Supporting Information For:

Anionic Two-Carbon Ring Expansions of Oxabicyclo[2.2.1]heptenes and Oxabicyclo[4.2.1]nonenes

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Experimental procedures for compounds **2**, **3**, **7**, and **8**.

Experimental

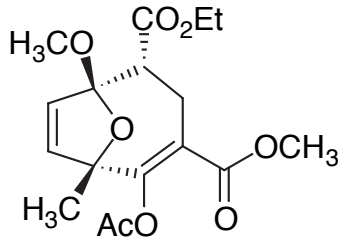
General Information.

NMR spectra were recorded on either a Bruker EM-500, Bruker AM-250, or a Varian 300 spectrophotometer. Chemical shifts were reported in δ, parts per million (ppm), relative to chloroform (δ = 7.24 ppm) as an internal standard. Coupling constants, *J*, were reported in Hertz (Hz) and refer to apparent peak multiplicities and not true coupling constants. Mass spectra were recorded at the Mass Spectrometry Facility at the Department of Chemistry of the University of Arizona on a Jeol HX-110A and are reported as % relative intensity to the molecular base peak. IR spectra were recorded on a Nicolet Impact 410. Ether, THF, hexanes, benzene, and toluene were distilled from sodium/benzophenone. CH_2Cl_2, $CHCl_3$, TMEDA, $(i\text{-Pr})_2NEt$, Et_3N, and Et_2NH were distilled from CaH_2. All other reagents were used without purification. Unless otherwise stated, all reactions were run under an atmosphere of argon in flame-dried glassware. Concentration refers to removal of solvent under reduced pressure (house vacuum at ca. 20 mm Hg) with a Büchi Rotavapor.

Representative Procedure for the Tandem Michael Addition-Anionic Fragmentation Reaction. The Generation of Oxabicyclo[4.2.1]nonenes 2 and 3 (R = OCH_3, R' = H): A solution of keto-ester **1** (0.11 g, 0.50 mmol), DMF (1.8 mL), and NaH (0.012 g, 0.50 mmol) was allowed to stir for 0.7 h at 0°C. To this was added a solution of methyl acrylate (0.07 mL, 0.75 mmol) and DMF (0.7 mL) over 1 h via syringe pump. After stirring for an additional 2 h while warming to rt, the reaction mixture was poured into pH 7.5 phosphate buffer (2 mL). The mixture was extracted with ether (3 X 10 mL) , dried ($MgSO_4$), and concentrated. Flash chromatography (3:1 hexanes:ethyl acetate)

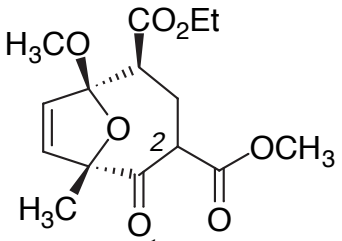
provided 85 mg (55%) of **2** (R = OCH_3, R' = H) along with 26 mg (15%) of **3** (R = OCH_3, R' = H) as colorless oils.

To simplify its characterization, **2** (1:1 mixture of C-2 isomers) was converted into the corresponding enol acetate. To a solution of **2** (R = OCH_3, R' = H) (0.085 g, 0.27 mmol), THF (1.5 ml), and KOt-Bu (0.032 g, 0.29 mmol) at 0°C was added AcCl (0.023 mL, 0.30 mmol). After 0.5 h the reaction mixture was poured into aqueous saturated $NaHCO_3$ (15 mL), extracted with ether (3 X 25 mL), dried ($MgSO_4$), and concentrated. Flash chromatography (3:1 hexanes:ethyl acetate) provided 62 mg (65%) of **2 acetate** (R = OCH_3, R' = H) as a colorless oil.



