SUPPORTING INFORMATION

(20 pages including this cover page)

The Synthesis of Bridged Medium Sized Rings through the Intramolecular Pauson-Khand Reaction

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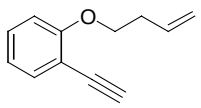
Experimental:

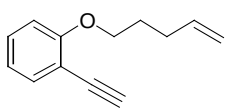
General: All chemicals and solvents were purchased from commercial vendors and were used as received unless indicated otherwise. All reactions involving air- or water-sensitive compounds were conducted in oven-dried glassware under an atmosphere of dry argon or nitrogen. Tetrahydrofuran and CH_2Cl_2 were distilled from sodium/benzophenone ketyl and from CaH_2 respectively under a nitrogen atmosphere. NMR spectra were obtained on a JEOL Eclipse+ 500 MHz spectrometer. ^1H NMR spectra were recorded in deuteriochloroform (unless otherwise indicated) at a spectrometer frequency of 500.16 MHz, residual protiochloroform was used as reference. The ^{13}C NMR spectra were obtained in deuteriochloroform (unless otherwise indicated) at 125.79 MHz, using $^{13}CDCl_3$ (δ = 77.0) as internal reference. Infrared (IR) spectra were obtained either on a BioRad 3240-SPC or a Bruker Vector 22 FT-IR spectrometer, using KBr pressed pellets for solids or neat films between NaCl plates for liquids and oils, and are reported in cm^{-1}. Mass spectra were recorded by electron impact (at 70 eV) on a Finnigan MAT TSQ-70 spectrometer or a Bear Instruments, Kodiak 1200 spectrometer. Elemental analyses were performed on a Perkin Elmer 2400 CHN Elemental Analyzer or determined by Quantitative Technologies Inc. (QTI), Whitehouse, New Jersey. Melting points were recorded on a Thomas Hoover Scientific capillary tube melting point apparatus and are uncorrected. Analytical thin layer chromatography (TLC) was performed on silica gel $60F_{254}$ aluminum backed precoated plates (0.25 mm layer).

2-Ethynyl-(3-butenyloxy)benzene (4b)



DEAD (2.74 g, 24 mmol) in THF (50 mL) was added dropwise to a solution of **3**[1] (1.03 g, 5.3 mmol), 3-butene-1-ol (1.4 mL, 10.0 mmol) and PPh_3 (4.14 g, 15.0 mmol) in THF (150 mL) and stirred at 0 °C for 2 h. The solvent was removed in vacuum, the residue was dissolved in pentane, washed with aq. KOH, dried ($MgSO_4$) and concentrated under reduced pressure to afford the ether, 2.39 g (contaminated with triphenyl phosphine and/or oxide). To this crude mixture, was added to a solution of K_2CO_3 (2.39 g, 17.3 mmol) in MeOH/THF (50 mL, 1:1). The reaction mixture was stirred overnight. This mixture was concentrated under reduced pressure, washed with EtOAc and filtered through a plug of silica gel. The residue was purified by flash chromatography (silica gel, 98/2 *n*-hexane/EtOAc) to furnish **4b** (1.23 g, 84%) as a colorless liquid: ^1H NMR: δ = 7.45 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.27–7.30 (m, 1H), 6.91 (dt, *J* = 7.5, 0.9, Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 5.93 (ddt, *J* = 17.1, 10.3, 3.4, Hz, 1H), 5.20 (app. dq, *J* = 17.1, 1.7 Hz, 1H), 5.11–5.14 (m, 1H), 4.08 (t, *J* = 6.9 Hz, 2H), 3.25 (s, 1H), 2.59 (dd, *J* = 8.5, 6.9 Hz, 2H); ^{13}C NMR: δ = 160.0, 134.5, 134.2, 130.2, 120.6, 112.2, 111.9, 81.1, 81.09, 80.13, 68.1 33.6; IR (neat, cm^{-1}): 3311, 2106, 1711, 1642, 1062, 733; EIMS (*m/z*): 172 (M^+, 100%). Anal. Calcd. For $C_{12}H_{12}O$: C, 83.69; H, 7.02. Found: C, 83.20; H, 7.05.

2-Ethynyl-(4-pentenyloxy)benzene (4c)

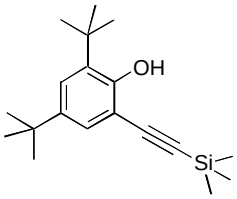


5-Bromopentene (4.01 g, 26.9 mmol) was added to a solution of *o*-ethynylphenol[1] (1.06 g, 8.9 mmol) and K_2CO_3 (3.72 g, 26.9 mmol) in acetone (20 mL). The mixture was stirred at room temperature for 16 h. The reaction mixture was concentrated under reduced pressure and filtered through a plug of silica gel (EtOAc). The residue was purified by flash chromatography (silica gel, 98/2 *n*-hexane/EtOAc) to afford **4c** (0.92 g, 55%) as a colorless liquid: ^1H NMR: δ = 7.46 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.29 (dt, *J* = 7.7, 1.7 Hz, 1H), 6.91 (t, *J* = 7.7 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 5.87 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.07 (dd, *J* = 16.9, 1.5, Hz, 1H), 5.02, (dd, *J* = 10.1 , 1.5 Hz, 1H), 4.05 (t, *J* = 6.8 Hz, 2H), 3.28 (s, 1H), 2.29 (q, *J* = 6.7 Hz, 2H), 1.94 (quint, *J* = 6.8 Hz, 2H); ^{13}C NMR: δ

1. Arcadi, A.; Cacchi, S.; Del Rosario, M.; Fabrizi, G.; Marinelli, F. *J. Org. Chem.* **1996**, *61*, 9280.

= 160.5, 137.9, 134.1, 130.2, 120.4, 115.3, 112.1, 111.7, 81.0, 80.2, 67.9, 30.1, 28.3; IR (neat, cm^{-1}): 3295, 3062, 2106, 1640, 1594, 1161, 751; EIMS (*m/z*): 186 (40%, M^+), 158 (100 %). Anal. Calcd. for $C_{13}H_{14}O$: C, 83.83; H, 7.58. Found: C, 83.77; H, 7.56.

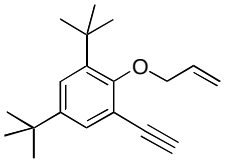
2,4-Di-*tert*-butyl-6-(trimethylsilylethynyl)phenol (8)



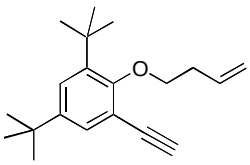
To a stirred solution of 3,5-di-*tert*-butyl-2-iodophenol[2] (3.54 g, 10.7 mmol) and Et_3N (5.9 mL) in dioxane (5.9 mL), (trimethylsilyl)acetylene (1.37 g, 13.9 mmol), $PdCl_2(PPh_3)_2$ (0.08 g, 0.1 mmol) and CuI (0.04 g, 0.2 mmol) were added. The reaction mixture was stirred at 55 °C (bath temperature) under argon for 5 h. Et_2O (50 mL) and 1M HCl (20 mL) were added, and the organic layer was separated, neutralized with a saturated $NaHCO_3$ (30 mL) solution, washed with H_2O, dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel, *n*-hexane) to afford **8** (3.03 g, 93%), a colorless solid. mp: 114–116 °C. ^{1}H NMR: δ = 7.27 (d, *J* = 2.4 Hz, 1H), 7.20 (d, *J* = 2.4 Hz, 1H), 6.08 (s, 1H), 1.39 (s, 9H), 1.27 (s, 9H), 0.27 (s, 9H); ^{13}C NMR: δ = 153.6, 142.1, 134.8, 125.7, 125.6, 109.2, 76.8, 35.0, 34.3, 31.5, 29.5, 0.1; IR (KBr, cm^{-1}): 3494, 2959, 2143, 1467, 1122, 758, 657; EIMS (*m/z*): 302 (M^+), 286 (100%), 73, 57. Anal. Calcd. for $C_{19}H_{30}OSi$: C, 75.43; H, 9.99. Found: C, 75.32; H, 10.01.

