), 2.67 (dd, J = 13.9, 7.4 Hz, 1H), 2.74 (ovrlp, 1H), 2.79 (dd, J = 13.9, 4.5 Hz, 1H), 3.19 (m, 8 lines, 1H), 3.23 (s, 3H), 4.39 (br s, 1H), 5.27 (br s, 1H), 5.39 (d, J = 9.7 Hz, 1H),**

5.47 (dd, 14.9, 5.6 Hz, 1H), 6.20 (d, J = 15.6 Hz, 1H), 7.18 (m, 3H), 7.26 (m, 2H); MS m/z calcd for (M+H)⁺: 432.2750. Found 432.2750 (HRFABMS).⁵¹

(5). B. Sodium Naphthalide Method. Ph₃P (466mg, 1.78 mmol) and 20 mL THF were added to **16** (265 mg, 0.59 mmol) and the solution was cooled to -78 °C. Diethyl azodicarboxylate (261 μL, 1.66 mmol) was added and the solution was stirred at -78 °C for 4 h [R_f isoxazolidin-5-one 0.48 **6** (7:3 hexanes:EtOAc)]. The solution was titrated with sodium naphthalide (0.5 M solution) until the green color remained (complete by TLC). Deionized water (50 mL) was added at -78 °C, then the reaction mixture was concentrated and the pH was adjusted to 2.5 with 1 M NaHSO₄. The solution was extracted 3 x with 50 mL of CH₂Cl₂. The combined CH₂Cl₂ extracts were dried over anhydrous MgSO₄ and concentrated to an oil which was purified by silica gel chromatography (4% methanol in CH₂Cl₂) to give 164.3 mg (64%) of **5** as an oil; R_f 0.09 (96:4 CH₂Cl₂:MeOH); ¹H NMR (400 MHz, CDCl₃) δ 1.02 (d, J = 6.6 Hz, 3H), 1.25 (d, J = 6.4 Hz, 3H), 1.45 (s, 9 H), 1.61 (s, 3H), 2.58 (m, 8 lines, 1H), 2.67 (dd, J = 13.9, 7.4 Hz, 1H), 2.74 (ovrlp, 1H), 2.79 (dd, J = 13.9, 4.5 Hz, 1H), 3.19 (m, 8 lines, 1H), 3.23 (s, 3H), 4.39 (br s, 1H), 5.27 (br s, 1H), 5.39 (d, J = 9.7 Hz, 1H), 5.47 (dd, 14.9, 5.6 Hz, 1H), 6.20 (d, J = 15.6 Hz, 1H), 7.18 (m, 3H), 7.26 (m, 2H); MS m/z calcd for (M+H)⁺: 432.2750. Found 432.2750 (HRFABMS).⁵¹