Supporting Information for:

Synthesis of furanophane derivatives through [8+2]-cycloaddition of dienylisobenzofurans and alkynes

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General Experimental. Nuclear Magnetic Resonance (¹H and ¹³C) spectra were recorded on a Varian 200 MHz or a 400 MHz spectrometer. Chemical shifts are reported in parts per million (δ) downfield from the reference tetramethylsilane. Coupling constants (J) are reported in hertz (Hz). The following symbols have been used to indicate multiplicities: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). A Nicolet Perkin-Elmer 1720X spectrometer was used to record the infrared spectra and band positions are reported in reciprocal centimeters (cm⁻¹). Only key diagnostic bands are reported, C-H stretching frequencies in the region 2800-3100 cm⁻¹ are not reported. Mass spectra (MS) were obtained at the University of California at Riverside or at the University of Nebraska. Flash column chromatography was performed using thick walled glass columns and “flash grade” silica. Thin layer chromatography was done using precoated 0.25mm silica gel plates purchased from Sorbtech. The relative proportion of solvents in mixed chromatography solvents refers to the volume: volume ratio. All commercially available reagents were purchased in reagent grade and used without purification. Diethyl ether, THF and dioxane were distilled from sodium benzophenone ketyl. All reactions were performed in an inert atmosphere created by a slight positive pressure (ca. 0.1 psi) of nitrogen.

Starting Material Preparations. Compounds **1A**,¹ **1C**,² **1D**,³ **2A**,⁴ **2B**,⁵ **2C**,⁶ and **7**⁷ were prepared based on literature preparations. The preparation for all other compounds is in this document.

General Procedure I: formation of 4 and 5. To a solution of carbene complex **2** (0.80 mmol) in dioxane (50 mL) was added alkyne **1** (0.60 mmol), and the reaction was heated to 85°C. Once the coupling reaction was complete (disappearance of alkyne **1** by TLC analysis), dimethyl acetylenedicarboxylate (DMAD) was added immediately at the same temperature, and the reaction continued for 2 h. The solvent was removed on a rotary evaporator and the residue was purified by flash column chromatography on silica gel using ethyl acetate/hexanes (1:10) as

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³ Garcia, J.G.; Ramos, B.; Pratt, L.M.; Rodriguez, A. *Tetrahedron Lett.* **1995**, 36, 7391-7394.

⁴ Dötz, K.H.; Dietz, R. *Chem. Ber.* **1978**, 111, 2517-2526.

⁵ Dötz, K.H.; Kuhn, W.; Ackermann, K.. *Z. Naturforsch., B* **1983**, 38B, 1351-1356.

⁶ Chan, K.S.; Peterson, G.A.; Brandvold, T.A.; Faron, K.L.; Challener, C.A.; Hyldahl, C.; Wulff, W.D. *J. Organomet. Chem.* **1987**, 334, 9-57.

⁷ Sharp, J. T; Wilson, P.; Parsons, S., Gould, R. O. *J. Chem. Soc., Perkin Trans. 1*, **2000**, 1139-1148

eluent, to give products **4** and **5** in polarity sequence. In the case of product **4G**, **5C** and **4F**, ethyl acetate: hexane: triethylamine (1: 10: 0.1) was used as eluent.

Entry A of Table. General Procedure I was followed using alkyne **1A** (0.157 g, 0.60 mmol), carbene complex **2A** (0.242 g, 0.80 mmol) and DMAD (0.20 mL, 1.63 mmol). After final purification was achieved by flash chromatography, compounds **4A** (0.235 g, 76% yield) and **5A** (0.010g, 3% yield) were obtained.

Compound **4A**: ^1H NMR (C_6D_6): δ 7.90 (d, 2 H, $J = 7.2$ Hz), 7.60 (d, 1 H, $J = 8.0$ Hz), 7.20 (m, 3 H), 7.04 (m, 2 H), 6.89 (td, 1 H, $J = 7.2, 0.8$ Hz), 4.39 (m, 1 H), 3.42 (s, 3 H), 3.35 (s, 3 H), 3.04 (s, 3 H), 2.86-2.78 (m, 1 H), 2.72-2.65 (m, 2 H), 2.64-2.57 (m, 1 H), 1.92-1.86 (m, 1 H), 1.80-1.64 (m, 2 H), 1.62-1.49 (m, 2 H), 1.47 – 1.23 (m, 2 H), 1.00-0.96 (m, 1H), 0.92 (t, 3 H, $J = 7.2$ Hz); ^{13}C NMR (CDCl_3): δ 168.3, 167.5, 148.7, 147.1, 146.6, 142.4, 137.9, 137.1, 136.8, 130.1, 129.4, 129.2, 128.4, 127.5, 123.9, 122.8, 117.2, 92.1, 57.7, 51.8, 45.6, 33.8, 31.9, 31.3, 27.0, 24.8, 22.8, 14.3; ^{13}C NMR (C_6D_6): δ 167.9, 167.5, 148.9, 147.5, 147.2, 142.8, 138.6, 137.9, 136.8, 130.1,

129.3, 129.2, 128.5, 128.4, 128.0, 127.8, 127.2, 124.1, 122.6, 117.2, 92.2, 57.0, 51.2, 51.0, 45.8, 33.8, 32.1, 31.4, 27.0, 24.4, 22.7, 14.2; DEPT (C_6D_6): 57.0 (up), 51.2 (up), 51.0 (up), 45.8 (up), 33.8 (down), 32.1 (down), 31.4 (down), 27.0 (down), 24.4 (down), 22.7 (down), 14.2 (up); IR (KBr) 2951, 2868, 1724 cm^{-1} ; Ms (EI): m/z 514 (M, 36), 483 (7), 457 (30), 450 (19), 427 (62), 395 (60), 366 (57), 105 (100); HRMS calcd for $\text{C}_{32}\text{H}_{34}\text{O}_6$ 514.23533, found 514.23454.

Compound **5A**: ^1H NMR (CDCl_3): δ 8.02 (d, 2 H, $J = 4.2$ Hz), 7.51-7.18 (m, 7 H), 5.99 (br s, 1 H), 3.77 (s, 3 H), 3.42 (s, 3 H), 3.17 (s, 3 H), 2.51 (m, 4 H), 2.01-1.98 (m, 2 H), 1.17-0.71 (m, 9 H); ^1H NMR (C_6D_6): δ 8.03 (d, 2 H, $J = 7.2$ Hz), 7.64 (dd, 4 H, $J = 7.6, 6.2$ Hz), 6.99 (m, 3 H), 5.88 (t, 1H, $J = 2.2$ Hz), 3.41 (s, 3 H), 3.21 (s, 3 H), 3.19 (s, 3 H), 2.57 (m, 3 H), 2.27 (m, 2 H), 1.82 (m, 3 H), 1.33 (m, 4 H), 0.90 (t, 3 H, $J = 7.4$ Hz); LC-MS (CI): 515 (M+1, 100), 482 (M – CH_3OH , 48).