2 acetate (R = OCH_3, R' = H)

2 acetate (R = OCH_3, R' = H): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.07 (d, *J* = 5.7 Hz, 1 H), 5.98 (d, *J* = 5.4 Hz, 1 H), 4.15 (m, 2 H), 3.68 (s, 3 H), 3.26 (s, 3 H), 3.15 (dd, *J* = 16.2, 5.5 Hz, 1 H), 3.07 (dd, *J* = 5.8, 4.5 Hz, 1 H), 2.43 (dd, *J* = 16.2, 4.2 Hz, 1 H), 2.17 (s, 3 H), 1.48 (s, 3 H), 1.22 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 170.0, 169.2, 167.0, 154.1, 135.4, 129.7, 119.6, 114.1, 87.2, 60.7, 52.5, 52.2, 51.3, 26.7, 21.1, 20.6, 14.1; IR (CCl_4) 1765, 1758, 1721 cm^{-1}; HRMS calcd for $C_{17}H_{23}O_8$ (MH^+) 355.1393, found 355.1404.

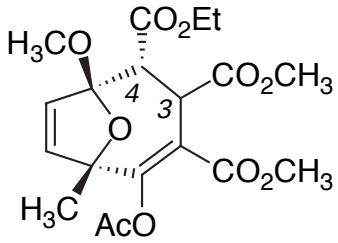


3 (R = OCH_3, R' = H)

3 (R = OCH_3, R' = H): ^{1}H NMR (300MHz, $CDCl_3$) δ 6.11 (d, *J* = 5.7 Hz, 1 H), 5.75 (d, *J* = 5.7 Hz, 1 H), 5.14 (dd, *J* = 11.6, 2.4 Hz, 1 H), 4.21 (m, 2H), 3.72 (s, 3H), 3.30 (s, 3 H), 3.04 (dd, *J* = 4.5, 2.4 Hz, 1 H), 2.08 (m, 2 H), 1.57 (s, 3 H), 1.29 (t, *J* = 6.9 Hz, 3 H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.5, 171.6, 171.5, 137.4, 133.1, 116.1, 92.8, 60.9, 53.9, 52.1, 51.2, 49.0, 26.9, 20.8, 14.2; IR (CCl_4) 1734 cm^{-1}; HRMS calcd for $C_{15}H_{21}O_7$ (MH^+) 313.1287, found 313.1288.

Oxabicyclo[4.2.1]nonenes 2 and 3 (R = OCH_3, R' = CO_2CH_3). According to the general procedure, **1** (0.117 g, 0.52 mmol), NaH (0.013 g, 0.52 mmol), dimethyl fumarate (0.088 g, 0.61 mmol), and DMF (2 mL) gave 0.11 g (55%) of **2** (R = OCH_3, R'

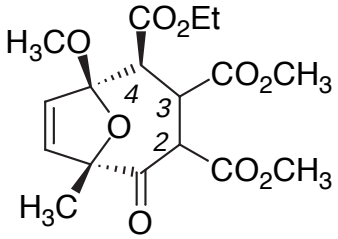
= CO_2CH_3) and 0.026 g (15%) of **3** (R = OCH_3, R' = CO_2CH_3) after flash chromatography (2:1 hexanes:ethyl acetate).



2 acetate (R = OCH_3, R' = CO_2CH_3)

To simplify its characterization, **2** (1:1 mixture of C-2 isomers) was converted into the corresponding enol acetate. To a solution of **2** (R = OCH_3, R' = CO_2CH_3) (0.107 g, 0.29 mmol), THF (2 ml), and KOt-Bu (0.038 g, 0.34 mmol) at 0°C was added AcCl (0.023 mL, 0.44 mmol). After 0.5 h the reaction mixture was poured into aqueous saturated $NaHCO_3$ (15 mL), extracted with ether (3 X 25 mL), dried ($MgSO_4$), and concentrated. Flash chromatography (3:1 hexanes:ethyl acetate) provided 70 mg (59%) of **2 acetate** (R = OCH_3, R' = CO_2CH_3) as a colorless oil.