2,4-Di-*tert*-butyl-6-ethynyl-(2-propenyloxy)benzene (10a)

Allyl bromide (0.48 g, 4.0 mmol) was added to a solution of **8** (0.30 g, 1.0 mmol) and K_2CO_3 (0.55 g, 4.0 mmol) in acetone (33 mL). The mixture was stirred at room temperature for 8 h. The mixture was then concentrated under reduced pressure and filtered through a plug of silica gel (EtOAc) to furnish the allylated phenol (0.35 g). K_2CO_3 (0.55 g, 4.0 mmol) was added to a solution of the phenol (0.35 g, 1.0 mmol) in MeOH/THF (10 mL/1:1). The mixture was stirred at room temperature for 4 h. The reaction mixture was concentrated under reduced pressure and filtered through a plug of silica gel plug (EtOAc). The residue was purified by flash chromatography (silica gel, 99/1 *n*-hexane/ether) to furnish **10a** (0.27 g, quantitative) as a colorless liquid: ^{1}H NMR: δ = 7.38 (d, *J* = 2.5 Hz, 1H), 7.36 (d, *J* = 2.5 Hz, 1H), 6.17 (m, 1H), 5.49 (dd, *J* = 10.4, 1.3 Hz, 1H), 5.29 (dd, *J* = 17.2, 1.3 Hz, 1H), 4.81 (d, *J* = 5.3 Hz, 2H), 3.31 (s, 1H), 1.32 (s, 9H), 1.42 (s, 9H), ^{13}C NMR: δ = 157.7, 145.4, 142.2, 134.2, 129.5, 125.6, 116.8, 115.5, 81.9, 81.5, 73.6, 35.3, 34.5, 31.4, 30.7; IR (neat, cm^{-1}): 3311, 2962, 2106, 1647, 1437, 1122, 742, 650; EIMS (*m/z*): 270 (M^+), 254 (100%), 198, 172, 57. Anal. Calcd. for $C_{19}H_{26}O$: C, 84.39; H, 9.69. Found: C, 84.34; H, 9.53.



2,4-Di-*tert*-butyl-6-ethynyl-(3-butenyloxy)benzene (10b)

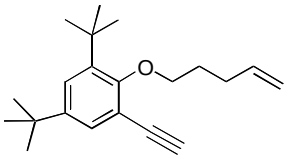


DEAD (1.83 g, 10.5 mmol) in THF (50 mL) was added dropwise to a solution of **8** (1.06 g, 3.5 mmol), 3-butene-1-ol (0.51 g, 7.0 mmol) and PPh_3 (2.75 g, 10.5 mmol) in THF (100 mL) and stirred at 0 °C for 6 h. The solvent was removed in vacuum; the residue dissolved in pentane, washed with aq. KOH (10%, 30 mL), dried ($MgSO_4$) and concentrated under reduced pressure to afford the ether (1.41 g) as a liquid. To this crude mixture (1.41 g, 4.1 mmol) in MeOH/THF (15 mL/1:1) was added K_2CO_3. The reaction mixture was stirred overnight. The reaction mixture was concentrated under reduced pressure, washed with EtOAc and filtered through a plug of silica gel. The residue was purified by flash chromatography (silica gel/pentane) to furnish **10b** (0.75 g, 80%) as a colorless liquid: ^{1}H NMR: δ = 7.34 (d, *J* = 2.4 Hz, 1H), 7.33 (d, *J* =

2. Gnaim, J. M.; Keehn, P. H.; Green, B. S. *Tetrahedron Lett.* **1992**, *33*, 2883.

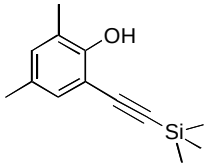
2.4 Hz, 1H), 5.98 (ddt, *J* = 17.0, 10.3, 6.8 Hz, 1H), 5.16-5.20 (m, 1H,), 5.09-5.11 (m, 1H), 4.30 (t, *J* = 7.0 Hz, 2H), 3.30 (s, 1H), 2.62-2.67 (m, 2H), 1.39 (s, 9H), 1.29 (s, 9H); ^{13}C NMR: δ = 158.1, 145.1, 142.0, 134.9, 129.5, 125.5, 116.8, 115.4, 82.1, 81.3, 72.2, 35.3, 34.8, 34.5, 31.4, 30.8, IR (neat,
cm^{-1}): 3304, 2959, 2107, 1656, 1122, 727; EIMS (*m*/*z*): 284 (M^+), 269, 215 (100%), 171, 57. Anal. Calcd. for $C_{20}H_{28}O$: C, 84.45; H, 9.92. Found: C, 84.48; H, 10.03.

2,4-Di-*tert*-butyl-6-ethynyl-(4-pentenyloxy)benzene (10c)



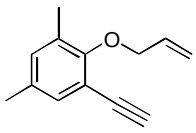
DEAD (0.78 g, 4.5 mmol) in THF (15 mL) was added dropwise to a solution of **8** (0.44 g, 1.5 mmol), 4-pentene-1-ol (0.25 g, 3.0 mmol) and PPh_3 (1.18 g, 3.0 mmol) in THF (50 mL). The reaction mixture was stirred at 0 °C for 2 h. The solvent was removed in vacuum; the residue was dissolved in pentane, washed with 10% aq. KOH (30 mL), dried (Na_2SO_4) and concentrated under reduced pressure to afford the pentenylated phenol (1.12 g) as a liquid. To this crude mixture in MeOH/THF (15 ml/1:1) was added K_2CO_3 (0.42 g, 3.0 mmol). The reaction mixture was stirred overnight. The reaction mixture was concentrated under reduced pressure, dissolved EtOAc and filtered through a plug of silica gel. The residue was purified by flash chromatography (silica gel, *n*-hexane) to furnish **10c** (0.36 g, 83%) as a yellow liquid: ^{1}H NMR: δ = 7.33 (d, *J* = 2.6 Hz, 1H), 7.32 (d, *J* = 2.6 Hz, 1H), 5.89 (ddt, *J* = 17.0, 10.1, 6.6 Hz, 1H), 5.06–5.10 (m, 1H), 4.98–5.00 (m, 1H), 4.24 (t, *J* = 6.8 Hz, 2H), 3.28 (s, 1H), 2.27 (app. dt, *J* = 7.3, 6.6 Hz, 2H), 1.97 (m, 2H), 1.38 (s, 9H), 1.28 (s, 9H); ^{13}C NMR: δ = 158.2, 145.1, 142.0, 138.4, 129.5, 125.5, 115.4, 114.8, 82.1, 81.2, 72.6, 35.3, 34.5, 31.4, 30.8, 30.3, 29.5; IR (neat, cm^{-1}): 3301, 2857, 2103, 1641, 1122, 730; EIMS (*m*/*z*): 298 (M^+), 282 (100%), 214, 57. Calcd. for $C_{21}H_{30}O$: C, 84.51; H, 10.13. Found: C, 84.58; H, 10.51.

