Supporting Information for

Studies Toward the Tricyclic Core of Phomactin A. Synthesis of the Reduced Furanochroman Subunit

Punit P. Seth and Nancy I. Totah*

Department of Chemistry, University of Iowa, Iowa City, IA 52242

General Methods:

All air sensitive reactions were performed in flame dried glassware under an atmosphere of argon. Reaction solvents were dried over CaH_2 (acetone, dichloromethane, pyridine) or sodium/benzophenone ketyl (tetrahydrofuran) and were distilled just prior to use. All other reagents were reagent grade and were purified as necessary. Analytical thin layer chromatography was performed on EM silica gel 60F glass plates (0.25 mm). Flash column chromatography was performed using EM silica gel 60 (230-400 mesh). ^{1}H NMR spectra were recorded on Bruker AC-300 or WM-360 spectrometers. Chemical shifts are reported in ppm, downfield from tetramethylsilane using residual $CHCl_3$ as the internal standard (δ 7.27 ppm). ^{13}C NMR spectra were recorded on a Bruker WM-360 (90 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm, downfield from tetramethylsilane using residual $CHCl_3$ as the internal standard (δ 77.0 ppm). IR spectra were obtained with a Mattson Cygnus 25 instrument. Elemental Analyses were performed by Atlantic Microlab, Inc.; Norcross, GA.

Experimental Procedures:

Enone 5: A suspension of $LiAlH_4$ (0.510g, 12.8 mmol) in 47 mL THF was cooled to -78°C, and a solution of enone **3** (1.26 g, 4.25 mmol) in 18 mL THF added dropwise via cannula. The reaction mixture was allowed to warm slowly to room temperature overnight, then cooled back to 0°C and the excess $LiAlH_4$ quenched carefully by the dropwise addition of ice cold water. After H_2 evolution had ceased, 10% aqueous HCl (*ca*. 45 mL) was added, and stirring continued until the solids dissolved. The reaction mixture was then diluted with EtOAc, the layers separated, and the aqueous layer

saturated with NaCl, then extracted with additional ethyl acetate (4x). The combined organics were dried over $MgSO_4$, filtered, and the solvent removed *in vacuo*. The residue was purified by column chromatography (SiO_2; 65% EtOAc in hexanes) to afford the enone **5** as a white solid (0.616 g, 64%), along with the ether **6** (0.115 g, 12%). This latter compound (**6**) could be converted to the desired enone **5** upon treatment with 2 equiv. LDA at -78°C in THF, followed by a standard reaction workup. enone **5**: ^{1}H NMR ($CDCl_3$, 360 MHz): δ 6.86 (1H, m), 5.99 (1H, m), 4.58 (1H, br d, *J* = 9.5 Hz), 4.11-4.04 (3H, m), 3.78 (1H, d, *J* = 11.3 Hz), 3.52 (1H, br s), 2.80 (1H, m), 2.50 (1H, m), 1.87 (1H, dd, *J* = 12.7, 4.6 Hz), 1.65 (1H, t, *J* = 12.7 Hz), 1.24 (3H, s), 1.18 (3H, s). ^{13}C NMR ($CDCl_3$): δ 204.4, 146.7, 128.6, 73.4, 70.4, 69.6, 63.8, 55.1, 42.8, 31.4, 30.2, 22.6. IR (film): 3388, 3319, 2972, 2925, 1653, 1458 cm^{-1} Anal. Calcd for $C_{12}H_{18}O_4$: C, 63.70%; H, 8.02%. Found: C, 63.69%; H, 8.06%. ether **6**: ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.30 (1H, d, *J* = 9.7 Hz), 4.28 (1H, m), 3.96 (1H, dd, *J* = 9.5, 3.6 Hz), 3.72 (1H, d, *J* = 9.7 Hz), 3.53 (1H, dt, *J* = 11.8, 5.3 Hz), 2.88 (1H, d, *J* = 12.1 Hz), 2.73 (2H, m), 2.36 (1H, d, *J* = 19.1 Hz), 2.02 (1H, t, *J* = 12.4 Hz), 1.85 (1H, dd, *J* = 13.0, 5.3 Hz), 1.59 (1H, m), 1.25 (3H, s), 1.22 (3H, s). ^{13}C NMR ($CDCl_3$): δ 213.5, 74.7, 69.3, 68.6, 68.3, 67.0, 53.0, 46.1, 42.4, 35.1, 31.4, 22.7.