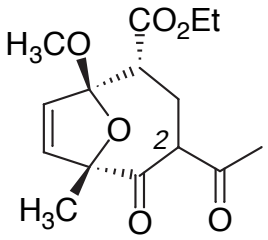
2 acetate (R = OCH_3, R' = CO_2CH_3): ^{1}H NMR (250 MHz, $CDCl_3$) δ 6.17 (s, 2 H), 4.62 (d, *J* = 5.4 Hz, 1 H), 4.14 (q, *J* = 7.1 Hz, 2 H), 3.71 (s, 3 H), 3.54 (s, 3H), 3.40 (d, *J* = 5.4 Hz, 1 H), 3.26 (s, 3 H), 2.17 (s, 3 H), 1.39 (s, 3 H), 1.23 (t, J = 7.1 Hz, 3 H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 170.2, 169.2, 168.6, 166.2, 159.5, 137.2, 128.9, 118.8, 114.3, 85.5, 60.7, 57.2, 52.5, 52.3, 51.1, 44.0, 21.4, 20.5, 13.9; IR (CCl_4) 1746, 1721 cm^{-1}; HRMS calcd for $C_{19}H_{25}O_{10}$ (MH^+) 413.1448, found 413.1444.



3 (R = OCH_3, R' = CO_2CH_3)

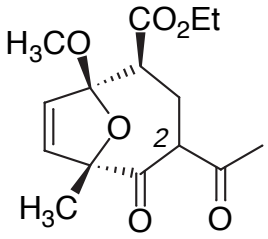
3 (R = OCH_3, R' = CO_2CH_3) (1:1 mixture of isomers): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.25 (d, *J* = 6.0 Hz, 1 H), 6.01 (d, *J* = 6.0 Hz, 1 H), 6.0 (d, *J* = 6.0 Hz, 1 H), 5.90 (d, *J* = 6.0 Hz, 1 H), 4.47 (d, *J* = 10.5 Hz, 1 H), 4.24-4.11 (m, 5 H), 3.80 (d, *J* = 10.5, 1 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.66 (s, 3 H), 3.60 (s, 3 H), 3.30-3.14 (m, 3 H), 3.25 (s, 3 H), 3.23 (s, 3 H), 1.55 (s, 3 H), 1.53 (s, 3 H), 1.29 (t, *J* = 6.9 Hz, 3 H), 1.24 (t, *J* = 6.9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.5, 204.5, 172.3, 171.7, 170.6, 170.2, 169.6, 167.3, 135.7, 130.4, 130.2, 115.8, 115.2, 93.1, 92.1, 61.4, 61.1, 60.3, 58.9, 58.5, 53.0, 52.8, 52.7, 52.4, 50.9, 50.5, 45.0, 43.5, 21.1, 20.6, 14.1, 14.0; IR (CCl_4) 1740 cm^{-1}; HRMS calcd for $C_{17}H_{23}O_9$ (MH^+) 371.1342, found 371.1335.

Oxabicyclo[4.2.1]nonenes 2 and 3 (R = CH_3, R' = H). According to the general procedure, **1** (0.12 g, 0.52 mmol), NaH (0.013 g, 0.52 mmol), methyl vinyl ketone (0.052 mL, 0.63 mmol) and DMF (2 mL), gave 0.062 g of **2** (R = CH_3, R' = H) (40%) along with 0.020 g of **3** (R = CH_3, R' = H) (13%) after flash chromatography (3:1 hexanes:ethyl acetate).



2 (R = CH_3, R' = H)