2,4-Dimethyl-6-(trimethylsilylethynyl)phenol (9)



To a stirred solution of 2-iodo-3,5-dimethylphenol[3] (0.60 g, 2.4 mmol) and Et_3N (1.3 mL) in dioxane (1.3 mL), (trimethylsilyl)acetylene (0.31 g, 3.2 mmol), $PdCl_2(PPh_3)_2$ (0.02 g, 0.02 mmol) and CuI (0.09 g, 0.05 mmol) were added. The reaction mixture was stirred at 55 °C (bath temperature) under argon for 5 h. Et_2O (50 mL) and 1M HCl (20 mL) were added, and the organic layer was separated, neutralized with a saturated $NaHCO_3$ (30 mL) solution, washed with H_2O, dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel, 99/1 *n*-hexane/Et_2O) to afford **9** (0.41 g, 78%), a liquid: ^{1}H NMR: δ = 6.99 (s, 1H); 6.90 (s, 1H), 5.76 (s, 1H), 2.21 (s, 3H), 2.20 (s, 3H), 0.26 (s, 9H); ^{13}C NMR: δ = 153.3; 133.0, 129.0, 123.6, 108.5, 101.6, 99.8, 20.4, 15.9, 0.1; IR (neat, cm^{-1}): 3514, 2959, 2142, 1475, 1026, 760; EIMS (*m*/z): 218 (M^+), 203 (100%). Anal. Calcd. for $C_{13}H_{18}OSi$: C, 71.50; H, 8.31. Found: C, 71.48; H, 8.42.

2-Ethynyl-4,6-dimethyl-(2-propenyloxy)benzene (11a)

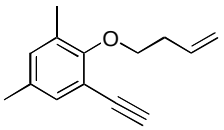


Allyl bromide (2.35 g, 19.4 mmol) was added to a solution of **9** (1.06 g, 4.9 mmol) and K_2CO_3 (2.68 g, 19.4 mmol) in acetone (150 mL). The mixture was stirred at room temperature for 12 h. This was then concentrated under reduced pressure and filtered through a plug of silica gel (EtOAc) to furnish the allylated phenol (1.19 g). To a solution of this crude mixture in MeOH/THF (25 mL/1:1) was added K_2CO_3 (1.91 g, 13.8 mmol). The mixture was stirred at room temperature for 2 h. The reaction mixture was

3. Omura, K. *J. Org. Chem.* **1984**, *49*, 3046.

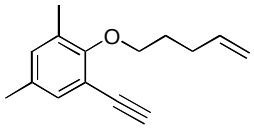
concentrated under reduced pressure and filtered through a plug of silica gel plug (EtOAc). The residue was purified by flash chromatography (silica gel, 70/30 *n*-hexane/benzene) to furnish **11a** (0.78 g, quantitative) as a colorless liquid: ^{1}H NMR: δ = 7.11 (s, 1H), 6.97 (s, 1H), 6.12 (ddt, *J* = 16.7, 10.4, 5.8 Hz, 1H), 5.38–5.42 (m, 1H), 5.22–5.25 (m, 1H), 4.56 (dd, *J* = 5.8, 1.1 Hz, 2H), 3.23 (s, 1H), 2.24 (s, 3H), 2.23 (s, 3H); ^{13}C NMR: δ = 156.9, 134.2, 133.0, 132.8, 132.1, 131.3, 117.6, 115.6, 80.9, 80.8, 74.2, 20.6, 16.4, IR (neat, cm^{-1}): 3291, 2922, 2105, 1646, 1474, 1103, 026, 803, 600; EIMS (*m*/*z*): 186 (M^+), 171, 159 (100%), 145, 115, 91. Anal. Calcd. for $C_{13}H_{14}O$: C, 83.83; H, 7.58. Found: C, 83.72; H, 7.54.

2-Ethynyl-4,6-dimethyl-(3-butenyloxy)benzene (11b)



DEAD (2.46 g, 13.8 mmol) in THF (50 mL) was added dropwise to a solution of **9** (1.01 g, 4.6 mmol), 3-butene-1-ol (0.66 g, 9.2 mmol) and PPh_3 (3.65 g, 13.8 mmol) in THF (110 mL) and stirred at 0 °C for 3 h. The solvent was removed in vacuum, the residue was dissolved in pentane, washed with 10% KOH (25 mL), dried ($MgSO_4$) and concentrated under reduced pressure to afford the butenylated phenol (1.25 g) as a liquid. To a solution of this crude mixture in MeOH/THF (23 mL/1:1) was added K_2CO_3 (1.89 g, 13.7 mmol). The mixture was stirred at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure and filtered through a plug of silica gel plug (EtOAc). The residue was purified by flash chromatography (silica gel/pentane) to furnish **11b** (0.71 g, 77%) as a colorless liquid: ^{1}H NMR: δ = 7.10 (s, 1H), 6.97 (s, 1H), 5.95 (ddt, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.18 (dd, *J* = 17.1, 1.3 Hz, 1H), 5.09 (dd, *J* = 10.3, 1.3 Hz, 1H), 4.06 (t, *J* = 6.8 Hz, 2H), 3.21 (s, 1H), 2.56 (dt, *J* = 6.8, 6.8 Hz, 2H), 2.23 (s, 3H), 2.22 (s, 3H); ^{13}C NMR: δ = 157.1, 134.9, 132.9, 132.1, 116.8, 115.5, 80.8, 80.7, 72.6, 34.9, 20.5, 16.2; IR (neat, cm^{-1}): 3302, 3082, 2103, 1646, 1168, 741, 647; EIMS (*m*/*z*); 200 (M^+), 185 (100%), 172, 159, 115, 91. Anal. Calcd. for $C_{14}H_{16}O$: C, 83.96; H, 8.05. Found: C, 83.81; H, 7.75.

2-Ethynyl-4,6-dimethyl-(4-pentenyloxy)benzene (11c)

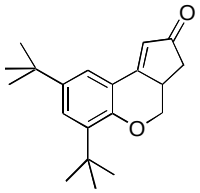


DEAD (2.76. g, 15.8 mmol) in THF (50 mL) was added dropwise to a solution of **9** (1.15 g, 5.28 mmol), 4-pentene-1-ol (0.91 g, 10.6 mmol) and PPh_3 (4.15 g, 15.8 mmol) in THF (110 mL) and stirred at 0 °C for 3 h. The solvent was removed in vacuum, the residue was dissolved in pentane, washed with 10% KOH (25 mL), dried ($MgSO_4$) and concentrated under reduced pressure to afford the pentenylated phenol (1.51 g) as a liquid. To solution of this crude mixture in MeOH/THF (26 mL/1:1) was added K_2CO_3 (2.22 g, 15.8 mmol). The mixture was stirred at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure and filtered through a plug of silica gel plug (EtOAc). The residue was purified by flash chromatography (silica gel/pentane) to furnish **11c** (0.97 g, 86%) as a colorless liquid: ^{1}H NMR: δ = 7.10 (s, 1H), 6.97 (s, 1H), 5.87 (ddt, *J* = 17.0, 10.2, 6.6 Hz, 1H), 5.07 (dd, *J* = 17.0, 1.7 Hz, 1H), 4.98–4.50 (m, 1H), 4.01 (t, *J* = 6.6 Hz, 2H), 3.21 (s, 1H), 2.28 (dt, *J* = 7.0, 6.6 Hz, 2H), 2.34 (s, 3H), 2.23 (s, 3H), 1.91 (tt, *J* = 7.0, 6.6 Hz, 2H); ^{13}C NMR: δ = 157.2, 138.3, 132.9, 132.8, 132.1, 131.2, 115.6, 114.9, 80.8, 80.7, 72.8, 30.3, 29.7, 20.5, 16.2; IR (neat, cm^{-1}): 3296, 2931, 2106, 1638, 1471, 1147, 780, 650; EIMS (*m*/*z*); 214 (M^+), 185 (100%), 172, 159, 146, 115, 91. Anal. Calcd. for $C_{15}H_{18}O$: C, 84.07; H, 8.47. Found: C, 84.30; H, 8.17.