Enone 7: To a solution of the enone **5** (0.330 g, 1.45 mmol) in 15 mL dry acetone were added anhydrous $CuSO_4$ (0.69 g), dimethoxypropane (1.8 mL, 14.5 mmol) and camphorsulfonic acid (17 mg, 0.70 mmol). The resulting mixture was stirred for 14 h at room temperature after which time it was partitioned between CH_2Cl_2 and water, and the layers separated. The organic phase was washed with H_2O, sat. $NaHCO_3$, and brine, then dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2; 40% EtOAc in hexanes) to give the protected enone **7** (0.36 g, 89%) as a white solid. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 6.56 (1H, m), 5.92 (1H, m), 4.07 (1H, dd, *J* = 12.4, 4.4 Hz), 3.91 (1H, d, *J* = 11.5 Hz), 3.87 (1H, m), 3.86 (1H, d, *J* = 11.5 Hz), 2.42 (2H, m), 1.99 (1H, t, *J* = 12.6 Hz), 1.54 (1H, dd, *J* = 12.6, 4.4 Hz), 1.49 (3H, s), 1.43 (3H, s), 1.28 (3H, s), 1.17 (3H, s). ^{13}C NMR ($CDCl_3$): δ 197.3, 141.1, 128.5, 99.3, 74.2, 70.6, 70.2, 63.4, 49.3, 37.9, 31.6, 29.3 (2C), 23.3, 18.4. IR (film): 2973, 2932, 1677 cm^{-1} Anal. Calcd for $C_{15}H_{22}O_4$: C, 67.65%; H, 8.33%. Found: C, 67.56%; H, 8.35%.

Ketone 9: To a suspension of CuI (0.55 g, 2.9 mmol) in THF (25 mL) at -78°C was added MeLi (4.2 mL of a 1.4 M solution in Et_2O, 5.8 mmol). The reaction was warmed to -40 C over 15 min during which time a colorless solution of Me_2CuLi formed. The

resulting solution was cooled back to -78°C, then treated with chlorotrimethylsilane (0.37 mL, 2.9 mmol) and stirred for 20 min. Over this period, the cuprate solution turned cloudy. The enone **7** (0.27 g, 0.97 mmol) in THF (20 mL) was added dropwise to the cuprate solution and the reaction mixture was stirred for 30 min. The reaction was quenched at -78°C by the addition of sat. NH_4Cl solution (1 mL) and then diluted with EtOAc. The organic phase was extracted with NH_4Cl solution (pH 8-9), brine, dried ($MgSO_4$), and concentrated to provide the silyl enol ether **8** as a colorless oil (8:1 mixture of diastereomers at C7). This compound was used in the next step without further purification. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.67 (1H, m), 4.04 (1H, dd, *J* = 12.8, 3.5 Hz), 3.75 (1H, d, *J* = 10.8 Hz), 3.71 (1H, d, *J* = 10.8 Hz), 3.54 (1H, m), 2.48 (1H, m), 1.72 (2H, m), 1.47 (3H, s), 1.45 (2H, m), 1.41 (3H, s), 1.28 (3H, s), 1.20 (3H, s), 0.93 (3H, d, *J* = 6.9 Hz), 0.25 (9H, s). ^{13}C NMR ($CDCl_3$): δ 151.1, 110.1, 98.8, 73.0, 71.9, 70.4, 70.0, 41.4, 38.6, 32.6, 31.7, 29.0, 25.9, 23.8, 22.1, 19.2, 0.8 (3C).

The crude silyl enol ether **8**, prepared as described above, was dissolved in 10 mL THF and the reaction mixture cooled to 0°C. MeLi (1.0 mL of a 1.4 M solution in Et_2O, 1.4 mmol) was added, and the resulting solution warmed to room temperature with stirring. After 90 min, this mixture was cooled back to 0°C, treated with MeI (0.31 mL, 5 mmol), and stirred for an additional 2 h. The reaction mixture was then quenched by the addition of H_2O, diluted with EtOAc, and the layers separated. The organic layer was washed with H_2O and brine, then dried over $MgSO_4$ and concentrated *in vacuo*. Purification of this intermediate by column chromatography (SiO_2; 20% EtOAc in hexanes) provided the monoalkylated ketone (0.26 g, 92%) as a white solid.

