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Supplementary Material

General Procedure:

2-(4'-Methoxyphenyl)-2,5-dihydrofuran (**5b**):¹

To a stirred solution of *p*-methoxyphenyllead triacetate (**1b**) (200 mg, 0.40 mmol) and NaOMe (88 mg, 1.60 mmol) in CH₃CN/benzene (1 : 1, 6 mL) was added Pd₂(dba)₃·CHCl₃ (20 mg, 5 mol %) followed by 2,3-dihydrofuran (**3**) (34 mg, 0.48 mmol) *via* syringe and the reaction mixture was stirred at room temperature for 3 h. The reaction mixture was extracted with ether (20 mL) and washed with water 3 times (20 mL x 3), and the organic layer was dried over anhydrous MgSO₄ evaporated *in vacuo*. The crude product was separated by SiO₂ column chromatography (EtOAc/hexanes 1 : 10, R_f = 0.32) to afford the coupled product **5b** (57 mg, 80%). TLC, SiO₂, EtOAc/hexanes 1 : 10, R_f = 0.32. ¹H NMR (400 MHz, CDCl₃) δ 3.80 (s, 3H), 4.74 (m, 1H), 4.83 (m, 1H), 5.75 (m, 1H), 5.87 (m, 1H), 6.04 (m, 1H), 6.89 (m, 2H), 7.24 (m, 2H). IR (neat) 3065, 2903, 1602, 1490, 1253, 793 cm⁻¹. MS (EI): *m/e* (relative intensity) = 176 (47), 175 (42), 147 (17), 145 (54), 135 (100), 115 (22).

Methyl *trans*-cinnamate (**4b**):

TLC, SiO₂, EtOAc/hexanes 1 : 10, R_f = 0.44. ¹H NMR (400 MHz, CDCl₃) δ 3.86 (s, 3H), 6.48 (d, 1H, *J* = 16.3 Hz), 7.41 (m, 3H), 7.55 (m, 2H), 7.73 (d, 1H, *J* = 16.3 Hz). IR (KBr) 3057, 2953, 1715, 1268 cm⁻¹. MS (EI): *m/e* (relative intensity) = 162 (63), 131 (100), 103 (49), 77 (34), 51 (29). Literature: *Beil.* 9, 581. Available from Aldrich Chemical Co. Inc..

(2*S*, 3*S*, 4*E*)-1-*O*-Benzyloxy-5-phenyl-4-penten-2,3-diol (**4c**):²

TLC, SiO₂, EtOAc/hexanes 1 : 1, R_f = 0.65. ¹H NMR (400 MHz, CDCl₃) δ 2.63 (m, 1H), 2.72 (m, 1H), 3.64 (m, 2H), 3.77 (m, 1H), 4.36 (m, 1H), 4.57 (m, 2H), 6.20 (dd, 2H, *J* = 16, 6.7 Hz), 6.66 (d, 1H, *J* = 16 Hz), 7.34 (m, 10H). IR (neat) 3399, 2919, 1113 cm⁻¹. MS (EI): *m/e* (relative intensity) = 133 (100), 115 (14), 91 (88), 77 (11).

(2*S*, 3*S*, 4*E*)-1-*O*-Benzyloxy-5-(4'-methoxyphenyl)-4-penten-2,3-diol acetonide (**4d**):

TLC, SiO₂, EtOAc/hexanes 1 : 15, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 1.48 (s, 6H), 3.64 (m, 2H), 3.74 (s, 3H), 3.98 (m, 1H), 4.39 (m, 1H), 4.59 (m, 2H), 6.03 (dd, 1H, *J* = 9.6, 4.2 Hz), 6.57 (d, 1H, *J* = 9.6 Hz), 6.84 (m, 2H), 7.30 (m, 7H). IR (neat) 3055, 2988, 1607, 1512, 1265

cm⁻¹. MS (EI): m/e (relative intensity) = 175 (25), 161 (21), 134 (72), 103 (24), 91 (100). HRMS calcd for C₂₂H₂₆O₄, 354.1763; found, 354.1831.

(2S, 3S, 4E)-1-O-Benzoyloxy-5-(4'-methoxyphenyl)-4-penten-2,3-diol cyclic carbonate (4e):

TLC, SiO₂, EtOAc/hexanes 1 : 2, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 3.66 (m, 1H), 3.80 (m, 1H), 3.82 (s, 3H), 4.50~4.65 (m, 3H), 5.13 (m, 1H), 6.05 (dd, 1H, J = 9.5, 5.1 Hz), 6.66 (d, 1H, J = 12 Hz), 6.88 (d, 1H, J = 12 Hz), 7.34 (m, 7H). IR (neat) 3065, 2986, 1802, 1607, 1264 cm⁻¹. MS (EI): m/e (relative intensity) = 249 (51), 161 (70), 147 (26), 134 (44), 121 (25), 91 (100), 65 (12). HRMS calcd for C₂₀H₂₀O₅, 340.1283; found, 340.1311.

2-Phenyl-2,5-dihydrofuran (5a):³

TLC, SiO₂, EtOAc/hexanes 1 : 30, R_f = 0.30. ¹H NMR (400 MHz, CDCl₃) δ 4.80 (m, 1H), 4.88 (m, 1H), 5.80 (m, 1H), 5.90 (m, 1H), 6.05 (m, 1H), 7.29~7.38 (m, 5H). IR (neat) 3028, 2964, 1612, 1598, 1427, 890 cm⁻¹. MS (EI): m/e (relative intensity) = 146 (50), 117 (30), 115(47), 105 (100), 91 (15), 77 (33).

2-(2'-Thienyl)-2,5-dihydrofuran (5c):⁴

TLC, SiO₂, EtOAc/hexanes 1 : 30, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 4.72 (m, 1H), 4.85 (m, 1H), 5.94 (m, 1H), 6.04 (m, 1H), 6.09 (m, 1H), 6.98 (m, 2H), 7.27 (m, 1H). IR (neat) 3098, 1594, 1400, 925, 857, 830 cm⁻¹. MS (EI): m/e (relative intensity) = 152 (3), 146 (47), 117 (40), 115 (51), 105 (100), 91 (19), 77 (42).

References for Supplementary Material

1. Ozawa, F.; Kubo, A.; Hayashi, T. *J. Am. Chem. Soc.* **1991**, *113*, 1417-1419.
2. Kang, S-K.; Jung, K-Y.; Park, C-H.; Jang, S-B. *Tetrahedron Lett.* **1995**, *36*, 8047-8050.
3. Larock, R. C.; Gong, W. H. *J. Org. Chem.* **1990**, *55*, 407-408.
4. Kang, S-K.; Yamaguchi, T.; Kim, J-S.; Choi, S-C.; Ahn, C. *Synth. Commun.* **1997**, *27*, 1105-1110.