General Procedure for the PK Cyclizations: $Co_2(CO)_8$ (2 eq.) was added to a stirred solution of enyne (1 eq.) in CH_2Cl_2. Stirring was continued at room temperature until the formation of the complex was complete (monitored by TLC). The reaction mixture was cooled to 0 °C (ice-bath)

then NMO (10-12 eq.) was added in three equal portions in thirty-minute intervals. After the reaction was complete (monitored by TLC), the reaction mixture was concentrated under reduced pressure and filtered though a silica gel plug (EtOAc). Flash chromatography on the residue gave products as colorless solids.

6,8-Di-*tert*-butyl-3a,4-dihydro-3*H*-cyclopenta[*c*]chromen-2-one (12a)

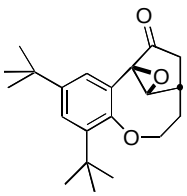


Cyclization following the general procedure utilizing **10a** (53 mg, 0.20 mmol) CH_2Cl_2 (10 mL), $Co_2(CO)_8$ (136 mg, 0.40 mmol) and NMO (281 mg, 2.40 mmol) followed by flash chromatography (silica gel, 85/15 *n*-hexane/EtOAc) afforded **12a** (41 mg, 70%) as a solid: m.p. 179-180 °C; ^{1}H NMR: δ = 7.44 (d, *J* = 2.4 Hz, 1H), 7.39 (d, *J* = 2.4 Hz, 1H), 6.35 (s, 1H,), 4.66 (dd, *J* = 10.4, 5.5, Hz, 1H), 3.84 (dd, *J* = 13.4, 10.4 Hz, 1H), 3.26–3.32 (m, 1H), 2.71 (dd, *J* = 17.9, 6.9 Hz, 1H), 2.08 (dd, *J* = 17.9, 4.3 Hz, 1H), 1.31 (s, 9H), 1.38 (s, 9H); ^{13}C NMR: δ = 206.3, 170.6, 153.1, 143.3, 138.5, 128.5, 121.3, 121.2, 117.0, 69.8, 38.1, 36.5, 35.3, 34.5, 31.4, 29.7, IR (KBr, cm^{-1}): 2962, 1698, 1599. 1479, 1189, 860, 680; EIMS (*m*/*z*): 298 (35%, M^+), 283 (100%), 215, 186, 171, 115, 57. Anal. Calcd. for $C_{20}H_{26}O_2$: C, 80.50; H, 8.78. Found: C, 80.50; H, 9.00.

Cyclization of 10b

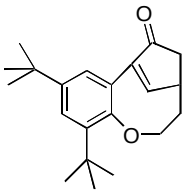
Cyclization following the general procedure utilizing **10b** (113 mg, 0.40 mmol) CH_2Cl_2 (15 mL), $Co_2(CO)_8$ (273 mg, 0.80 mmol) and NMO (489 mg, 4.18 mmol) followed by flash chromatography (silica gel, 87.4/12.5/0.1 *n*-hexane/EtOAc/Et_3N) afforded **14a** (47 mg, 36%) and **15a** (22 mg, 18%) as colorless solids.

Epoxy ketone (14a)



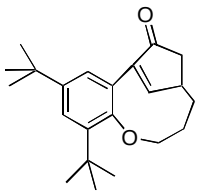
mp: 216–219 °C; ^{1}H NMR: δ = 7.39 (d, *J* = 2.4 Hz, 1H), 7.35 (d, *J* = 2.4 Hz, 1H), 4.45 (app. d, *J* = 13.9 Hz, 1H), 4.04 (s, 1H), 3.40 (app. t, *J* = 13.0 Hz, 1H), 2.93–2.95 (m, 1H), 2.76 (dd, *J* = 6.3, 18.1 Hz, 1H), 2.53–2.50 (m, 1H), 1.98 (app. d, *J* = 18.1 Hz, 1H), 1.94 (app. d, *J* = 15.6 Hz, 1H), 1.32 (s, 9H), 1.31 (s, 9H); ^{13}C NMR: δ = 207.0, 157.7, 145.5, 141.3, 125.4, 125.0, 70.1, 67.5, 56.8, 36.6, 35.2, 34.7, 32.4, 31.5, 30.7; IR (KBr, cm^{-1}): 2954, 1744, 1435, 1113, 737, 647; EIMS (*m*/*z*): 328 (13%, M^+), 299 (100%). Anal. Calcd. for $C_{21}H_{28}O_3$: C, 76.59; H, 8.59; Found: C, 76.92; H 8.79;

Enone (15a)



mp: 236–238 °C; ^{1}H NMR: δ = 8.25 (s, 1H), 7.26 (s, 1H), 7.24 (s, 1H), 4.11 (app. d, *J* = 13.9 Hz, 1H), 3.41 (bs, 1H), 3.33 (app. t, *J* = 13.0 Hz, 1H), 2.77 (dd, *J* = 5.0, 17.8 Hz,1H), 2.64 (app. t, *J* = 13.6 Hz, 1H), 2.28 (app. d, *J* = 17.8 Hz, 1H), 2.04 (app. d, *J* = 15.4 Hz, 1H); ^{13}C NMR: δ = 208.2, 176.0, 158.1, 145.4, 141.3, 125.5, 125.1, 125.0, 67.5, 56.8, 36.6, 35.2, 34.7, 32.4, 31.6, 30.7; IR (KBr, cm^{-1}): 2955, 1701, 1637, 1477, 1112, 836, 737, 747; EIMS (*m*/*z*): 312 (84%, M^+), 297 (100%)

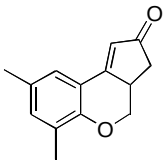
Enone (17a)



Cyclization following the general procedure utilizing **10c** (121 mg, 0.41 mmol) CH_2Cl_2 (20 mL), $Co_2(CO)_8$ (278 mg, 0.81 mmol) and NMO (569 mg, 4.86 mmol) followed by flash chromatography (silica gel, 9/1 *n*-hexane/EtOAc) afforded **17a** (41 mg, 31%) as a colorless solid: mp: 191–192 °C; ^{1}H NMR: δ = 7.52 (s, 1H), 7.28 (d, *J* = 2.5 Hz, 1H), 7.07 (d, *J* = 2.5 Hz, 1H), 4.37 (app. t, *J* = 10.3 Hz, 1H), 3.34–3.35 (m, 1H), 3.20–3.22 (m, 1H), 2.67 (dd, *J* = 18.0, 5.8 Hz, 1H), 2.27 (app. d, *J* = 18.0 Hz, 1H), 1.93–2.04 (m, 1H), 1.80–1.89 (m, 1H), 1.42–1.47 (m, 1H), 1.34 (s, 9H), 1.30 (s, 9H), ^{13}C NMR: δ = 208.5, 166.4, 153.3, 145.3, 141.5, 141.0, 127.8, 123.9, 123.6. 76.3,

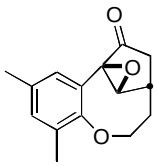
40.5, 39.3, 35.0, 34.6, 33.7, 31.6, 31.2, 25.6; IR (KBr, cm^{-1}): 2956, 1706, 1594, 1441, 1110, 750, 676; EIMS (*m*/*z*): 326 (37%, M^+), 58 (100%).