Entry B of Table. General Procedure I was followed using alkyne **1B** (see ahead) (0.132 g, 0.60 mmol), carbene complex **2A** (0.242 g, 0.80 mmol) and DMAD (0.20 mL, 1.63 mmol). After final purification was achieved by flash chromatography, compound **4B** (0.174 g, 61% yield) was obtained.

Compound **4B**: ^1H NMR (C_6D_6): δ 7.89 (d, 2 H, $J = 7.2$ Hz), 7.44 (d, 2 H, $J = 7.2$ Hz), 7.21 (m, 3 H), 6.94 (dd, 2 H, $J = 14.8$, 8.0 Hz), 4.44 (m, 1 H), 3.38 (s, 3 H), 3.33 (s, 3 H), 3.04 (s, 3 H), 2.79-2.68 (m, 2 H), 2.11 (s, 3 H), 1.96-1.92 (m, 1 H), 1.61-1.52 (m, 2 H), 1.00 – 0.97 (m, 1 H); ^{13}C NMR (C_6D_6): δ 167.6, 167.0, 148.3, 148.0, 146.5, 142.8, 138.0, 137.4, 136.1, 128.8, 127.8, 127.5, 127.0, 126.9, 123.7, 122.5, 111.0, 92.1, 56.5, 56.4, 50.7, 45.5, 33.6, 31.8, 24.1, 13.1; IR (KBr) 2951, 2879, 1741; 1726; Ms (EI): m/z 472 (M, 69), 457 (17), 413 (24), 395 (23), 385 (100), 366 (144), 353 (58), 247 (31), 194 (54); HRMS calcd for $\text{C}_{29}\text{H}_{28}\text{O}_6$ 472.188589, found 472.19043.

Entry C of Table. General Procedure I was followed using alkyne **1C** (0.124 g, 0.60 mmol), carbene complex **2A** (0.242 g, 0.080 mmol) and DMAD (0.20 mL g, 1.63 mmol). After final purification was achieved by flash chromatography, compounds **4C** (0.198 g, 72% yield) and **5C** (0.024 g, 9% yield) were obtained.

Compound **4C**: ^1H NMR (C_6D_6): δ 7.90 (d, 2 H, $J = 8.1$ Hz), 7.19-6.95 (m, 7 H), 5.78 (s, 1 H), 4.67 (m, 1 H), 3.47 (s, 3 H), 3.30 (s, 3 H), 3.12 (s, 3 H), 2.93 (dd, 1 H, $J = 18.6$, 7.2 Hz), 2.67 (m, 1 H), 1.96 (m, 1 H), 1.75-1.52 (m, 2 H), 1.02 (m, 1 H); ^{13}C NMR (CDCl_3): δ 168.6, 166.7, 152.2, 145.4, 144.5, 143.7, 137.6, 137.2, 136.7, 129.5, 129.2, 128.9, 128.7, 128.2, 127.7, 123.9, 120.1, 101.1, 93.8, 57.8, 51.9 (2x), 45.3, 34.0, 32.1, 31.3, 25.1; IR (KBr) 2950, 2852, 1770, 1737 cm^{-1} ; Ms (EI): m/z 458 (M, 4), 442 (6), 426 (8), 399 (28), 383 (26), 367 (21), 351 (64), 339 (100); HRMS: Calc for $\text{C}_{28}\text{H}_{26}\text{O}_6$ 458.172939, found 458.172448.

Compound **5C**: ^1H NMR (CDCl_3): δ 7.86 (d, 2H, $J = 7.9$ Hz), 7.49-7.05 (m, 7 H), 6.12 (br s, 1 H), 5.99 (s, 1 H), 3.77 (s, 3 H), 3.45 (s, 3 H), 3.36 (s, 3 H), 2.62 (m, 2 H), 2.53 (m, 2 H), 2.04 (quintet, 2 H, $J = 7.2$ Hz); LC-MS (CI): 459 (M+1, 42), 427 (M – CH_3OH , 90), 339 (100).

Entry D of Table. General Procedure I was followed using alkyne **1D** (0.100 g, 0.36 mmol), carbene complex **2A** (0.128 g, 0.42 mmol) and DMAD (0.150 g, 1.01 mmol). After final purification was achieved by flash chromatography, compounds **4D** (0.010 g, 5% yield) and **5D** (0.118 g, 62% yield) were obtained.

Compound **4D**: ^1H NMR (CDCl_3): δ 7.59 (d, 2 H, $J = 6.2$ Hz), 7.42 (d, 2 H, $J = 3.3$ Hz), 7.38 (m, 4 H), 6.93 (d, 1 H, $J = 7.4$ Hz), 3.88 (m, 1 H), 3.65 (s, 3 H), 3.45 (s, 3 H), 3.32 (s, 3 H), 2.50-1.50 (m, 6 H), 0.23 (s, 9 H); Ms (EI): m/z 530 (M, 5), 515 (38), 440 (13), 395 (10), 385 (100), 357 (11), 353 (58), 291 (13), 253 (56), 167(100); HRMS calcd for $\text{C}_{31}\text{H}_{34}\text{O}_6\text{Si}$ 530.212468 found 530.211742.

Compound **5D**: ^1H NMR (CDCl_3): δ 7.96 (d, 2 H, $J = 4.4$ Hz), 7.46-7.36 (m, 4 H), 7.21 (d, 1 H, $J = 1.3$ Hz), 7.05 (d, 2 H, $J = 7.7$ Hz), 5.94 (br s, 1 H), 3.73 (s, 3 H), 3.70 (s, 3 H), 3.38 (s, 3 H), 2.54 (dd, 4 H, $J = 7.1, 7.5$ Hz), 2.01 (m, 2 H); ^{13}C NMR (CDCl_3): δ 164.3, 164.2, 158.4, 158.1, 150.9, 148.9, 148.4, 137.4, 134.5, 128.2, 128.0, 127.7, 125.1, 121.9, 120.5, 106.7, 95.4, 92.0, 53.6, 51.5, 36.8, 32.7, 23.3, 2.2; IR (KBr) 2952, 2852, 1727, 1719; Ms (EI): m/z 530 (M, 3), 515 (29), 499 (22), 379 (71), 105 (60), 73 (100), 59 (40); HRMS calcd for $\text{C}_{31}\text{H}_{34}\text{O}_6\text{Si}$ 530.212468, found 530.210927.