To a solution of the monoalkylated ketone (0.30 g, 1.0 mmol) in 5 mL THF was added NaH (95%, 0.12 g, 5.0 mmol), and the reaction mixture warmed to reflux. After 1 h, MeI (1.25 mL, 20 mmol) was added, and the resulting mixture stirred an additional 2h at reflux, then cooled to room temperature. Excess NaH was quenched carefully by the dropwise addition of H_2O, the reaction mixture was diluted with EtOAc, and the layers were separated. The organics were then washed with 10% $Na_2S_2O_3$ (aq.) and brine, dried over $MgSO_4$ and concentrated *in vacuo*. Purification of the residue by column chromatography (SiO_2; 15% EtOAc in hexanes) provided the ketone **9** (0.26 g, 85%) as a white solid (8:1 mixture of diastereomers at C7). ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.11 (1H, d, *J* = 11.9 Hz), 3.92 (1H, dd, *J* = 12.4, 4.0 Hz), 3.77 (1H, d, *J* = 11.9 Hz), 3.67 (1H, m), 2.22 (1H, m), 1.90 (1H, t, *J* = 12.6), 1.69 (1H, dt, *J* = 14.4, 4.1 Hz), 1.56 (1H, m), 1.47 (3H, s), 1.46 (1H, dd, *J* = 12.7, 3.9 Hz), 1.42 (3H, s), 1.26 (3H, s), 1.18

(3H, s), 1.10 (3H, s), 1.06 (3H, s), 0.92 (3H, d, J = 7.0 Hz). ^{13}C NMR ($CDCl_3$): δ 211.4, 99.2, 73.6, 71.9, 71.0, 64.8, 50.8, 48.5, 37.8, 33.9, 31.9, 31.8, 29.4, 24.7, 23.4, 21.7, 18.7, 15.7. IR (film): 2973, 2874, 1703 cm^{-1} Anal. Calcd for $C_{18}H_{30}O_4$: C, 69.64%; H, 9.74%. Found: C, 69.55%; H, 9.79%.

Alcohol 10: A solution of the ketone **9** (0.092 g, 0.29 mmol) and DABCO (0.33 g, 2.96 mmol) in 8 mL THF was cooled to 0°C. MeLi (2.11 mL of a 1.4 M solution in Et_2O, 2.96 mmol) was added, then the reaction mixture was warmed to room temperature and stirred for 14 h. After this time, excess MeLi was carefully quenched by the addition of H_2O, the resulting solution was diluted with EtOAc, and the layers were separated. The organic layer was washed with H_2O and brine, dried over $MgSO_4$ and concentrated *in vacuo*. Purification of the residue by column chromatography (SiO_2; 10% EtOAc in hexanes) provided the tertiary alcohol **10** (0.080 g, 87%) as a white solid. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.39 (1H, s), 4.18 (1H, d, J = 12.2 Hz), 4.08 (1H, dd, J = 13.3, 4.9 Hz), 3.47 (1H, m), 3.41 (1H, d, J = 12.2 Hz), 2.75 (1H, t, J = 12.6 Hz), 2.01 (1H, m), 1.62 (3H, s), 1.56 (1H, dt, J = 14.6, 3.5 Hz), 1.48 (3H, s), 1.44 (3H, s), 1.41 (1H, dd, J = 12.2, 5.0 Hz), 1.36 (3H, s), 1.30 (1H, m), 1.29 (3H, s), 1.03 (3H, s), 0.97 (3H, s), 0.86 (3H, d, J = 6.9 Hz). ^{13}C NMR ($CDCl_3$): δ 99.5, 81.2, 75.5, 74.5, 74,2, 67.0, 43.5, 41.3, 38.2, 32.9, 31.2, 29.7, 28.8, 24.1, 23.8, 21.1, 20.2, 19.3, 16.4. IR (film): 3473, 2970, 2885, 1368 cm^{-1} Anal. Calcd for $C_{19}H_{34}O_4$: C, 69.90%; H, 10.50%. Found: C, 69.90%; H, 10.56%.