6,8-Dimethyl-3a,4-dihydro-3*H*-cyclopenta[c]chromen-2-one (12b)



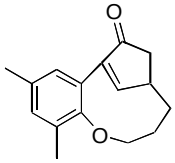
Cyclization following the general procedure utilizing **11a** (49 mg, 0.26 mmol) CH_2Cl_2 (12 mL), $Co_2(CO)_8$ (179 mg, 0.52 mmol) and NMO (363 g, 3.10 mmol) followed by flash chromatography (silica gel, 8/2 *n*-hexane/EtOAc) afforded **12b** (41 mg, 74%) as a colorless solid: mp: 162–164 °C; ^{1}H NMR: δ = 7.18 (s, 1H), 7.05 (s, 1H), 6.31 (s, 1H), 4.62 (dd, *J* = 10.4, 5.5 Hz, 1H), 3.84 (dd, *J* = 13.4, 10.4 Hz, 1H), 3.23–3.29 (m, 1H), 2.71 (dd, *J* = 17.8, 6.8 Hz, 1H), 2.06 (dd, *J* = 17.8, 4.4 Hz, 1H); ^{13}C NMR: δ = 206.4, 169.7, 152.5, 135.7, 130.1, 127.0, 124.5, 121.4, 116.6, 70.3, 38.0, 36.6, 20.5, 15.9, IR (KBr, cm^{-1}): 2914, 1695, 1602, 1477, 1188, 752, 673; EIMS (*m*/*z*): 214 (100%, M^+). Anal. Calcd. for $C_{10}H_{14}O_2$: C, 78.48; H, 6.59. Found: C, 78.22; H, 6.60.

Epoxy ketone (14b)



Cyclization following the general procedure utilizing **11b** (101 mg, 0.51 mmol) CH_2Cl_2 (25 mL), $Co_2(CO)_8$ (340 mg, 0.99 mmol) and NMO (710 mg, 6.07 mmol) afforded **14b** (52 mg, 42%) after flash chromatography (silica gel, 90/9.9/0.1 n-hexane/EtOAc/Et_3N) as a colorless solid: mp: 80–82 °C; ^{1}H NMR: δ = 6.86 (s, 1H), 6.81 (s, 1H), 4.31 (bs, 1H), 3.80 (bs, 1H), 3.27 (app. d, *J* = 11.4 Hz, 1H), 2.84-2.89 (m, 1H), 2.68 (dd, *J* = 18.4, 10.1 Hz, 1H), 2.19 (s, 3H), 2.62–2.75 (m, 2H), 2.42 (app. d, *J* = 18.4 Hz, 1H), 2.24 (s, 3H), 1.96–2.00 (m, 1H); ^{13}C NMR: δ = 219.9; 154.4, 133.5, 131.0, 130.6, 129.9, 72.9, 50.6, 47.9, 35.5, 33.8, 31.6, 20.8, 16.1, IR (KBr, cm^{-1}): 2920, 1730, 1476, 1153, 786, 712; EIMS (*m*/*z*): 244 (74%, M^+), 162 (100%).

Enone (17b)



Cyclization following the general procedure utilizing **11c** (79 mg, 0.37 mmol) CH_2Cl_2 (15 mL), $Co_2(CO)_8$ (252 mg, 0.74 mmol) and NMO (518 mg, 4.43 mmol) followed by flash chromatography (silica gel, 8/2 *n*-hexane/EtOAc) afforded **17b** (18 mg, 21%) as an orange colored waxy oil: ^{1}H NMR: δ = 7.60 (s, 1H), 7.01 (s, 1H), 6.92 (s, 1H), 4.11 (dd, *J* = 5.3, 11.2 Hz, 1H), 3.45 (app. t, *J* = 8.5 Hz, 1H), 3.35–3.38 (m, 1H), 2.65 (dd, 1H, *J* = 5.7, 18.1 Hz), 2.28 (s, 3H), 2.19 (s, 3H), 2.01–2.05 (m, 1H), 1.84–1.89 (m, 1H), 1.66–1.69 (m, 1H), 1.28–1.38 (m, 1H); ^{13}C NMR: δ = 208.5, 166.9, 152.9, 133.3, 131.5, 130.7, 127.7, 126.8, 75.2, 40.0, 38.9, 33.0, 24.4, 20.9, 16.3; IR ($CHCl_3$, cm^{-1}): 3019, 1702, 1520, 1433, 1133, 754, 669; EIMS (*m*/*z*): 242 (100%, M^+).

Table S1. Crystal, Data Collection, and Refinement Parameters.

	14a	**17a**
formula	$C_{21}H_{28}O_3$	$C_{22}H_{30}O_2$
fw	328.4	326.5
crystal size, mm	0.46 x 0.24 x 0.16	0.42 x 0.36 x 0.16
a, Å	11.001(5)	12.156(2)
b, Å	10.587(7)	10.4419(13)
c, Å	16.211(2)	15.9723(6)
β, deg	99.692(3)	103.7395(9)
V, Å^3	1861.1(15)	1969.4(4)
Cell detn, reflns	5624	8721
cell detn, 2θ range, deg	24-52	4-52
d(calcd), g cm^{-3}	1.17	1.10
space group	$P2_1/a$	$P2_1/a$
Z	4	4
F000	712	712
radiation	MoKα, graphite mon	MoKα, graphite mon
λ, Å	0.7107	0.7107
temp, K	293	293
linear abs coeff, mm^{-1}	0.08	0.07
diffractometer	Rigaku Mercury CCD	Rigaku Mercury CCD
scan technique	ω	ω
2θ range, deg	4-52	4-52
h,k,l ranges	-13,13;-13,13;-19,19	-14,14;-12,12;-19,19
absorption correction	empirical	empirical
absorption range	0.67 - 1.00	0.88 - 1.00
refl meas	14662	15747
unique refls	3621	3873
R for merge	0.066	0.038
solution method	direct methods	direct methods
refls in refinement	3621	3873
parameters refined (on F^2)	223	224
R(F), (F>4σ(F))	0.078	0.061
R, Rw (on F^2, all reflns)	0.187, 0.257	0.132, 0.203
GOF	0.99	0.99
largest Δ/σ	0.00	0.00
H atom treatment	idealized	idealized
final diff map, e Å^{-3}	-0.21(5), +0.32(5)	-0.16(4), +0.28(4)
Programs used	Shelx-97	Shelx-97

Table S2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters (A^2 $x10^3$) for **14a**.

	x	y	z	U(eq)
O(1)	6514(3)	5976(2)	2165(2)	57(1)
O(2)	5416(3)	8109(3)	-99(2)	76(1)
O(3)	8296(3)	8195(3)	903(2)	64(1)
C(1)	6252(3)	7235(4)	2316(2)	49(1)
C(2)	5822(3)	7518(4)	3060(2)	51(1)
C(3)	5654(4)	8810(4)	3216(3)	57(1)
C(4)	5893(4)	9778(4)	2695(2)	52(1)
C(5)	6308(4)	9455(4)	1971(3)	56(1)
C(6)	6529(4)	8194(4)	1787(2)	52(1)
C(7)	7065(4)	7789(4)	1055(3)	53(1)
C(8)	8127(4)	6902(4)	1160(3)	58(1)
C(9)	7818(4)	5884(4)	505(3)	67(1)
C(10)	7094(4)	4895(4)	934(3)	65(1)
C(11)	6067(4)	5404(4)	1373(3)	61(1)
C(12)	6344(4)	7567(4)	200(3)	61(1)
C(13)	7005(5)	6542(4)	-214(3)	71(1)
C(21)	5547(4)	6495(4)	3678(3)	59(1)
C(22)	5156(7)	7061(6)	4461(4)	106(2)
C(23)	6712(5)	5722(5)	3995(3)	81(2)
C(24)	4525(5)	5613(5)	3240(3)	88(2)
C(25)	5735(4)	11154(4)	2947(3)	59(1)
C(26)	6614(6)	11438(5)	3759(4)	112(2)
C(27)	4469(6)	11377(6)	3142(6)	165(4)
C(28)	6007(11)	12045(5)	2300(5)	183(5)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **14a**.