Entry E of Table. General Procedure I was followed using alkyne **1E** (see ahead) (0.181 g, 0.60 mmol), carbene complex **2A** (0.242 g, 0.80 mmol) and DMAD (0.20 mL, 1.63 mmol). After final purification was achieved by flash chromatography, compounds **4E** (0.150 g, 45% yield) and **5E** (0.101 g, 30% yield) were obtained.

Compound **4E**: ^1H NMR (C_6D_6): δ 7.57 (d, 1 H, $J = 7.6$ Hz), 7.39 (d, 1 H, $J = 6.8$ Hz), 7.27 (s, 1 H), 7.21 (t, 1 H, $J = 7.2$ Hz), 7.08-6.98 (m, 4 H), 6.92 (t, 1 H, $J = 7.6$ Hz), 4.65 (m, 1 H), 3.42 (s, 3 H), 3.38 (s, 3 H), 3.06 (s, 3 H), 2.88-2.65 (m, 3 H), 2.58 (ddd, 1 H, $J = 14.4, 7.6, 5.6$ Hz), 2.04 (ddd, 1 H, $J = 8.4, 7.2, 7.2$ Hz), 1.78-1.54 (m, 4 H), 1.42 (m, 1 H), 1.38 (m, 1 H), 1.13 (m, 1 H), 0.90 (t, 3 H, $J = 7.2$ Hz); ^{13}C NMR (C_6D_6): δ 167.8, 166.8, 156.2, 152.8, 148.2, 147.5, 143.8, 143.5, 137.7, 135.1, 130.5, 128.6, 128.4, 127.3, 125.4, 124.0, 123.2, 123.0, 121.8, 117.7, 111.8, 109.8, 86.5, 57.0, 51.2, 51.1, 45.9, 34.0, 32.2, 31.3, 27.0,

24.5, 22.6, 14.2; DEPT: CH₃'s at 57.0, 51.2, 51.1, and 14.2, CH₂'s at 34.0, 32.2, 31.3, 27.0, 24.5, 22.6, CH's at 128.6, 128.4, 127.3, 125.4, 124.0, 123.3, 123.0, 121.8, 111.8, 109.8, 46.0; IR (KBr) 2959, 2871, 1734, 1724 cm⁻¹; Ms (EI): *m/z* 554(M, 100), 495 (16), 467 (98), 435 (25), 403 (65), 370 (47), 349 (19), 289 (28), 234 (57), 179 (18), 131 (28), 91 (24); HRMS calcd for C₃₄H₃₄O₇ 554.230454, found 554.228925.

Compound **5E**: ¹H NMR (C₆D₆): δ 7.71 (d, 1 H, *J* = 7.2 Hz), 7.64 (d, 1 H, *J* = 7.2 Hz), 7.36 (dd, 1 H, *J* = 6.8, 2.4 Hz), 7.30 (dd, 1 H, *J* = 7.2, 1.6 Hz), 7.10-7.00 (m, 4 H), 6.98 (t, 1 H, *J* = 7.2 Hz), 5.84 (t, 1 H, *J* = 2.0 Hz), 3.47 (s, 3), 3.21 (s, 3 H), 3.19 (s, 3 H), 2.60-2.40 (m, 4 H), 2.25 (m, 2 H), 1.77 (quintet, 2 H, *J* = 7.2 Hz), 1.69 (m, 2 H), 1.26 (m, 2 H), 0.75 (t, 3 H, *J* = 6.8 Hz); ¹³C NMR (C₆D₆): δ 163.8, 163.3, 158.9, 155.6, 151.4, 150.8, 150.2, 148.0, 147.0, 136.7, 134.7, 128.4, 128.3, 125.9, 125.1, 123.1, 122.6, 121.8, 121.7, 112.7, 111.7, 108.3, 94.9, 87.8, 54.8, 51.3, 36.3, 33.6, 33.0, 28.4, 23.8, 22.8, 13.8; IR (KBr) 2951, 2852, 1737, 1723 cm⁻¹; Ms (EI): *m/z* 554(M, 19), 495 (35), 463 (21), 435 (25), 403 (10), 377 (23), 349 (100), 273 (13), 234 (17), 164 (16), 145 (93), 95 (97); HRMS calcd for C₃₄H₃₄O₇ 554.230454, found 554.232056.

Entry F of Table. To a solution of alkyne **1A** (0.157g, 0.60 mmol) in dioxane (25 mL) was added carbene complex **2B** (0.276 g, 1.00 mmol) in dioxane (20 mL) dropwise at 85°C over 0.5 h. DMAD (0.20 mL, 1.63 mmol) was added and the reaction mixture was hereafter treated as described in General Procedure I. After final purification was achieved by flash chromatography, compounds **4F** (0.165 g, 56% yield) and **5F** (0.55g, 19%) were obtained.

Compound **4F**: ¹H NMR (C₆D₆): δ 7.89 (d, 2 H, *J* = 7.2 Hz), 7.51 (d, 1 H, *J* = 7.6 Hz), 7.20-7.09 (m, 4 H), 7.05 (dd, 1 H, *J* = 8.0, 6.8 Hz), 6.92 (dd, 1 H, *J* = 7.2, 7.6 Hz), 3.64 (br d, 1 H, *J* = 14.8 Hz), 3.37 (s, 3 H), 3.32 (s, 3 H), 3.07 (s, 3 H), 2.76 (d, 1 H, *J* = 14.8 Hz), 2.71 (dt, 1 H, *J* = 14.0, 8.0 Hz), 2.51 (dt, 1 H, *J* = 14.0, 6.4 Hz), 2.00 (s, 3 H), 1.57 (quintet, 2 H, *J* = 7.2 Hz), 1.36-1.21 (m, 2 H), 0.86 (t, 3 H, *J* = 6.8 Hz); ¹³C NMR (C₆D₆): δ 168.2, 167.6, 150.2, 150.0, 146.6, 141.9, 138.9, 137.0, 135.6, 128.9, 128.7, 128.4, 128.3, 124.1, 122.6, 119.2, 116.2, 91.7, 57.2, 51.5, 51.2, 37.9, 30.8, 26.9, 22.6, 19.6, 14.1; IR (KBr) 2957, 2871, 1722; Ms (EI): *m/z* 488 (M, 67), 445 (100), 429 (23), 385 (25), 239 (25), 189 (12), 105 (27); HRMS calcd for C₃₀H₃₂O₆ 488.219889, found 488.221745.