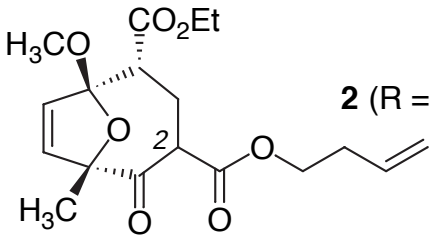
2 (R = CH_3, R' = H) (1:1 mixture of C-2 isomers): ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.15 (d, *J* = 5.8 Hz, 2 H), 6.03 (d, *J* = 5.8 Hz, 1 H), 5.98 (d, *J* = 5.8 Hz, 1 H), 4.14 (m, 4 H), 3.43 (dd, *J* = 12.8, 4.0 Hz, 1 H), 3.37 (dd, *J* = 5.2, 2.6 Hz, 1 H), 3.27 (s, 3 H), 3.22 (s, 3 H), 3.09 (dd, *J* = 12.7 Hz, 3.7 Hz, 1 H), 2.38 (dd, *J* = 4.6, 4.0 Hz, 1 H), 2.35 (dd, *J* = 5.1, 4.0 Hz, 1 H), 2.14 (s, 3 H), 2.13 (s, 3 H), 2.10 (dd, *J* = 3.6, 1.8 Hz, 1 H), 1.84 (m, 2 H), 1.57 (s, 3 H), 1.49 (s, 3 H), 1.26-1.19 (m, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.8, 209.5, 205.7, 200.7, 172.0, 171.4, 136.2, 135.2, 130.2, 129.7, 117.3, 116.9, 92.4, 92.1, 66.5, 62.9, 61.0, 60.8, 54.9, 51.1, 50.9, 30.0, 29.2, 26.9, 26.3, 21.8, 20.9, 20.7, 14.1, 14.0; IR (CCl_4) 1740, 1709 cm^{-1}; HRMS calcd for $C_{15}H_{21}O_6$ (MH^+) 297.1338, found 297.1331.



3 (R = CH_3, R' = H)

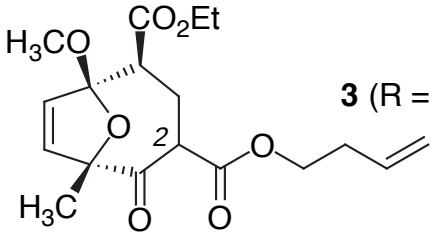
3 (R = CH_3, R' = H): ^{1}H NMR (500MHz, $CDCl_3$) δ 6.07 (d, *J* = 5.5 Hz, 1H), 5.72 (d, *J* = 5.5 Hz, 1 H), 5.28 (d, *J* = 11.5, 1.5 Hz, 1 H), 4.16 (m, 1 H), 4.12 (m, 1 H), 3.30 (s, 3 H), 3.00 (dd, *J* = 5.0, 2.5 Hz, 1 H), 2.15 (s, 3 H), 2.10 (partially obscured ddd, *J* = 15.8, 2.1, 2.1 Hz, 1 H), 1.82 (ddd, *J* = 15.8, 11.4, 4.9 Hz, 2 H), 1.57 (s, 3 H), 1.26 (t, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.6, 206.6, 171.7, 137.3, 130.1, 116.4, 93.0, 60.9, 59.4, 51.1, 48.7, 30.8, 25.7, 20.9, 14.1; IR (CCl_4) 1727, 1709 cm^{-1}; HRMS calcd for $C_{15}H_{21}O_6$ (MH^+) 297.1338, found 297.1346.

Oxabicyclo[4.2.1]nonenes 2 and 3 (R = $OCH_2CH_2CHCH_2$, R' = H). According to the general procedure, **1** (0.13 g, 0.57 mmol), NaH (0.014 g, 0.57 mmol), 3-butenyl acrylate (excess), and DMF (2 mL) gave 0.12 g of **2** (61%) and 36 mg of **3** (18%) after flash chromatography (3:1 hexanes:ethyl acetate).



2 (R = $OCH_2CH_2CHCH_2$, R' = H)