O(1)-C(1)	1.394(5)
O(1)-C(11)	1.430(5)
O(2)-C(12)	1.200(5)
O(3)-C(8)	1.452(5)
O(3)-C(7)	1.481(5)
C(1)-C(6)	1.395(5)
C(1)-C(2)	1.401(5)
C(2)-C(3)	1.409(5)
C(2)-C(21)	1.540(5)
C(3)-C(4)	1.380(5)
C(4)-C(5)	1.373(5)
C(4)-C(25)	1.531(5)
C(5)-C(6)	1.397(5)
C(6)-C(7)	1.474(5)
C(7)-C(8)	1.487(6)
C(7)-C(12)	1.496(6)
C(8)-C(9)	1.511(6)
C(9)-C(13)	1.513(6)
C(9)-C(10)	1.550(6)
C(10)-C(11)	1.532(6)
C(12)-C(13)	1.523(6)
C(21)-C(22)	1.529(6)
C(21)-C(23)	1.535(6)
C(21)-C(24)	1.541(6)
C(25)-C(28)	1.479(8)
C(25)-C(27)	1.498(8)
C(25)-C(26)	1.527(7)

C(1)-O(1)-C(11)	120.8(3)
C(8)-O(3)-C(7)	60.9(2)
O(1)-C(1)-C(6)	121.0(3)
O(1)-C(1)-C(2)	117.7(3)
C(6)-C(1)-C(2)	120.9(4)
C(1)-C(2)-C(3)	115.8(3)
C(1)-C(2)-C(21)	122.8(4)
C(3)-C(2)-C(21)	121.3(3)
C(4)-C(3)-C(2)	124.5(4)
C(5)-C(4)-C(3)	117.6(4)
C(5)-C(4)-C(25)	122.3(4)
C(3)-C(4)-C(25)	120.1(4)
C(4)-C(5)-C(6)	121.0(4)
C(1)-C(6)-C(5)	120.0(4)
C(1)-C(6)-C(7)	116.2(3)
C(5)-C(6)-C(7)	123.9(4)
C(6)-C(7)-O(3)	123.4(3)
C(6)-C(7)-C(8)	120.4(4)
O(3)-C(7)-C(8)	58.6(2)
C(6)-C(7)-C(12)	124.8(3)
O(3)-C(7)-C(12)	104.3(3)
C(8)-C(7)-C(12)	106.9(3)
O(3)-C(8)-C(7)	60.5(2)
O(3)-C(8)-C(9)	119.7(4)

C(7)-C(8)-C(9)	106.9(4)
C(8)-C(9)-C(13)	104.3(4)
C(8)-C(9)-C(10)	103.8(3)
C(13)-C(9)-C(10)	112.0(4)
C(11)-C(10)-C(9)	116.5(4)
O(1)-C(11)-C(10)	113.4(3)
O(2)-C(12)-C(7)	125.5(4)
O(2)-C(12)-C(13)	126.9(4)
C(7)-C(12)-C(13)	107.6(4)
C(9)-C(13)-C(12)	104.7(3)
C(22)-C(21)-C(23)	105.5(4)
C(22)-C(21)-C(2)	112.2(4)
C(23)-C(21)-C(2)	110.4(3)
C(22)-C(21)-C(24)	109.5(4)
C(23)-C(21)-C(24)	109.5(4)
C(2)-C(21)-C(24)	109.6(4)
C(28)-C(25)-C(27)	110.8(6)
C(28)-C(25)-C(26)	108.2(5)
C(27)-C(25)-C(26)	105.3(5)
C(28)-C(25)-C(4)	111.9(4)
C(27)-C(25)-C(4)	110.9(4)
C(26)-C(25)-C(4)	109.5(4)

Table S4. Anisotropic displacement parameters ($A^2 \times 10^3$) for **14a**.