Compound **5F**: ^1H NMR (CDCl_3): δ 7.52 (d, 2 H, J = 7.6 Hz), 7.48-7.31 (m, 5 H), 7.05 (t, 1 H, J = 3.6 Hz), 5.37 (s, 1 H), 5.19 (s, 1H), 3.71 (s, 3 H), 3.62 (s, 3 H), 3.39 (s, 3 H), 2.29-2.26 (m, 2 H), 1.94 (s 3 H), 1.42-1.18 (m, 4 H) 0.82 (t, J = 7.2 Hz); ^{13}C NMR (C_6D_6): δ 164.3, 163.9, 157.7, 154.1, 151.2, 149.7, 149.3, 138.1, 134.9, 130.8, 128.6, 128.1, 127.6, 125.6, 125.3, 122.3, 121.5, 119.6, 112.0, 93.7, 92.7, 54.6, 51.1, 33.4, 28.6, 23.0, 22.1, 13.8; Ms (EI): m/z 488 (M, 2), 429 (8), 397 (7), 323 (11), 215 (5), 165 (19.28), 105 (100); HRMS calcd for $\text{C}_{30}\text{H}_{32}\text{O}_6$ 488.219889, found 488.220699

Entry G of Table. General Procedure I was followed using alkyne **1A** (0.157 g, 0.60 mmol), carbene complex **2C** (0.254 g, 0.80 mmol), and DMAD (0.20 mL, 1.63 mmol). After final purification was achieved by flash chromatography, compounds **4G** (0.125 g, 39% yield) was obtained.

Compound **4G**: ^1H NMR (C_6D_6): δ 7.89 (d, 2 H, J = 6.4 Hz), 7.57 (d, 1 H, J = 7.6 Hz), 7.23-7.13 (m, 3 H), 7.04 (dd, 1 H, J = 8.0, 6.8 Hz), 6.93 (d, 1 H, J = 7.2 Hz), 6.86 (dd, 1 H, J = 7.2, 6.8 Hz), 4.34 (dd, 1 H, J = 12.4, 5.6 Hz), 3.69 (s, 3 H), 3.58 (m, 2 H), 3.36 (s, 3 H), 3.07 (s, 3 H), 2.75 (t, 2 H, J = 7.2 Hz), 1.86-1.78 (m, 2 H), 1.68-1.52 (m, 2 H), 1.47-1.32 (m, 2 H), 1.20 – 1.14 (m, 1 H), 1.05-0.97 (m, 1 H), 0.94 (t, 3H, J = 7.6 Hz); ^{13}C NMR (C_6D_6): δ 167.6, 167.5, 146.9, 142.0, 141.0, 140.8, 138.5, 138.1, 136.1, 129.2, 129.1, 128.6, 128.4, 128.2, 127.1, 123.6, 122.6, 119.1, 91.9, 65.9, 59.3, 51.3, 51.1, 39.8, 31.8, 27.5, 25.8, 23.1, 23.0, 14.3; IR (KBr) 2928, 2869, 1728; Ms (EI): m/z 530 (M, 10), 499 (14), 458 (58), 411 (56), 362 (47), 339 (28), 265 (19), 227 (43), 194 (48), 165 (39), 105 (100), 59 (36); HRMS calcd for $\text{C}_{32}\text{H}_{34}\text{O}_7$ 530.230454, found 530.228394

Reaction in Scheme 2

Preparation of 2-bromophenyl(phenyl)methanol. A solution of phenylmagnesium bromide (15 mmol) in ether (30 mL) was added dropwise to a solution of 2-bromobenzaldehyde (2.775 g, 15 mmol) in ether (20 mL) at 0°C . After completion of the addition, the mixture was warmed to room temperature and stirring was continued for 5 h. Saturated aqueous ammonium chloride solution was added, and the aqueous phase was extracted with ether two times. The combined organic layers were dried over sodium sulfate. After removal of solvent on a rotary evaporator, the residue was purified via flash chromatography on silica gel using 10:1 hexane:ethyl acetate to afford 2-bromo- α -phenylbenzenemethanol (3.599 g, 91%). ^1H NMR (CDCl_3): δ 7.61 (d, 1H, J = 8.0 Hz), 7.57 (d, 1H, J = 8.0 Hz), 7.43- 7.27 (m, 6H), 7.19 (td, 1H, J = 8.0, 1.6 Hz), 6.22 (d, 1H,

$J = 4.0$ Hz), 2.40 (d, 1H, $J = 4.0$ Hz); ^{13}C NMR (CDCl_3): δ 142.8, 133.0, 129.3, 128.7, 128.0, 127.4, 127.3, 122.3, 74.9; IR (KBr, cm^{-1}) 3358, 1494, 1439. The spectral data were consistent with those previously reported for this compound.⁸

Preparation of methyl 2-ethenyl-1-cyclopentenecarboxylate. Carbon monoxide was bubbled through a solution of 1-bromo-2-ethenylcyclopentene (1.000 g, 5.78 mmol), $\text{Pd}(\text{PPh}_3)_4$ (0.646 g, 0.56 mmol), triethylamine (1.6 mL, 11.56 mmol), lithium chloride (0.245 g, 5.78 mmol), and dry methanol (10.3 mL, 229 mmol) in anhydrous DMF (38 mL) for 30 min.⁹ The mixture was heated at 85 °C for 15 h under 1 atmosphere of carbon monoxide (a balloon was placed over the reflux condenser). The solution was cooled to room temperature and ether (30 mL) was added. The mixture was filtered through a pad of Celite, and the pad rinsed with diethyl ether (3×30 mL). The combined filtrates were washed with water until neutral, dried over sodium sulfate,

concentrated, and purified by flash column chromatography using 10:1 hexane:ether as eluent to give methyl 2-ethenyl-1-cyclopentenecarboxylate (0.608 g, 69%). ^1H NMR (CDCl_3): δ 7.49 (m, 1H), 5.35 (dd, 2H, $J = 16.4, 5.6$ Hz), 3.50 (s, 3H), 3.10 (s, 3H), 2.64-2.56 (m, 4H), 1.81-1.74 (m, 2H). The spectral data were consistent with those previously reported for this compound.¹⁰