2 (R = $OCH_2CH_2CHCH_2$, R' = H) (1:1 mixture of C-2 isomers): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.14 (d, *J* = 6.0 Hz, 1 H), 6.10 (d, *J* = 5.7 Hz, 1 H), 6.04 (d, *J* = 6.0 Hz, 1 H), 5.98 (d, *J* = 6.0 Hz, 1 H), 6.0-5.7 (m, 2 H), 5.12-5.02 (m, 4 H), 4.25-4.04 (m, 8 H), 3.60 (dd, *J* = 12.6, 4.2 Hz, 1 H), 3.50 (dd, *J* = 12.6, 4.2 Hz, 1 H), 3.44 (dd, *J* = 5.7, 2.1 Hz, 1 H), 3.27 (s, 3 H), 3.21 (s, 3 H), 3.13 (dd, *J* = 12.6, 4.2 Hz), 2.41-2.28 (m, 4 H), 2.10-1.83 (m, 4 H), 1.54 (s, 3 H), 1.50 (s, 3 H), 1.25 (t, *J* = 5.7 Hz, 6 H); ^{13}C NMR (75.0 MHz, $CDCl_3$) δ 207.2, 207.0, 172.7, 172.0, 171.2, 170.3, 169.9, 167.7, 136.6, 136.2, 136.0, 133.9, 133.7, 133.4, 129.7, 129.2, 117.6, 117.2, 116.8, 116.5, 116.0, 92.5, 92.2, 91.9, 64.6, 64.3, 63.5, 61.0, 60.8, 59.6, 57.4, 54.8, 51.4, 51.0, 50.8, 32.9, 32.6, 31.5, 29.4, 27.8, 26.6, 22.1, 21.4, 20.9, 14.1; IR (CCl_4) 1721 cm^{-1}; HRMS calcd for $C_{18}H_{25}O_7$ (MH^+) 353.1600, found 353.1600.

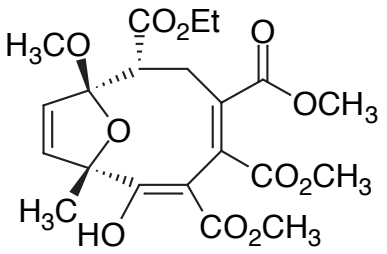


3 (R = $OCH_2CH_2CHCH_2$, R' = H)

3 (R = $OCH_2CH_2CHCH_2$, R' = H): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.10 (d, *J* = 5.7 Hz, 1 H), 5.75 (d, *J* = 5.7 Hz, 1 H), 5.77-5.74 (m, 1H), 5.11-5.02 (m, 2 H), 4.29-4.10 (m, 4 H), 3.29 (s, 3 H), 3.02 (dd, *J* = 4.8, 2.4 Hz, 1 H), 2.39 (tq, *J* = 6.9, 1.5 Hz, 2 H), 2.10-2.00 (m, 2 H), 1.56 (s, 3 H), 1.29 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (300 MHz, $CDCl_3$) δ 208.1, 171.4, 171.0, 137.4, 133.8, 130.1, 117.2, 116.1, 92.8, 64.1, 60.8, 54.0, 51.1, 49.0, 32.9, 26.8, 20.8, 14.2; IR (CCl_4) 1746, 1721 cm^{-1}; HRMS calcd for $C_{18}H_{25}O_7$ (MH^+) 353.1600, found 353.1604.

Representative Procedure for the Tandem Michael Addition-Anionic Fragmentation Reaction. The Generation of Oxabicyclo[6.2.1]dodecene 7 (R = CO_2CH_3, R' = CH_3): To a solution of **2** (R = OCH_3, R' = H) (0.043 g, 0.14 mmol), THF (0.7 mL), and NaH (0.003 g, 0.14 mmol) at 0°C was added DMAD (0.024 mL, 0.15 mmol). After stirring for 2 h the reaction mixture was poured into pH 7.5 phosphate buffer (15 mL). The mixture was extracted with ethyl acetate (3 × 25mL), dried ($MgSO_4$), and

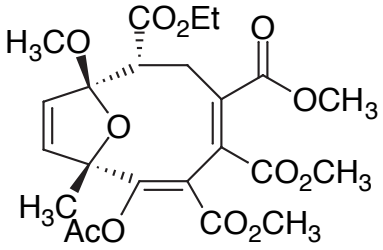
concentrated. Flash chromatography (3:1 hexanes:ethyl acetate) provided 58 mg (92%) of **7** (R = CO_2CH_3, R' = CH_3) as a colorless oil.