	U11	U22	U33	U23	U13	U12
O(1)	75(2)	41(2)	58(2)	1(1)	17(1)	4(1)
O(2)	83(2)	77(2)	67(2)	8(2)	8(2)	13(2)
O(3)	63(2)	61(2)	74(2)	-2(2)	26(1)	-7(1)
C(1)	55(2)	37(2)	54(2)	1(2)	7(2)	0(2)
C(2)	55(2)	47(2)	53(2)	2(2)	15(2)	-5(2)
C(3)	63(2)	54(3)	58(3)	-8(2)	18(2)	4(2)
C(4)	66(2)	41(2)	48(2)	-4(2)	10(2)	-1(2)
C(5)	66(2)	39(2)	63(3)	5(2)	10(2)	-4(2)
C(6)	57(2)	45(2)	54(2)	-4(2)	14(2)	-2(2)
C(7)	62(2)	37(2)	63(3)	-3(2)	21(2)	-6(2)
C(8)	55(2)	49(3)	74(3)	-2(2)	21(2)	-1(2)
C(9)	74(3)	57(3)	74(3)	-8(2)	26(2)	7(2)
C(10)	71(3)	49(3)	76(3)	-11(2)	20(2)	1(2)
C(11)	64(2)	48(2)	69(3)	-2(2)	12(2)	-6(2)
C(12)	75(3)	50(3)	59(3)	4(2)	18(2)	-4(2)
C(13)	94(3)	57(3)	63(3)	-15(2)	21(2)	-7(2)
C(21)	68(3)	54(3)	58(3)	11(2)	18(2)	-2(2)
C(22)	161(6)	82(4)	92(4)	12(3)	70(4)	8(4)
C(23)	84(3)	81(4)	79(3)	27(3)	14(3)	8(3)
C(24)	75(3)	87(4)	103(4)	19(3)	14(3)	-21(3)
C(25)	72(3)	40(2)	65(3)	-14(2)	12(2)	10(2)
C(26)	133(5)	71(4)	119(5)	-35(4)	-14(4)	14(4)
C(27)	96(5)	86(5)	309(12)	-86(6)	24(6)	20(4)

```
    C(28)   412(16)     39(3)     113(5)       7(3)      88(8)
36(6)
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The anisotropic displacement factor exponent takes the form:
pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

Table S5. H coordinates ($x10^4$) and isotropic displacement parameters (A^2 $x10^3$) for **14a**.

	x	y	z	U(eq)
H(3)	5362	9026	3703	69
H(5)	6445	10084	1598	67
H(8)	8498	6649	1728	70
H(9)	8558	5523	336	80
H(10A)	6734	4289	514	77
H(10B)	7678	4440	1343	77
H(11A)	5591	6023	1013	73
H(11B)	5518	4715	1454	73
H(13A)	6418	5955	-520	85
H(13B)	7496	6908	-597	85
H(22A)	5043	6396	4843	159
H(22B)	4395	7513	4306	159
H(22C)	5783	7629	4725	159
H(23A)	7011	5349	3528	122
H(23B)	6519	5067	4362	122
H(23C)	7334	6265	4293	122
H(24A)	4781	5241	2757	132
H(24B)	3782	6090	3070	132
H(24C)	4374	4959	3620	132
H(26A)	7447	11275	3683	167
H(26B)	6414	10909	4198	167
H(26C)	6535	12308	3907	167
H(27A)	4470	12132	3470	247
H(27B)	4230	10672	3451	247
H(27C)	3894	11470	2630	247
H(28A)	6785	11833	2144	275
H(28B)	6042	12890	2518	275
H(28C)	5369	11993	1818	275

Table S6. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($A^2 x10^3$) for **17a**.

	x	y	z	U(eq)
O(1)	3636(2)	5956(2)	2816(1)	58(1)
O(2)	5095(2)	6832(2)	4769(1)	79(1)
C(1)	3730(2)	7252(2)	2644(2)	49(1)
C(2)	4078(2)	7648(2)	1908(2)	54(1)
C(3)	4126(2)	8970(2)	1789(2)	56(1)
C(4)	3868(2)	9883(2)	2345(2)	51(1)
C(5)	3594(2)	9437(2)	3087(2)	52(1)
C(6)	3534(2)	8133(2)	3249(2)	47(1)
C(7)	3255(2)	7578(2)	4021(2)	48(1)
C(8)	2220(2)	7380(2)	4141(2)	55(1)
C(9)	2192(2)	6323(2)	4762(2)	59(1)
C(10)	1781(2)	5127(3)	4217(2)	65(1)
C(11)	2532(3)	4675(2)	3625(2)	66(1)
C(12)	2524(2)	5460(2)	2816(2)	63(1)
C(13)	4082(2)	6867(2)	4690(2)	55(1)
C(14)	3429(2)	6219(3)	5261(2)	61(1)
C(21)	4400(3)	6698(3)	1270(2)	70(1)
C(22)	4749(5)	7384(4)	530(3)	117(2)
C(23)	3393(4)	5830(4)	871(2)	102(1)
C(24)	5386(3)	5863(4)	1739(3)	103(1)
C(25)	3856(2)	11317(2)	2144(2)	58(1)
C(26)	4421(4)	12092(3)	2936(2)	100(1)
C(27)	4425(5)	11631(3)	1423(3)	131(2)
C(28)	2618(3)	11745(3)	1870(3)	108(1)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Table S7. Bond lengths (A) and angles (deg) for **17a**.

O(1)-C(1)	1.392(3)
O(1)-C(12)	1.448(3)
O(2)-C(13)	1.208(3)
C(1)-C(6)	1.395(3)
C(1)-C(2)	1.402(3)
C(2)-C(3)	1.397(4)
C(2)-C(21)	1.537(3)
C(3)-C(4)	1.389(4)
C(4)-C(5)	1.386(3)
C(4)-C(25)	1.530(3)
C(5)-C(6)	1.391(3)
C(6)-C(7)	1.474(3)
C(7)-C(8)	1.333(3)
C(7)-C(13)	1.481(4)
C(8)-C(9)	1.490(3)
C(9)-C(14)	1.528(4)
C(9)-C(10)	1.536(4)
C(10)-C(11)	1.536(4)
C(11)-C(12)	1.528(4)
C(13)-C(14)	1.504(4)