Preparation of 2-ethenyl-1-cyclopentenecarboxylic acid. A solution of 2-ethenyl-1-cyclopentenecarboxylate (1.214 g, 7.99 mmol) and lithium hydroxide hydrate (5.3 g, 126.19 mmol) in methanol (15 mL) and water (12 mL) was stirred for 12 h at room temperature, and then washed with hexanes (2×30 mL). The aqueous layer was acidified (pH 2) with hydrochloric acid, and extracted with diethyl ether (3×40 mL). The combined layers were washed with water (3×30 mL) and saturated aqueous sodium chloride solution (3×30 mL), dried over sodium sulfate, and concentrated on a rotary evaporator. The residue was purified by flash chromatography (silica gel, 2:1 hexane:ether) to afford 2-ethenyl-1-cyclopentenecarboxylic acid

(1.003 g, 91%). ^1H NMR (CDCl_3): δ 12.31 (br s, 1 H), 7.60 (dd, 1 H, $J = 18.0, 10.8$ Hz), 5.50 (d, 1 H, $J = 18.0$ Hz), 5.45 (d, 1 H, $J = 10.8$ Hz), 2.77 (t, 1 H, $J = 7.6$ Hz), 2.73 (t, 1 H, $J = 9.2$ Hz), 1.94-1.86 (m, 2 H); ^{13}C NMR (CDCl_3): δ 171.8, 155.0, 132, 129.5, 121.6, 34.3, 34.2, 21.3. The spectral data were in agreement with those previously reported for this compound.¹⁰

⁸ Effenberger, F.; Spiegler, W. *Chem. Ber.* **1985**, 118, 3872-3899.

⁹ This procedure was adapted from a literature procedure. Qing, F.; Yue, X. *Chin. J. Chem.* **2000**, 18, 76-87.

¹⁰ Hettrick, C.M.; Kling, J.K.; Scott, W.J. *J. Org. Chem.* **1991**, 56, 1489-1492.

Preparation of N-methoxy-N-methyl 2-ethenyl-1-cyclopentenecarboxamide.¹¹ To a solution of 2-ethenyl-1-cyclopentenecarboxylic acid (0.752 g, 5.45 mmol) in dichloromethane (15 mL) was added oxalyl chloride (1.90 mL, 10.9 mmol), followed by catalytic DMF (10 drops). The reaction was stirred at room temperature until no gas evolution was evident. The solution was distilled at ambient pressure. The crude acid chloride was dissolved in dichloromethane (15 mL) and cooled 0 °C. To this solution was added N-methyl-O-methylhydroxylamine hydrochloride (0.588 g, 6.00 mmol), followed by pyridine (1 mL, 13.08 mmol), and the reaction was warmed to room temperature and stirred overnight. The mixture was diluted with ethyl acetate and saturated aqueous cupric sulfate solution (15 mL) was added. The aqueous layer was extracted three times with ethyl acetate. The organic layers were dried over sodium sulfate. Concentration on a rotary evaporator afforded a yellow oil, which was purified by flash chromatography (silica gel, 3:1 hexane:ether). Purification afforded the Weinreb amide (0.709 g, 72%), and recovered 2-ethenyl-1-cyclopentenecarboxylic acid (0.126 g). Based on the recovery of acid, the yield of

the Weinreb amide is 86%. ¹H NMR (CDCl₃): δ 6.57 (dd, 1H, J = 18.0, 11.2 Hz), 5.12 (d, 1H, J = 14.6 Hz), 5.08 (d, 1H, J = 10.0 Hz), 3.50 (s, 3H), 3.10 (s, 3H), 2.62 (t, 2H, J = 7.2 Hz), 2.46 (t, 2H, J = 8.0 Hz), 1.85 (tt, 2H, J = 7.2, 7.6 Hz); ¹³C NMR (CDCl₃): δ 143.2, 135.1, 131.2, 117.5, 61.6, 35.4, 32.3, 22.6; IR (KBr) 2936, 2850, 1636; Ms (EI): *m/z* 181 (M, 16), 150 (9), 121 (100), 93 (76), 77 (44), 65 (17), 41 (7); HRMS calcd for C₁₀H₁₅NO₂ 181.110279, found 181.110297

In situ isobenzofuran formation – [8+2]-cycloaddition. A solution of 2-bromo- α -phenylbenzenemethanol (0.700 g, 2.66 mmol) in dry THF (20 mL) was cooled to -78 °C and *n*-butyllithium (3.30 mL of a 1.6 M solution in hexane, 5.32 mmol) was added. The mixture was stirred for 10 min at -78 °C and then warmed to 0 °C for 45 min. To this solution was added a solution of the Weinreb amide from the previous experiment (0.367 g, 2.03 mmol) in THF (10 mL) at 0 °C. The reaction was allowed to warm to room temperature and stirred for 12 h. The reaction was diluted with diethyl ether (30 mL), and a saturated aqueous ammonium chloride solution was added. The organic layer was separated, and the aqueous was extracted three times with ether. The organic layers were combined and then dried over sodium sulfate. The solvent was removed and residue purified with column chromatography (flash silica gel, ether: hexanes: triethylamine 1:3:0.1), give a mixture of keto alcohol **8** and hemiketal from **8** (0.460g, 75%). Although this substance is a single spot by TLC analysis, the NMR spectra of this substance was very complex due to the presence of keto-alcohol and the α and β anomers of the hemiketal. To a solution keto alcohol **8** (0.110 g, 0.36 mmol) in toluene (35 mL) was added flash silica gel (0.050 g) and the mixture was heated to 85 °C.¹² After 30 min, DMAD (0.200 g, 1.40 mmol) was added and heating was continued for 1h. After the solution had cooled to room temperature, excess diethylamine was added. The mixture was concentrated *in vacuo* and purified via flash chromatography using ethyl acetate: hexane: triethylamine (1:10:0.1) to afford **10** as a white solid (0.099 g, 64%). If triethylamine was not used in the course of purification, **10** was obtained in 46% yield.

¹¹ This is an adaptation of a literature procedure. Brunette, S. R.; Lipton, M. A. *J. Org. Chem.* **2000**, 65, 5114-5119.

¹² For isobenzofuran generation from similar precursors, see Smith, J. G.; Fogg, D. E.; Munday, I. J.; Sandborn, R. E.; Dibble, P. W. *J. Org. Chem.* **1988**, 53 2942-2953.