Diol 11: Thionyl chloride (0.16 mL, 2.15 mmol) was added to a solution of the alcohol **10** (0.14 g, 0.43 mmol) in 7 mL pyridine at 0°C. The reaction mixture was warmed to room temperature and stirred for 12 h, after which time it was carefully quenched by the addition of H_2O (0.5 mL). The reaction mixture was then diluted with EtOAc, the layers separated, and the organics washed with H_2O, 1% HCl, and brine, dried over $MgSO_4$ and concentrated *in vacuo*. The residue was dissolved in 2 mL THF and the reaction cooled to 0°C. 10% HCl (2 mL) was then added, and the resulting solution stirred at 0°C for 2 h. After this time, the reaction mixture was diluted with EtOAc. The organic phase was washed with H_2O, sat. $NaHCO_3$ and brine, then dried over $MgSO_4$ and concentrated. Purification by column chromatography (SiO_2; 40% EtOAc in hexanes) then provided the olefin **11** (0.107 g, 93%) as a white solid. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 6.06 (1H, s), 5.56 (1H, s), 4.15 (1H, dd, J = 12.4, 3.8 Hz), 4.01 (1H, dd, J = 11.1, 2.7 Hz), 3.87 (1H, s), 3.60 (1H, t, J = 11.1 Hz), 3.35 (1H, t, J = 2.8 Hz), 2.20 (1H, dd, J = 9.6, 2.9 Hz), 2.04 (1H, t, J = 12.5 Hz), 2.01 (1H, m), 1.54 (2H, m), 1.48 (1H, dd, J = 12.5, 3.9 Hz), 1.22 (3H, s), 1.20 (3H, s), 1.19 (3H, s), 1.00 (3H, s), 0.86 (3H, d,

J = 7.2 Hz). ^{13}C NMR ($CDCl_3$): δ 152.2, 116.7, 79.0, 72.7, 70.2, 69.4, 49.9, 39.6, 39.1, 33.1, 32.6, 31.6, 28.4, 25.5, 23.3, 16.6. IR (film): 3362, 2971, 2877, 1458 cm^{-1} Anal. Calcd for $C_{16}H_{28}O_3$: C, 71.60%; H, 10.52%. Found: C, 71.47%; H, 10.51%.

Epoxy Diol 12: To a solution of the olefin **11** (0.051 g, 0.19 mmol) in 3 mL CH_2Cl_2 was added mCPBA (70%, 0.096 g, 0.56 mmol), and the resulting solution stirred at room temperature for 4 h. After this time, 10% $Na_2S_2O_3$ (1 mL) and sat. $NaHCO_3$ (1 mL) were added, and stirring continued at room temperature for an additional 1 h. The layers were separated and the aqueous phase extracted with additional CH_2Cl_2. The combined organics were washed with sat. $NaHCO_3$ and brine, then dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2; 50% EtOAc in hexanes) to provide the epoxide **12** (0.049 g, 91%) as a colorless oil. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.50 (1H, d, *J* = 11.4 Hz), 4.14 (1H, ddd, *J* = 12.6, 4.8, 0.9 Hz), 3.93 (1H, d, *J* = 1.3 Hz), 3.71 (1H, d, *J* = 4.4 Hz), 3.63 (1H, t, *J* = 10.9 Hz), 3.44 (1H, d, *J* = 9.6 Hz), 3.39 (1H, t, *J* = 2.9 Hz), 3.03 (1H, d, *J* = 4.4 Hz), 2.07 (1H, m), 1.82 (1H, t, *J* = 12.8 Hz), 1.66 (1H, dd, *J* = 13.2, 4.9 Hz), 1.60 (1H, dt, *J* = 13.2, 2.7 Hz), 1.50 (1H, dt, *J* = 14.1, 3.4 Hz), 1.24 (3H, s), 1.21 (3H, s), 1.08 (3H, s), 0.88 (3H, d, *J* = 7.3 Hz), 0.78 (3H, s). ^{13}C NMR ($CDCl_3$): δ 76.0, 72.2, 72.1, 69.4, 65.2, 52.0, 44.1, 41.1, 39.0, 33.4, 32.8, 31.8, 25.6, 22.5, 19.7, 16.0. IR (film): 3445, 2972, 2932 cm^{-1}.