C(21)-C(22)	1.526(4)
C(21)-C(24)	1.526(5)
C(21)-C(23)	1.535(5)
C(25)-C(27)	1.514(4)
C(25)-C(26)	1.521(4)
C(25)-C(28)	1.531(4)
C(1)-O(1)-C(12)	117.87(19)
O(1)-C(1)-C(6)	118.0(2)
O(1)-C(1)-C(2)	120.5(2)
C(6)-C(1)-C(2)	121.4(2)
C(3)-C(2)-C(1)	115.9(2)
C(3)-C(2)-C(21)	121.4(2)
C(1)-C(2)-C(21)	122.7(2)
C(4)-C(3)-C(2)	124.6(2)
C(5)-C(4)-C(3)	116.9(2)
C(5)-C(4)-c(25)	121.0(2)
C(3)-C(4)-c(25)	122.0(2)
C(4)-C(5)-C(6)	121.5(2)
C(5)-C(6)-C(1)	119.4(2)
C(5)-C(6)-C(7)	125.0(2)
C(1)-C(6)-C(7)	115.6(2)
C(8)-C(7)-C(6)	126.4(2)
C(8)-C(7)-C(13)	108.7(2)
C(6)-C(7)-C(13)	123.4(2)
C(7)-C(8)-C(9)	112.4(2)
C(8)-C(9)-C(14)	103.0(2)
C(8)-C(9)-C(10)	106.3(2)
C(14)-C(9)-C(10)	112.6(2)
C(11)-C(10)-C(9)	116.0(2)
C(12)-C(11)-C(10)	118.0(2)
O(1)-C(12)-C(11)	111.9(2)
O(2)-C(13)-C(7)	126.2(2)

O(2)-C(13)-C(14)	126.5(3)
C(7)-C(13)-C(14)	107.3(2)
C(13)-C(14)-C(9)	104.3(2)
C(22)-C(21)-C(24)	108.5(3)
C(22)-C(21)-C(23)	107.2(3)
C(24)-C(21)-C(23)	108.8(3)
C(2)-C(21)-C(2)	111.8(2)
C(24)-C(21)-C(2)	109.7(2)
C(23)-C(21)-C(2)	110.7(2)
C(27)-C(25)-C(26)	108.6(3)
C(27)-C(25)-C(4)	112.8(2)
C(26)-C(25)-C(4)	111.5(2)
C(27)-C(25)-C(28)	108.3(3)
C(26)-C(25)-C(28)	107.6(3)
C(4)-C(25)-C(28)	107.8(2)

Table S8. Anisotropic displacement parameters (A^2 x10^3) for **17a**.

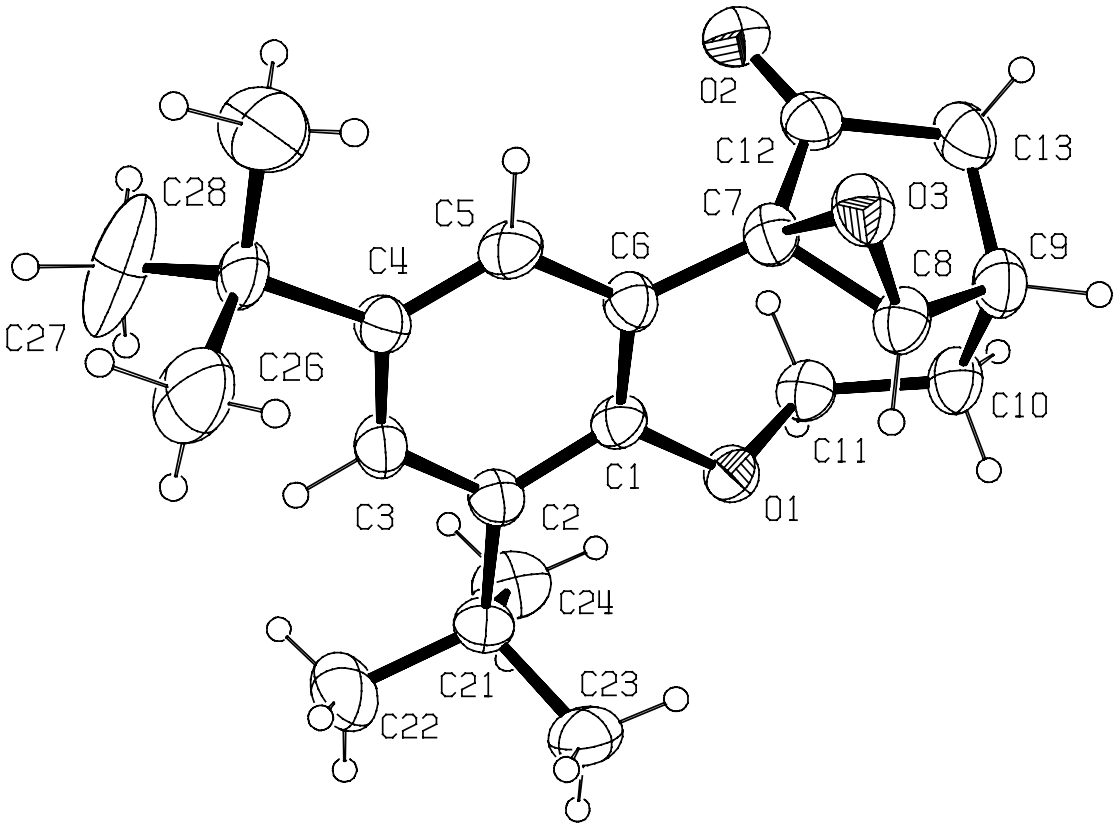
	U11	U22	U33	U23	U13	U12
O(1)	67(1)	45(1)	66(1)	0(1)	22(1)	2(1)
O(2)	51(1)	100(2)	82(2)	17(1)	11(1)	-6(1)
C(1)	53(1)	43(1)	52(1)	1(1)	15(1)	2(1)
C(2)	61(2)	53(1)	52(1)	-2(1)	21(1)	3(1)
C(3)	66(2)	59(2)	48(1)	4(1)	21(1)	-2(1)
C(4)	53(1)	50(1)	50(1)	4(1)	14(1)	-2(1)
C(5)	58(2)	48(1)	52(1)	-2(1)	16(1)	-2(1)
C(6)	48(1)	48(1)	48(1)	1(1)	16(1)	0(1)
C(7)	56(2)	43(1)	48(1)	-4(1)	18(1)	-2(1)
C(8)	53(2)	56(1)	62(2)	8(1)	21(1)	8(1)
C(9)	56(2)	64(2)	62(2)	12(1)	27(1)	3(1)
C(10)	56(2)	66(2)	74(2)	13(1)	14(1)	-8(1)
C(11)	75(2)	48(1)	75(2)	2(1)	20(2)	-8(1)
C(12)	71(2)	53(1)	63(2)	-4(1)	14(1)	-12(1)
C(13)	53(2)	60(1)	52(1)	-2(1)	13(1)	-9(1)
C(14)	65(2)	68(2)	51(1)	11(1)	15(1)	-5(1)
C(21)	91(2)	68(2)	59(2)	-7(1)	33(2)	5(2)
C(22)	191(4)	98(3)	91(3)	-10(2)	93(3)	3(3)
C(23)	128(3)	108(3)	71(2)	-30(2)	29(2)	-9(2)
C(24)	106(3)	108(3)	102(3)	-16(2)	40(2)	37(2)
C(25)	65(2)	49(1)	58(2)	7(1)	12(1)	-3(1)
C(26)	136(3)	54(2)	93(2)	2(2)	-6(2)	-11(2)
C(27)	215(5)	69(2)	146(4)	21(2)	118(4)	-8(3)

C(28) 89(3) 66(2) 152(4) 21(2) -7(2) 3(2)

The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

Table S9. H coordinates ($x10^4$) and isotropic displacement parameters (A^2 $x10^3$) for **17a**.

	x	y	z	U(eq)
H(3)	4347	9259	1302	67
H(5)	3447	10022	3485	62
H(8)	1586	7850	3868	67
H(9)	1695	6530	5144	70
H(10A)	1713	4431	4603	78
H(10B)	1030	5300	3863	78
H(11A)	3307	4640	3966	79
H(11B)	2311	3806	3445	79
H(12A)	2260	4927	2311	75
H(12B)	1999	6169	2782	75
H(14A)	3547	6649	5813	73
H(14B)	3654	5329	5357	73
H(22A)	4918	6763	136	175
H(22B)	5408	7898	755	175
H(22C)	4140	7923	234	175
H(23A)	3251	5254	1302	152
H(23B)	3565	5346	407	152
H(23C)	2734	6346	654	152
H(24A)	5187	5446	2218	154
H(24B)	6043	6389	1944	154
H(24C)	5547	5230	1349	154
H(26A)	4037	11940	3388	150
H(26B)	4382	12986	2793	150
H(26C)	5199	11838	3127	150
H(27A)	5208	11385	1589	196
H(27B)	4373	12535	1311	196
H(27C)	4057	11174	912	196
H(28A)	2241	11280	1364	162
H(28B)	2587	12646	1745	162
H(28C)	2249	11578	2327	162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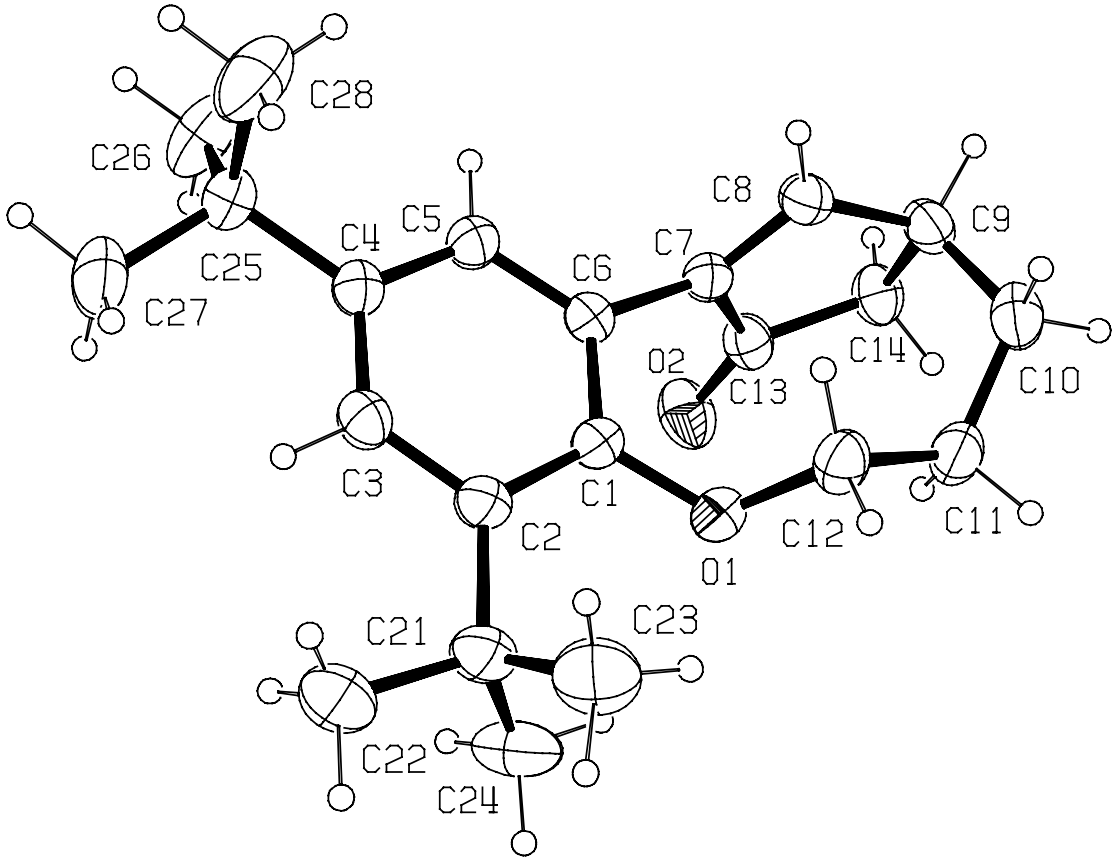
C13
C28
C5
C7
O3
C4
C6
C8
C9
C27
C26
C10
C11
C1
C3
C2
O1
C24
C21
C23
C22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C26
C25
C27
C4
C5
C6
C7
C8
C9
C10
C14
O2
C13
C3
C1
C2
O1
C12
C11
C21
C23
C22
C24