^1H NMR (C_6D_6): δ 7.96 (d, 2 H, J = 6.8 Hz), 7.29-7.05 (m, 6 H), 6.93 (dd, 1 H, J = 8.0, 6.8 Hz), 5.72 (d, 1 H, J = 9.6 Hz), 4.10 (d, 1 H, J = 14.0 Hz), 3.30 (s, 3 H), 3.08 (s, 3 H), 2.96 (dd, 1 H, J = 14.0, 9.6 Hz), 2.56-2.38 (m, 3 H), 2.35-2.30 (m, 1 H), 1.61-1.55 (m, 1 H), 1.50 – 1.40 (m, 1 H), ^{13}C NMR (C_6D_6): δ 168.3, 168.2, 145.5, 144.8, 144.3, 142.6, 138.9, 137.4, 136.7, 129.0, 128.9, 128.5, 128.4, 127.2, 124.1, 121.2, 117.7, 115.8, 92.9, 51.7, 51.1, 37.9, 32.8, 29.3, 24.2; IR (KBr) 2952, 2852, 1727, 1719 cm^{-1} ; Ms (EI): m/z 428 (M, 58), 396 (18), 369 (98), 337 (76), 309 (100), 281 (42), 265 (13), 203 (26), 189 (7), 117 (4), 71 (12); HRMS calcd for $\text{C}_{27}\text{H}_{24}\text{O}_5$ 428.162374, found 428.163356.

The unusual coupling pattern observed for the lettered protons of **10** (see picture) is consistent with the conformation obtained by energy minimization through Hartree-Fock 6-31G calculations. The observed coupling constant between H_A and H_B is 0 Hz, and the dihedral angle is 112° . The observed coupling constant between H_A and H_C is 9.6 Hz, and the dihedral angle is -4° .

Alkyne 1E. To a solution 2,3-benzofuran (1.207 g, 10.21 mmol) in THF (10 mL) was added *n*-butyllithium (6.50 mL of a 1.6M hexane solution, 10.4 mmol) dropwise, at 0°C .¹³ The solution was then warmed to room temperature and after 1.5 h cooled to 0°C . To this solution was added a solution of 2-(1-hexyn-1-yl)-N-methoxy-N-methylbenzamide¹¹ (1.845 g, 8.96 mmol) in THF (7 mL). After the addition was complete, the reaction was allowed to

warm to room temperature and stirred for 3 h. The reaction was diluted with ether and a saturated aqueous ammonium chloride solution (15 mL) was added. The aqueous phase was extracted three times with ether. The combined organic layers were dried over sodium sulfate and concentrated on a rotary evaporator. The resulting residue was purified via flash chromatography using hexane: ethyl acetate (15:1) as eluent to afford **1E** (2.000 g, 74%). ^1H NMR (CDCl_3): δ 7.65 (dd, 1 H, J = 8.0, 1.2 Hz), 7.58 (dt, 1 H, J = 8.0, 0.8 Hz), 7.52 (br d, 1 H, J = 8.0 Hz), 7.50 (br d, 1 H, J = 8.0 Hz), 7.45 (td, 1 H, J = 8.0, 1.2 Hz), 7.43 (td, 1 H, J = 8.0, 1.2 Hz), 7.36 (td, 1 H, J = 8.0, 1.2 Hz), 7.30 (d, 1 H, J = 0.8 Hz), 7.27 (td, 1 H, J = 8.0, 0.8 Hz), 2.15 (t, 2 H, J = 6.8 Hz), 1.25-1.14 (m, 4 H), 0.66 (t, 3 H, J = 7.2 Hz); ^{13}C NMR (CDCl_3): δ 186.0, 156.3, 152.6, 140.6, 133.1, 130.7, 128.6, 128.2, 127.4, 127.3, 124.0, 123.5, 123.0, 117.3, 112.7, 96.5, 78.3, 30.4, 21.9, 19.2, 13.5; Ms (EI): m/z 302 (M, 100), 273 (33), 260 (88), 202 (50), 130 (11), 69 (19); HRMS calcd for $\text{C}_{21}\text{H}_{18}\text{O}_2$ 302.130680, found 302.130251

¹³ This is an adaptation of a literature procedure. Yokoyama, M.; Lkeue, T.; Ochiai, Y.; Momotake, A.; Yamaguchi, K.; Togo, H. *J. Chem. Soc., Perkin Trans. 1*, **1998**, 2185-2191.

Alkyne 1B. A mixture of palladium (II) chloride (0.065 g, 0.37 mmol) and triphenylphosphine (0.194 g, 0.74 mmol) in diethylamine (10 mL) was stirred at room temperature for 10 min, then 2-iodobenzophenone (2.758 g, 8.95 mmol), copper (I) iodide (0.065 g, 0.34 mmol) and diethylamine (30 mL) were added. A slow current of methylacetylene, which was prepared from 1,2-dibromopropane and KOH in refluxing n-butanol, was immediately passed through the reaction mixture without isolation.¹⁴ The mixture was refluxed for 5h under 1 atmosphere of methylacetylene (dry ice condenser placed over the reflux condenser). After the reaction was complete, the solvent was evaporated and the residue was purified by flash column chromatography using hexane:ethyl acetate (10:1) as eluent to afford **1D** (1.628 g, 82%). ¹H NMR (CDCl₃): δ 7.82 (m, 2H), 7.56-7.36 (m, 7H), 1.75 (s, 3H); ¹³C NMR (CDCl₃): δ 197.2, 141.2, 137.4, 132.9, 132.7, 130.0, 128.2, 127.2, 122.6, 91.8, 77.7, 4.1; IR (KBr) 3061, 2916, 2850, 1668 cm⁻¹; Ms (EI): *m/z* 220 (M, 100), 191 (53), 165 (11), 143 (27), 115 (28); HRMS calcd for C₂₁H₁₈NO₂ 220.088815, found 220.088589.

X-ray Data for Compound 4B.

Ortep Drawing for Compound 4B

¹⁴ For generation of propyne using this procedure, see Sashida, H.; Sadamori, K.; Tsuchiya, T. *Synth. Commun.* **1998**, 28, 713-727.

Table 1. Crystal data and structure refinement for C₂₉H₂₈O₆.