7 (R = CO_2CH_3, R' = CH_3)

7 (R = CO_2CH_3, R' = CH_3): ^{1}H NMR (250 MHz, $CDCl_3$) δ 13.6 (s, 1H), 6.54 (d, *J* = 5.8 Hz, 1 H), 5.79 (d, *J* = 5.8 Hz, 1 H), 4.12 (q, *J* = 7.3 Hz, 2 H), 3.84 (s, 3 H), 3.69 (s, 3 H), 3.67 (s, 3 H), 3.15 (s, 3 H), 3.03 (dd, *J* = 9.7, 3.9 Hz, 1 H), 2.53 (m, 2 H), 1.68 (s, 3 H), 1.23 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 176.5, 173.3, 171.5, 169.7, 167.1, 143.8, 138.2, 125.9, 117.6, 94.9, 91.6, 77.2, 61.1, 52.6, 52.5, 50.6, 49.6, 31.5, 25.2, 14.1; IR (CCl_4) 3402, 1732 cm^{-1}; HRMS calcd for $C_{21}H_{27}O_{11}$ (MH^+) 455.1553, found 455.1555.

7 (R = CO_2CH_3, R' = CH_3) was converted into the corresponding enol acetate. To a solution of **7** (R = CO_2CH_3, R' = CH_3) (0.063 g, 0.14 mmol), THF (1.0 mL), and NaH (0.004 g, 0.14 mmol) at 0°C was added AcCl (0.011 mL, 0.15 mmol). After 2 h the reaction mixture was poured into aqueous saturated $NaHCO_3$ (10 mL), extracted with ether (3 X 10 mL), dried ($MgSO_4$), and concentrated. Flash chromatography (hexanes: ethyl acetate = 1:1) provided 35 mg of **7 acetate** (R = CO_2CH_3, R' = CH_3) (51%) as a colorless oil.



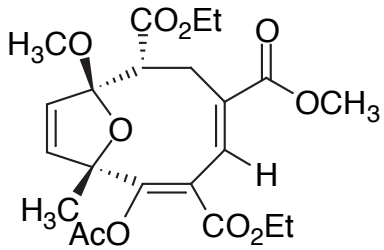
7 acetate (R = CO_2CH_3)

7 acetate (R = CO_2CH_3, R' = CH_3): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.23 (d, J = 6 Hz, 1 H), 5.92 (d, J = 6 Hz, 1 H), 4.11 (q, J = 6.9 Hz, 2 H), 3.85 (s, 3 H), 3.68 (s, 3 H), 3.60 (s, 3 H), 3.15 (s, 3 H), 3.11 (partially obscured d, *J* = 11.7 Hz, 1 H), 2.78 (dd, *J* = 14.1, 11.7 Hz, 1 H), 2.78-2.58 (dd, *J* = 14.1, 1.5 Hz, 1 H), 2.26 (s, 3 H), 1.62 (s, 3 H), 1.22 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 169.4, 167.7, 165.6, 164.6, 158.6, 145.6, 136.4, 127.4, 125.2, 118.1, 114.1, 91.1, 61.1, 52.8, 52.5, 52.4, 50.7, 49.4, 31.4, 24.7, 21.4, 14.1; IR (CCl_4): 1783, 1738 cm^{-1}; HRMS calcd for $C_{23}H_{29}O_{12}$ (MH^+) 497.1659, found 479.1662.

Oxabicyclo[6.2.1]dodecene 7 (R = H, R' = CH_2CH_3) and Oxabicyclo[4.2.1]nonene 8 (R = H, R' = CH_2CH_3): According to the general

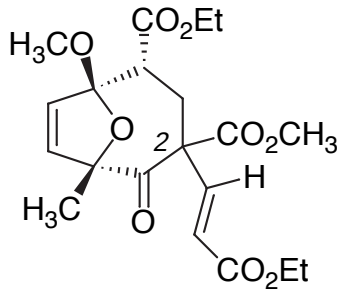
procedure, **2** (R = OCH_3, R' = H) (0.090 g, 0.29 mmol), THF (1.5 mL), NaH (0.007 g, 0.29 mmol), and ethyl propiolate (0.031 g, 0.32 mmol) gave 52 mg (44%) of an inseparable mixture of **7** (R = H, R' = CH_2CH_3) and **8** (R = H, R' = CH_2CH_3) after flash chromatography (3:1 hexanes:ethyl acetate).