Ketone 13: A solution of the epoxy diol **12** (0.105 g, 0.370 mmol), Et_3N (0.1 mL, 0.7 mmol) and DMAP (4.3 mg, 0.035 mmol) in 3.5 mL DMF was cooled to 0°C. *tert*-Butyldimethylsilyl chloride (0.080 g, 0.53 mmol) was then added, the cooling bath removed and the resulting solution stirred at room temperature for 2h. After this time, the reaction mixture was diluted with CH_2Cl_2, washed sequentially with 1% HCl, sat. $NaHCO_3$ and brine, then dried over $MgSO_4$ and concentrated *in vacuo*. The crude material thus obtained was redissolved in CH_2Cl_2 (5 mL) and PCC (0.113 g, 0.530 mmol) was added. After 3 h, the reaction mixture was diluted with Et_2O (5 mL) and hexanes (5 mL), and filtered through a bed of celite. The filter cake was thoroughly washed with additional Et_2O/hexanes (1:1, 50 mL), and the combined filtrates concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2; 15% EtOAc in hexanes) to provide the ketone **13** (0.137 g, 94%) as a white solid. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.51 (1H, t, *J* = 3.1 Hz), 4.18 (1H, d, *J* = 9.5 Hz), 3.87 (1H, d, *J* = 9.5 Hz), 2.86 (1H, d, *J* = 3.7 Hz), 2.44 (1H, d, *J* = 15.9 Hz), 2.36 (1H, d, *J* = 15.9 Hz), 2.29 (1H, d, *J* = 3.7 Hz), 2.06 (1H, m), 1.78 (1H, dt, *J* = 12.3, 3.0 Hz), 1.69 (1H, dt, *J* = 14.9, 3.6 Hz), 1.26 (3H, s), 1.25 (3H, s), 0.91 (3H, d, *J* = 6.9 Hz), 0.91 (3H, s), 0.85

(9H, s), 0.77 (3H, s), 0.04 (3H, s), 0.02 (3H, s). ^{13}C NMR ($CDCl_3$): δ 206.2, 72.8, 68.5, 63.0, 60.1, 57.1, 52.0, 51.6, 38.4, 33.7, 32.3, 31.1, 26.1, 25.8 (3C), 25.4, 19.1, 18.2, 15.8, -5.6, -5.8. IR (film): 2959, 2858, 1714 cm^{-1} Anal. Calcd for $C_{22}H_{40}O_4Si$: C, 66.62%; H, 10.16%. Found: C, 66.74%; H, 10.20%.

Ketone 15: To a solution of the ketone **13** (0.030 g, 0.075 mmol) in 3 mL THF at -78°C was added KHMDS (0.45 mL of a 0.5 M solution in toluene, 0.23 mmol), and the resulting solution stirred for 45 min. Chlorotrimethylsilane (0.03 mL, 0.23 mmol) was then added, and the reaction mixture gradually warmed to 0°C over 90 min. The reaction mixture was partitioned between H_2O and CH_2Cl_2, the layers separated, and the aqueous extracted with additional CH_2Cl_2. The combined organics were washed with sat. $NaHCO_3$ and brine, dried over $MgSO_4$ and concentrated *in vacuo* to give the crude silyl enol ether **14** that was used without further purification. ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.56 (1H, s), 4.17 (1H, t, *J* = 3.0 Hz), 4.02 (1H, d, *J* = 9.3 Hz), 3.87 (1H, d, *J* = 9.3 Hz), 2.80 (1H, d, *J* = 4.8 Hz), 2.73 (1H, d, *J* = 4.8 Hz), 2.01 (1H, m), 1.75 (1H, dt, *J* = 13.6, 2.9 Hz), 1.60 (1H, m), 1.21 (3H, s), 1.21 (3H, s), 0.91 (3H, s), 0.89 (3H, d, *J* = 7.2 Hz), 0.88 (9H, s), 0.76 (3H, s), 0.20 (9H, s), 0.04 (3H, s), 0.03 (3H, s). ^{13}C NMR ($CDCl_3$): δ 149.8, 110.4, 72.0, 68.7, 63.8, 61.4, 51.7, 49.2, 38.1, 34.3, 32.7, 30.3, 26.3, 25.9 (3C), 25.7, 19.6, 18.2, 15.8, 0.2 (3C), -5.5, -5.6