Identification code	a:nmsultm
Empirical formula	C ₂₉ H ₂₈ O ₆
Formula weight	472.51
Temperature	223 (2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 10.5671(16) Å alpha = 76.184 (3)
deg.	b = 11.0060(17) Å beta = 65.340 (2)
deg.	c = 11.9493(18) Å gamma = 87.249 (3)
deg.	
Volume	1224.3 (3) Å ³
Z, Calculated density	2, 1.282 Mg/m ³
Absorption coefficient	0.089 mm ⁻¹
F(000)	500
Crystal size	0.48 x 0.32 x 0.10 mm
Theta range for data collection	1.91 to 24.73 deg.
Index ranges	-12 ≤ h ≤ 12, -12 ≤ k ≤ 10, -12 ≤ l ≤ 14
Reflections collected / unique	6303 / 4129 [R(int) = 0.0197]
Completeness to 2theta = 24.73	98.6%
Absorption correction	Not applied
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4129 / 0 / 320
Goodness-of-fit on F ²	0.496
Final R indices [I > 2sigma(I)]	R ₁ = 0.0472, wR ₂ = 0.1274
R indices (all data)	R ₁ = 0.0610, wR ₂ = 0.1514
Largest diff. peak and hole	0.196 and -0.208 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C₂₉H₂₈O₆. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	8208(1)	2199(1)	1456(1)	30(1)
O(2)	9165(2)	4161(2)	3558(2)	53(1)
O(3)	4384(2)	1506(2)	1230(2)	50(1)
O(4)	3508(2)	2017(2)	3102(2)	46(1)
O(5)	4735(2)	4423(1)	3665(1)	36(1)
O(6)	3921(2)	4787(2)	2164(2)	44(1)
C(8)	7125(2)	1774(2)	1161(2)	31(1)
C(1)	7825(2)	1690(2)	2745(2)	33(1)
C(2)	8018(2)	2383(2)	3448(2)	39(1)
C(3)	8454(2)	3729(2)	2951(2)	39(1)
C(4)	8178(2)	4617(2)	2112(2)	36(1)
C(5)	7403(2)	4530(2)	1297(2)	31(1)
C(6)	6049(2)	3731(2)	1845(2)	27(1)
C(7)	5898(2)	2610(2)	1651(2)	28(1)
C(9)	6735(2)	470(2)	2055(2)	36(1)
C(10)	7112(2)	465(2)	3045(2)	39(1)
C(11)	6802(3)	-591(2)	4044(2)	54(1)
C(12)	6153(3)	-1630(3)	4015(3)	66(1)
C(13)	5818(3)	-1636(2)	3020(3)	63(1)
C(14)	6090(3)	-578(2)	2023(3)	48(1)
C(15)	7209(2)	5916(2)	759(2)	41(1)
C(16)	8533(3)	6592(2)	563(3)	50(1)
C(17)	8730(3)	5975(2)	1753(3)	49(1)
C(18)	7765(3)	1789(3)	4809(2)	54(1)
C(19)	10550(3)	3753(4)	3209(3)	70(1)
C(20)	4465(2)	2029(2)	2101(2)	32(1)
C(21)	4767(2)	4360(2)	2551(2)	30(1)
C(22)	3070(3)	833(3)	1604(3)	67(1)
C(23)	3529(3)	4979(3)	4464(2)	51(1)
C(24)	7791(2)	1832(2)	-248(2)	31(1)
C(25)	8816(2)	1005(2)	-673(2)	38(1)
C(26)	9562(2)	1092(3)	-1954(2)	47(1)
C(27)	9284(3)	1998(3)	-2824(2)	49(1)
C(28)	8253(3)	2804(3)	-2407(2)	49(1)
C(29)	7501(2)	2726(2)	-1123(2)	39(1)

Table 3. Bond lengths [Å] and angles [deg] for C₂₉H₂₈O₆.

O(1)-C(1)	1.393(2)
O(1)-C(8)	1.456(2)
O(2)-C(3)	1.403(3)
O(2)-C(19)	1.427(3)
O(3)-C(20)	1.337(3)
O(3)-C(22)	1.448(3)
O(4)-C(20)	1.199(3)
O(5)-C(21)	1.337(3)
O(5)-C(23)	1.449(3)
O(6)-C(21)	1.198(3)
C(8)-C(24)	1.516(3)
C(8)-C(9)	1.526(3)
C(8)-C(7)	1.543(3)
C(1)-C(2)	1.339(3)
C(1)-C(10)	1.468(3)
C(2)-C(3)	1.475(4)
C(2)-C(18)	1.513(3)
C(3)-C(4)	1.340(3)
C(4)-C(17)	1.525(3)
C(4)-C(5)	1.533(3)
C(5)-C(6)	1.520(3)
C(5)-C(15)	1.548(3)
C(6)-C(7)	1.337(3)
C(6)-C(21)	1.504(3)
C(7)-C(20)	1.497(3)
C(9)-C(10)	1.395(3)
C(9)-C(14)	1.384(3)
C(10)-C(11)	1.389(3)
C(11)-C(12)	1.376(4)
C(12)-C(13)	1.375(5)
C(13)-C(14)	1.393(4)
C(15)-C(16)	1.520(3)
C(16)-C(17)	1.516(4)
C(24)-C(25)	1.388(3)
C(24)-C(29)	1.379(3)
C(25)-C(26)	1.379(3)
C(26)-C(27)	1.376(4)
C(27)-C(28)	1.374(4)
C(28)-C(29)	1.386(3)
C(1)-O(1)-C(8)	106.58(15)
C(3)-O(2)-C(19)	113.18(19)
C(20)-O(3)-C(22)	115.43(19)
C(21)-O(5)-C(23)	115.70(17)
O(1)-C(8)-C(24)	106.33(15)
O(1)-C(8)-C(9)	100.52(16)
C(24)-C(8)-C(9)	116.56(17)
O(1)-C(8)-C(7)	105.31(15)
C(24)-C(8)-C(7)	117.82(17)
C(9)-C(8)-C(7)	108.19(16)
C(2)-C(1)-O(1)	119.6(2)
C(2)-C(1)-C(10)	133.8(2)
O(1)-C(1)-C(10)	106.23(17)