To effect separation, the mixture of **7** (R = H, R' = CH_2CH_3) and **8** (R = H, R' = CH_2CH_3) was converted into the corresponding enol acetate. To a solution of the mixture and THF (1 mL) at 0°C was added KO*t*-Bu (0.014 g, 0.13 mmol). After stirring for 0.5 h, acetyl chloride (0.01 mL, 0.14 mmol) was added. The reaction mixture was stirred for an additional 0.5 h, diluted with ether (10 mL), washed with aqueous saturated $NaHCO_3$ (10 mL), dried ($MgSO_4$), and concentrated. Flash chromatography (2:1 hexanes:ethyl acetate) provided 0.010 g (17% from **2**) of **7 acetate** (R = H, R' = CH_2CH_3) along with 0.030 g (25% from **2**) of **8** (R = H, R' = CH_2CH_3).



7 acetate (R = H, R' = Et)

7 acetate (R = H, R' = CH_2CH_3): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (s, 1 H), 6.26 (d, *J* = 6.0 Hz, 1 H), 5.95 (d, *J* = 6.0 Hz, 1 H), 4.17 (m, 4 H), 3.76 (s, 3H), 3.14 (s, 3H), 3.06 (d, *J* = 10.2 Hz, 1 H), 2.56 (br d, *J* = 13.5 Hz, 1 H), 2.46 (dd, *J* = 14.1, 10.5 Hz, 1 H), 2.24 (s, 3 H), 1.54 (s, 3 H), 1.28 (t, *J* = 7.2 Hz, 3 H), 1.25 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 168.0, 166.8, 164.6, 155.7, 136.3, 134.4, 132.4, 126.7, 118.3, 116.3, 90.9, 61.5, 60.8, 52.1, 50.7, 50.5, 27.6, 24.9, 21.3, 14.2, 14.1; IR (CCl4): 1728, 1635 cm^{-1}; HRMS calcd for $C_{22}H_{28}O_{10}Cs$ (MCs^+) 585.0737, found 585.0727.



8 (R = H, R' = Et)

8 (R = H, R' = CH_2CH_3) (1:1 mixture of C-2 isomers): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13 (d, *J* = 16.2 Hz, 1 H), 6.15 (d, *J* = 5.8 Hz, 1 H), 6.07 (d, *J* = 5.7 Hz, 1H), 6.01 (d, *J* = 5.8 Hz, 1H), 5.98 (d, J = 12.4 Hz, 1 H), 5.90 (d, *J* = 12.4 Hz, 1 H), 5.78 (d, *J* = 16.2 Hz, 1 H), 4.15 (m, 8 H), 3.77 (s, 3H), 3.75 (s, 3H), 3.67 (dd, *J* = 12.5, 4.3 Hz, 1H), 3.53 (dd, *J* = 12.6, 4.0 Hz, 1H), 3.22 (s, 3H), 3.21 (s, 3H), 2.71 (dd, *J* = 14.9, 12.7 Hz, 1H),

2.23 (dd, *J* = 14.9, 4.1 Hz, 1 H), 2.20 (dd, *J* = 14.9, 4.4 Hz), 1.85 (dd, *J* = 14.9 Hz, 12.6 Hz, 1 H), 1.56 (s, 3 H), 1.53 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 6 H), 1.24 (t, *J* = 7.2 Hz, 6 H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 202.4, 171.5, 171.4, 170.4, 167.9, 165.4, 164.9, 144.5, 140.9, 136.3, 136.2, 129.5, 128.9, 123.8, 122.5, 116.5, 92.6, 92.4, 63.8, 62.8, 61.0, 60.7, 60.4, 53.0, 52.8, 51.6, 50.9, 33.3, 32.6, 22.3, 22.1, 14.2, 14.1; IR (CCl_4) 1734, 1221; HRMS calcd for $C_{20}H_{27}O_9$ (MH^+) 411.1655, found 411.1659.