The crude enol ether **14**, prepared as described above, was dissolved in 3 mL CH_2Cl_2 and the resulting solution cooled to 0°C. mCPBA (0.026 g, 0.15 mmol) was added, and the reaction mixture was warmed to room temperature and stirred for 90 min. After this time it was diluted with CH_2Cl_2, treated with 10% $Na_2S_2O_3$ (2 mL) and sat. $NaHCO_3$ (2 mL), then stirred for 30 min more. The layers were separated and the organic phase washed with sat. $NaHCO_3$ and brine, dried over $MgSO_4$, filtered, and concentrated. Column chromatography (SiO_2; 5% EtOAc in hexanes) provided the ketone **15** (0.030 g, 83%) as a colorless oil (15:1 mixture of diastereomers at C3). ^{1}H NMR ($CDCl_3$, 360 MHz): δ 4.51 (1H, t, *J* = 3.0 Hz), 4.26 (1H, d, *J* = 9.9 Hz), 3.98 (1H, s), 3.75 (1H, d, *J* = 9.9 Hz), 2.93 (1H, d, *J* = 3.5 Hz), 2.20 (1H, d, *J* = 3.5 Hz), 2.13 (1H, m), 1.79 (1H, dt, *J* = 12.6, 3.4 Hz), 1.67 (1H, dt, *J* = 14.9, 3.1 Hz),1.28 (3H, s), 1.12 (3H, s), 0.93 (3H, s), 0.92 (3H, d, *J* = 6.8 Hz), 0.84 (9H, s), 0.77 (3H, s), 0.14 (9H, s), 0.07 (3H, s), 0.04 (3H, s). ^{13}C NMR ($CDCl_3$): δ 203.6, 81.3, 77.7, 68.2, 61.7, 61.1, 58.1, 52.6, 38.3, 33.4, 32.3, 28.5, 25.8 (3C), 25.4, 19.0, 18.8, 18.2, 15.9, 0.7 (3C), -5.5, -5.8. IR (film): 2956, 2857, 1719 cm^{-1} Anal. Calcd for $C_{25}H_{48}O_5Si_2$: C, 61.94%; H, 9.98%. Found: C, 62.02%; H, 10.02%.

Dihydrofuran 2: To a solution of the ketone **15** (24.0 mg, 0.05 mmol) in 2.5 mL THF at 0°C was added *n*Bu_4NF (0.2 mL of a 1M solution in THF, 0.2 mmol) The reaction mixture was stirred for 15 min, after which time it was diluted with H_2O and extracted with EtOAc. The combined organics were washed with brine, then dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (SiO_2; 50% EtOAc in hexanes) to give the hemiketal **2** (12.4 mg, 95%) as a white solid. ^1H NMR ($CDCl_3$, 360 MHz): δ 4.83 (1H, dd, *J* = 12.9, 2.7 Hz), 4.45 (1H, dd, *J* = 12.9, 1.0 Hz), 4.28 (1H, m), 3.99 (1H, s), 3.49 (1H, d, *J* = 5.9 Hz), 3.14 (1H, d, *J* = 5.9 Hz), 1.84 (1H, m), 1.72 (2H, m), 1.31 (3H, s), 1.28 (3H, s), 1.09 (3H, s), 0.96 (3H, s), 0.94 (3H, d, *J* = 6.8 Hz). ^{13}C NMR ($CDCl_3$): δ 145.2, 129.0, 107.0, 79.7, 75.9, 72.1, 61.7, 36.7, 34.2, 33.8, 28.9, 26.6, 21.9, 17.6, 14.8. IR (film): 3467, 3306, 2967, 2924, 1460 cm^{-1} Anal. Calcd for $C_{15}H_{24}O_4$: C, 67.14%; H, 9.01%. Found: C, 67.06%; H, 9.11%.