C(1)-C(2)-C(3)	122.4(2)
C(1)-C(2)-C(18)	119.8(2)
C(3)-C(2)-C(18)	117.9(2)
C(4)-C(3)-O(2)	115.2(2)
C(4)-C(3)-C(2)	131.0(2)
O(2)-C(3)-C(2)	113.5(2)
C(3)-C(4)-C(17)	121.9(2)
C(3)-C(4)-C(5)	130.8(2)
C(17)-C(4)-C(5)	107.26(19)
C(6)-C(5)-C(4)	121.23(18)
C(6)-C(5)-C(15)	111.54(17)
C(4)-C(5)-C(15)	103.41(17)
C(7)-C(6)-C(21)	118.85(18)
C(7)-C(6)-C(5)	126.31(18)
C(21)-C(6)-C(5)	114.51(17)
C(6)-C(7)-C(20)	119.61(18)
C(6)-C(7)-C(8)	123.26(17)
C(20)-C(7)-C(8)	116.50(17)
C(10)-C(9)-C(14)	121.2(2)
C(10)-C(9)-C(8)	107.75(19)
C(14)-C(9)-C(8)	131.0(2)
C(9)-C(10)-C(11)	120.1(2)
C(9)-C(10)-C(1)	106.64(19)
C(11)-C(10)-C(1)	133.2(2)
C(12)-C(11)-C(10)	118.6(3)
C(13)-C(12)-C(11)	121.2(3)
C(12)-C(13)-C(14)	121.1(3)
C(9)-C(14)-C(13)	117.7(3)
C(16)-C(15)-C(5)	103.26(19)
C(17)-C(16)-C(15)	102.9(2)
C(16)-C(17)-C(4)	105.62(19)
O(4)-C(20)-O(3)	123.60(19)
O(4)-C(20)-C(7)	125.72(19)
O(3)-C(20)-C(7)	110.68(17)
O(6)-C(21)-O(5)	124.38(19)
O(6)-C(21)-C(6)	125.74(19)
O(5)-C(21)-C(6)	109.83(17)
C(25)-C(24)-C(29)	119.1(2)
C(25)-C(24)-C(8)	117.81(19)
C(29)-C(24)-C(8)	122.91(19)
C(26)-C(25)-C(24)	120.7(2)
C(25)-C(26)-C(27)	120.0(2)
C(28)-C(27)-C(26)	119.5(2)
C(27)-C(28)-C(29)	120.9(2)
C(28)-C(29)-C(24)	119.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C₂₉H₂₈O₆.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
O(1)	26(1)	38(1)	29(1)	-11(1)	-14(1)	6(1)
O(2)	38(1)	85(1)	59(1)	-41(1)	-30(1)	14(1)
O(3)	39(1)	66(1)	49(1)	-28(1)	-15(1)	-11(1)
O(4)	32(1)	62(1)	40(1)	-16(1)	-7(1)	-8(1)
O(5)	31(1)	47(1)	37(1)	-22(1)	-15(1)	11(1)
O(6)	37(1)	56(1)	49(1)	-18(1)	-24(1)	19(1)
C(8)	29(1)	33(1)	33(1)	-12(1)	-14(1)	2(1)
C(1)	28(1)	45(1)	28(1)	-10(1)	-13(1)	13(1)
C(2)	32(1)	60(2)	33(1)	-21(1)	-17(1)	18(1)
C(3)	26(1)	62(2)	40(1)	-30(1)	-16(1)	10(1)
C(4)	23(1)	49(1)	41(1)	-24(1)	-12(1)	3(1)
C(5)	27(1)	34(1)	33(1)	-13(1)	-12(1)	4(1)
C(6)	26(1)	32(1)	28(1)	-8(1)	-14(1)	6(1)
C(7)	27(1)	33(1)	28(1)	-9(1)	-13(1)	4(1)
C(9)	33(1)	32(1)	37(1)	-13(1)	-6(1)	8(1)
C(10)	36(1)	42(1)	32(1)	-10(1)	-9(1)	16(1)
C(11)	58(2)	46(2)	40(1)	-2(1)	-10(1)	18(1)
C(12)	73(2)	42(2)	47(2)	5(1)	1(1)	12(1)
C(13)	63(2)	29(1)	66(2)	-11(1)	4(2)	-1(1)
C(14)	48(1)	36(1)	50(2)	-19(1)	-6(1)	3(1)
C(15)	37(1)	37(1)	45(1)	-12(1)	-13(1)	1(1)
C(16)	43(1)	41(1)	56(2)	-15(1)	-10(1)	-7(1)
C(17)	37(1)	52(2)	61(2)	-27(1)	-17(1)	-4(1)
C(18)	59(2)	74(2)	38(1)	-20(1)	-26(1)	20(1)
C(19)	36(1)	112(3)	81(2)	-43(2)	-35(2)	13(2)
C(20)	30(1)	35(1)	34(1)	-10(1)	-15(1)	1(1)
C(21)	26(1)	33(1)	33(1)	-11(1)	-12(1)	2(1)
C(22)	51(2)	84(2)	74(2)	-35(2)	-25(2)	-20(2)
C(23)	42(1)	66(2)	48(2)	-33(1)	-14(1)	19(1)
C(24)	28(1)	34(1)	33(1)	-13(1)	-14(1)	-1(1)
C(25)	33(1)	48(1)	38(1)	-16(1)	-17(1)	10(1)
C(26)	32(1)	70(2)	44(1)	-29(1)	-14(1)	14(1)
C(27)	43(1)	74(2)	31(1)	-21(1)	-13(1)	-1(1)
C(28)	58(2)	58(2)	38(1)	-12(1)	-27(1)	7(1)
C(29)	42(1)	42(1)	38(1)	-16(1)	-20(1)	10(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for C₂₉H₂₈O₆.

	x	y	z	U(eq)
H(5)	8064	4213	569	37
H(11)	7031	-596	4725	64
H(12)	5933	-2348	4687	79
H(13)	5399	-2367	3013	76
H(14)	5844	-577	1352	58
H(15A)	7124	6024	-45	49
H(15B)	6380	6222	1362	49
H(16A)	9326	6469	-199	60
H(16B)	8416	7493	491	60
H(17A)	8206	6391	2437	58
H(17B)	9719	6016	1588	58
H(18A)	6940	1221	5207	81
H(18B)	7628	2438	5267	81
H(18C)	8564	1327	4826	81
H(19A)	10527	2847	3354	104
H(19B)	10955	4007	3719	104
H(19C)	11110	4127	2319	104
H(22A)	2823	251	2423	100
H(22B)	3160	374	977	100
H(22C)	2348	1424	1660	100
H(23A)	3451	5811	4001	76
H(23B)	3633	5036	5219	76
H(23C)	2695	4462	4706	76
H(25)	9004	380	-83	46
H(26)	10259	532	-2232	56
H(27)	9795	2065	-3698	58
H(28)	8055	3416	-3001	58
H(29)	6796	3281	-849	46